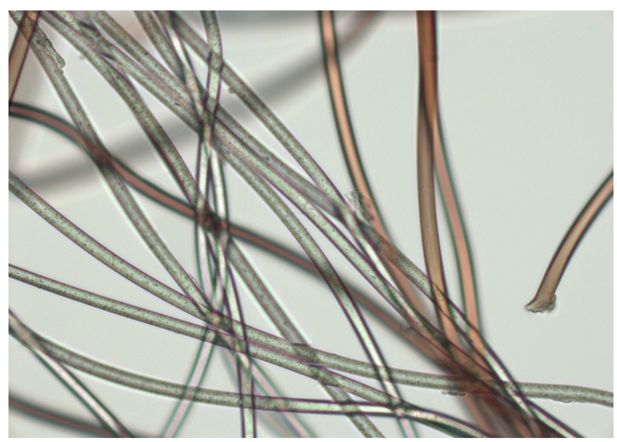
European Commission

JRC SCIENTIFIC AND POLICY REPORTS

Fibre Labelling
Polytrimethylene terephthalate - PTT- DuPont

Final report
Administrative Arrangement N. 2011- 32490
Analysis conducted on behalf of DG ENTERPRISE

P. Piccinini, C. Senaldi, J. F. Alberto Lopes
2014



Report EUR 26564 EN

Joint Research Centre

European Commission
Joint Research Centre
Institute for Health and Consumer Protection

Contact information
Paola Piccinini
Address: Joint Research Centre, Via Enrico Fermi 2749, TP 260, 21027 Ispra (VA), Italy
E-mail:JRC-IHCP-CAT@ec.europa.eu
Tel.: +39 0332 789124
Fax: +39 0332 786012

http://jrc.ec.europa.eu/

This publication is a Reference Report by the Joint Research Centre of the European Commission.

This document replaces "Fibre Labelling. Polytrimethylene terephthalate - PTT- DuPont. Final Report." with ISBN number 978-92-79-35276-8 and PUBSY request number JRC 87535. The corrections made in the new document revised few sentences in the executive summary.

JRC 89087

EUR 26564 EN

ISBN 978-92-79-36513-3 (PDF)

ISSN 1831-9424 (online)

doi: 10.2788/34915

Luxembourg: Publications Office of the European Union, 2014

Printed in Luxembourg

Fibre Labelling
Polytrimethylene terephthalate - PTT- DuPont

Final report
Administrative Arrangement N. 2011- 32490
Analysis conducted on behalf of DG ENTERPRISE

P. Piccinini, C. Senaldi, J. F. Alberto Lopes

TABLE OF CONTENTS

1. Executive summary

In 2011 the Directorate General Enterprise and Industry (DG ENTR) of the European Commission received a petition presented by E. I. du Pont de Nemours and Company (DuPont) requesting the establishment of a new generic fibre name under the Directive 2008/121/EC on textile names, now repealed by the EU Regulation 1007/2011. This would allow the differentiation among their fibre, polytrimethylene terephthalate (PTT) and, in particular, polyethylene terephthalate (PET) and polybutylene terephthalate (PBT), the two most common types of polyesters on the market. According to the petitioner, although the three polyesters are very similar in terms of chemical composition, PTT fibres have a set of improved properties that justify the petition, such as durability, resilience, easy-care, UV and bleach resistance and comfort-stretch properties, among several others.

In agreement with the EU Regulation 1007/2011, PTT belongs to the fibre class of polyester and can be labelled as such. However, the European Commission, in line with the views of the Commission Expert Group on Textile Names and Labelling, decided to analyse the fibre's properties and the possibility to create subclasses of generic fibre names.

As identification and quantification methods are required in order to allow the market surveillance of textile products, the European Commission's Directorate General Joint Research Centre (DG JRC) was responsible for the verification of the methods proposed by the applicant and for the development and validation of the new required ones.

Methods for the identification of PTT are available. Fourier Transform Infrared Spectroscopy (FT-IR) can distinguish between PTT, PET and PBT. In addition, Differential Scanning Calorimetry (DSC) can achieve the same, but only on the basis of their crystallisation peaks, since PTT and PBT's melting point take place almost at the same temperature.

The mechanical properties of PTT were studied and, on the basis of results, PTT could not be considered an elastic fibre, since it showed a modest elastic recovery at 25% elongation.

Regarding quantification, the usual pre-treatment protocol described in the EU Regulation 1007/2011 was evaluated and is applicable to PTT fibres. The correction factor *b* for mass loss during pre-treatment was established as 0%. The experimental value for the *agreed allowance* (moisture regain) was determined as 0.34%, however the members of the European Network of National Experts on Textile Labelling (ENNETL) agreed on the value 1.50% for consistency with the values adopted for polyester and elastomultiester. Fifteen quantification methods listed in the above mentioned regulation were tested in order to establish the solubility properties of PTT and to evaluate its correction factors *d* (mass loss in each dissolution method), in the case it was proved insoluble. PTT is completely soluble in method 14, whereas Method 15 is not applicable to binary mixtures containing PTT. Manual separation and chemical dissolution methods, whenever feasible, can be used for quantification purposes. They were used for the quantification of PTT in mixtures with other fibres. Results obtained via manual separation were considered as reference values against which the trueness of other methods was established. The JRC developed a new DSC method that was proved to be accurate and adequate for the quantification of PTT in blends with PET. The method uses calibration curves prepared with PTT and PET yarns manually separated from the sample under analysis, thus ensuring a common thermal history. Different types of integration, as well as multipoint and single point calibration curves based on PTT or PET melting peaks were evaluated. The JRC organised the validation of the optimised DSC method at European level, as a balanced uniform–level experiment with six levels and 15 laboratories. Statistical analysis showed that the best results were obtained using multipoint calibration curves based on the integration of PTT melting peak with a linear integration. The method was successfully validated and showed good accuracy, in terms of both trueness and precision, as proved by the following parameters: bias values (0.06 -1.30%), confidence limits at 95 % probability level (0.60 - 1.07%) and HORRAT values (0.5 – 2).

The experimental results were presented in two meetings of the European Network of National Experts on Textile Labelling, held in Ispra, Italy, on 30th November 2012 and 4th October 2013. The definition proposed by DuPont for PTT ("*fibre formed of linear macromolecules comprising at least 85% (by mass) in the chain of an ester of 1,3-propane diol and terephthalic acid*") was consistent with the evaluation carried out. As regards the proposed name of the fibre (*triexta*), there was no consensus among the experts belonging to ENNETL.

2. Extended summary for DG ENTR

In January 2011, E. I. du Pont de Nemours and Company (DuPont) submitted an application to the European Commission's Directorate General Enterprise and Industry (DG ENTR) for the establishment of a new generic fibre name under Directive 2008/121/EC on textile names, now repealed by the EU Regulation 1007/2011. A new classification was requested by the applicant, in order to allow the differentiation among their fibre, polytrimethylene terephthalate (PTT), and the other types of polyester: polyethylene terephthalate (PET) and polybutylene terephthalate (PBT). Chemically the three polyesters differ only on the number of methylene groups of the aliphatic chain that links the terephthalic moieties: two (PET), three (PTT) and four (PBT) methylene groups. The proposed new name was *triexta*, with the following definition: "*fibre formed of linear macromolecules comprising at least 85 % (by mass) in the chain of an ester of 1,3-propane diol and terephthalic acid*". The PTT fibre was claimed to present some interesting properties for consumers, such as durability, resilience, easy care, dyeability, UV and bleach resistance, elasticity, softness and comfort-stretch properties.

According to the legislation in force, PTT is part of the fibre class of polyesters, whose definition is: "*fibre formed of linear macromolecules comprising at least 85% (by mass) in the chain of an ester of a diol and terephthalic acid*". In the absence of an amendment of Annex I of the EU regulation 1007/2011, it should be labelled as such. Nevertheless, in May 2011, on the basis of the fibre characteristics, the European Commission, in agreement with the Commission Expert Group on Textile Names and Labelling, decided to evaluate technically and experimentally the application and the possibility to create subclasses of generic fibre names. In November 2011 the European Commission's Directorate General Joint Research Centre (DG JRC) was entrusted to perform the technical evaluation of the petition. The work included the verification of the validity and applicability of the testing methods, proposed by DuPont, for the identification and quantification of the PTT fibre and, if needed, the development and validation of new test methods. The results of this study were presented and discussed during the 12th and 13th meetings of the European Network of National Experts on Textile Labelling (ENNETL), held in Ispra, Italy, on 30th November 2012 and 4th October 2013.

Name and definition

The DG JRC and the members of the ENNETL network concurred with the PTT definition proposed by the petitioner. However, they were of the opinion that the proposed name *triexta* does not fulfil the criteria set up in 2002 for the establishment of a new generic fibre name, which stated that a generic fibre name shall give information about the chemical composition of the fibre.

Identification methods

Solubility properties can be used only to identify the polyester nature of the fibre, but not to differentiate among the types of polyesters. Optical microscopy is not an adequate method for PTT identification. Results confirmed that PTT can be identified using Fourier Transform Infrared Spectroscopy (FT-IR) and Differential Scanning Calorimetry (DSC). FT-IR was able to distinguish among the three types of polyester. DSC could differentiate PTT and PET on the basis of their melting peaks and among PTT, PET and PBT on the basis of their crystallisation peaks.

Mechanical properties

The elongation at break of PTT yarns from bobbin was always lower than 64% and no higher than 105% in the case of PTT yarns manually separated from fabric samples. Many PTT yarn samples broke before reaching 50% elongation, and so their elastic properties were tested at 25% elongation to avoid rupture. Under these conditions, PTT showed an elastic recovery in the range 65.7 – 78.1%, corresponding to permanent deformations varying from 5.4 to 8.8% for yarns from bobbin. The permanent deformation values went up to 12.0 % in the case of PTT yarns separated from fabric samples. On this basis, PTT could not be considered an elastic fibre, but a fibre showing some moderate elastic properties.

Parameters for quantification

The normal pre-treatment described in the EU Regulation 1007/2011 proved to be applicable to PTT and its correction factor *b* for mass loss during pre-treatment was established (0%). Experts agreed to adopt 1.50% as the PTT's *agreed allowance* (humidity regain) for consistency with the already established values for polyester and elastomultiester, even if the experimental value was 0.34 %. The *d* correction factors (mass loss of the insoluble component) were established for all the methods of the Regulation. PTT was soluble in method 14, insoluble in methods 1-11, 13 and 16, and partially soluble in method 15. The numbers of the methods are the ones reported in the EU Regulation 1007/2011.

Method	Solvent	T (ºC)	*d*
1	acetone	RT	1.01
2	sodium hypochlorite	20	1.00
3	formic acid/zinc chloride	40	1.00
4	formic acid, 80%	RT	1.01
5	benzylalcohol	52	1.01
6	dichlorometane	RT	1.03
7	sulfuric acid, 75%	50	1.00
8	dimethylformamide	100	1.03
9	carbon disulfide/acetone	RT	1.01
10	acetic acid	RT	1.01
11	sulfuric acid, 75%	RT	1.00
13	xylene	boiling	1.02
14	sulfuric acid, conc.	RT	soluble
15	cyclohexanone	boiling	1.05
16	formic acid	90	1.03

In order to quantify mixtures with elastane, the solubility properties of this fibre were studied since the *d* correction factors were not mentioned in the EU Regulation 1007/2011. The tested methods were 3, 4, 7, 8 and 14. Elastane was soluble in all the mentioned methods, except in method 4 for which the *d* factor was 1.02.

Quantification methods (manual separation and chemical dissolution)

Manual separation was applied, whenever possible, to quantify binary and ternary mixtures containing various percentages of PTT. Comparison of the results obtained in two different laboratories confirmed that this method showed good trueness and precision. The results were used as reference values against which the trueness of alternative methods was determined.

Chemical dissolution methods 1-11, 13, 14 and 16 could be used to quantify binary blends containing PTT. Binary mixtures PTT/cotton and PTT/elastane were quantified with method 7, blends PTT/polyamide with method 4 and PTT/wool with method 2. The trueness (expressed as bias) of methods 4 and 2 could be determined and confirmed the accuracy of these methods. PTT/PET binary mixtures could not be quantified by chemical methods because their solubility properties are basically the same.

Quantification methods (DSC)

DSC was used to quantify binary mixtures PTT/PET. The proposed DSC method foresaw quantification based on the integration of the PTT melting peak on the second heating cycle after cancellation of the thermal history of samples during the first one.

Calibration curves were prepared with handmade physical mixtures of pure PTT and PET yarns, independent from the blends under analysis. Good linear calibration curves were obtained but the quantification was not sufficiently accurate, with bias values higher than 1 % up to 3.5 %. The same was true when the PTT melting peaks were integrated on the first heating cycle, where the fusion endotherm peak was better defined and easier to integrate.

Several experiments proved that various pure PTT samples showed statistically different fusion enthalpies. These differences were responsible for the not satisfactory quantification results. On the request of ENNETL experts, three approaches were tested to try erasing the thermal history of PTT samples or homogenising their crystallisation degree: 1) variation of the cooling rate between the first and the second DSC heating cycle; 2) heat-treatment of samples before the DSC analysis (annealing); 3) pre-treatment of samples before DSC analysis and then quenching them with liquid nitrogen. None of these approaches proved to give sufficient accurate results, invalidating the use of calibration curves prepared with whatever PTT and PET yarns (independent calibration curves).

The JRC developed a DSC method relying on calibration curves prepared with PTT and PET yarns manually separated from each fabric sample under evaluation. In this way the calibrations were prepared with yarns that had experienced exactly the same thermal process as the blends being quantified. It was evaluated the influence on quantitative results of five different types of integration and the use of multipoint or single point calibrations based on the integration of either the PTT or the PET melting peak. In-house, the method was proved to be sufficiently accurate and it was subjected to a validation exercise.

Method validation (DSC)

The JRC organised a validation exercise at European level according to ISO 5725:1994 involving fifteen laboratories. The collaborative trial was a balanced uniform–level experiment including six levels. Quantification of six binary mixtures PTT/PET were carried out with both multipoint and single point calibrations based on the integration of both PTT and PET melting peaks. The homogeneity study demonstrated that all samples were homogeneous. The statistical analysis of test results was performed applying the robust method described in Part 5 of ISO 5725. The best quantification results were obtained using multipoint calibration curves based on the integration of PTT melting peak, and consequently this approach was selected as the one to be followed. The method proved to be able to give results very close to reference values, with biases between 0.06 and 1.30 %. Moreover, the confidence limits, calculated at 95 % probability level, were between 0.60 and 1.07 %, thus confirming also a good precision. Finally, the HORRAT parameter values were in the range 0.5 - 2, indicating the acceptability of the method of analysis with regards to the reproducibility, which is the among-laboratory precision. In conclusion, the DSC method could be considered fully validated with the final values of PTT content, limit of repeatability, limit of reproducibility and bias for the six levels reported hereafter.

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
PTT, %	29.21	59.38	64.02	71.62	74.88	76.10
bias, %	0.71	nd	1.11	1.30	0.79	0.06
r, %	2.09	5.31	4.17	3.64	3.10	2.85
R, %	3.57	5.31	4.19	4.86	7.63	8.16

Based on the results of the collaborative trial and discussion with experts of the ENNETL network, the following final Standard Operating Procedure (SOP) was agreed upon:

- 5 to 8 mg of sample;
- temperature programme: 150 °C - 5 °C/min - 300 °C;
- nitrogen flow of 50 ml/min;
- DSC lid pans pierced to allow an inert atmosphere inside the pans;
- multipoint calibration curve based on the linear integration of PTT melting peak.

3. Introduction

E. I. du Pont de Nemours and Company (DuPont) in the beginning of 2011 requested to the European Commission's Directorate General Enterprise and Industry (DG ENTR) the establishment of a new generic fibre name under Directive 2008/121/EC on textile names [1], now substituted by the EU Regulation 1007/2011 [2]. The applicant applied for a new classification to enable the distinction between the polytrimethylene terephthalate (PTT) fibre and a more common type of polyester fibre usually made by polyethylene terephthalate (PET). This request was supported by the claimed properties of PTT, which could be of importance to the general public, such as durability, resilience, easy care, dyeability, UV and bleach resistance, and in particular elasticity, softness and comfort-stretch properties. Under the current legislation, PTT can be labelled with the name polyester, as chemically speaking it is a polyester and the polyester definition (*fibre formed of linear macromolecules comprising at least 85 % (by mass) in the chain of an ester of a diol and terephthalic acid*) applies. The applicant requested a new generic name with the following definition: "*fibre formed of linear macromolecules comprising at least 85 % (by mass) in the chain of an ester of 1,3-propane diol and terephthalic acid*". The proposed name was *triexta*.

The application was evaluated on 25th May 2011 during a meeting of the Working Group on Textile Names and Labelling, composed of Member States' governmental experts. The following agreed set of criteria were used for the evaluation of the petition:

1. the fibre should be radically different from other fibres by chemical composition and/or by manufacturing route and production process;
2. fibre characteristics could be taken into account, but need to be examined on a case by case basis;
3. the fibre should be detectable and distinguishable from other fibres by standardised test methods;
4. consumer relevance should be shown by active commercial use of the fibre;
5. a new generic name is justified only if the fibre cannot be classified into existing groups.

Even though the first criterion is not fulfilled by PTT, the group considered that the application could be technically and experimentally evaluated on the basis of its fibre characteristics. Therefore, it was judged that experimental work was needed to verify the applicability of the proposed analytical methods for identifying and quantifying PTT in blends. The work required in particular validated test methods at EU level in order to enable market surveillance authorities in Member States to determine the composition of textile products containing the PTT fibre. Delegated act(s) may subsequently be adopted by the European Commission to amend Annex I (list of textile fibre names), Annex VIII (methods for quantitative analysis) and Annex IX (*agreed allowances*) to Regulation (EU) No 1007/2011.

In November 2011, the European Commission's Joint Research Centre (JRC) was entrusted by DG Enterprise and Industry (DG ENTR) to conduct the experimental work to verify the validity and applicability of the testing methods proposed by the applicant for the identification and quantification of the PTT fibre (Administrative Arrangement between JRC and DG ENTR, JRC Ref. Contract n. 32490).

4. Background information

The work plan included:

1) the verification of the applicability of the pre-treatment, described in the EU Regulation 1007/2011, to the PTT fibre;
2) the determination of the percentage mass loss due to pre-treatment (*b*);
3) the evaluation of PTT's *agreed allowance*;
4) the solubility properties of PTT;
5) the determination of its correction factors *d* (mass loss due to the application of the selected chemical methods);
6) the measurement of its elongation and elastic properties, according to the standardised methods.

The most important issue concerned the verification of identification and quantification methods proposed by DuPont (based on microscopic and FT-IR analysis, Differential Scanning Calorimetry (DSC) and mechanical properties analysis). The applicant also proposed a Nuclear Magnetic Resonance (NMR) based method for the quantification of PTT in mixtures. However, it was discarded, because the NMR equipment is not available in many laboratories and, moreover, it is a very expensive technique, especially when compared with the other proposed methods.

The JRC collaborated with the applicant to identify relevant samples for the experimental phase, taking into consideration possible range of compositions in blends. In view of possible difficulties in the quantification of blends containing PET, the binary mixtures PTT/PET were judged as the most interesting ones. DuPont was asked to provide various samples of pure PTT, both yarns from bobbin and staple fibres, with different linear densities (expressed in dtex[1]), together with binary and ternary mixtures with polyester, elastane, polyamide, cotton, wool and modal. Table 1 lists all samples received from DuPont. The samples used in this project were both yarns and fabrics, received by the JRC from end February 2012 until August 2012. Samples **293-297, 300,** and **317** were yarns from bobbin made of pure PTT, sample **301** was a fabric of pure PTT, while sample **299** was the only one made of staple fibre of pure PTT. Sample **316** was yarn from bobbin made of pure PET. All the binary and ternary blends provided were fabric samples. Two JRC samples were also used in the studies: PET (**015**) and PBT (**086**).

Table 1: Samples provided by DuPont.

JRC code	Composition	Sample type	Colour	Arrival date	Linear density dtex	Filament number
	Pure fibre					
293	100% PTT	yarn from bobbin	white	2012.02.23	81	72
294	100% PTT	yarn from bobbin	white	2012.02.23	56	34
295	100% PTT	yarn from bobbin	white	2012.02.23	78	34
296	100% PTT	yarn from bobbin	white	2012.02.23	83	72
297	100% PTT	yarn from bobbin	grey	2012.02.23	1379	70
299	100% PTT	staple fiber	white	2012.03.21	1.7	
300	100% PTT	yarn from bobbin	white	2012.03.21	55.6	24
301	100% PTT	woven, plain weave	brown	2012.03.21	81	36
317	100% PTT	yarn from bobbin	white	2012.07.09		
316	100% PET	yarn from bobbin	white	2012.07.09		
	Binary Mixtures					
298	66% PTT - 34% PET	woven fabric	grey	2012.02.23		
302	65% PTT - 35% PET	woven, plain weave	grey	2012.03.21	81	36
303	50% PTT - 50% PET	knit, mesh	pink	2012.03.21	81	72
308	55% PTT - 45% PET	woven fabric	light blue	2012.06.14		
309	75% PTT - 25% PET	woven fabric	grey	2012.06.14		
310	70% PTT - 20% PET	woven fabric	black	2012.07.09		
311	60% PTT - 40% PET	woven fabric	brown	2012.07.09		
312	60% PTT - 40% PET	woven fabric	blue	2012.07.09		
313	48% PTT - 52% PET	woven fabric	black	2012.07.09		
315	79% PET - 21% PTT	woven fabric	black	2012.07.09		
305	80% PTT - 20% Elastane	knit, warp knit	blue	2012.03.21	55.6	24
306	58% Polyamide - 41% PTT	woven fabric	purple	2012.06.14		
314	70% Cotton - 30% PTT	woven fabric	white	2012.07.09		
321	70% Cotton - 30% PTT	woven fabric	white	2012.08.28		
323	60% Cotton - 40% PTT	knit	blue	2012.08.28		
322	76% PTT - 24% Merino Wool	knit	grey	2012.08.28		
	Ternary mixtures					
304	68% Modal - 28% PTT - 5% Elastane	knit, single jersey	black	2012.03.21		
307	76% PTT - 17% PET - 7% Polyamide	woven fabric	black	2012.06.14		
324	58% ProModal - 37% PTT - 5% Elastane	knit	red	2012.08.28		

[1] **dtex** is a unit to express linear density, numerically equal to the weight in grams of 10 000 meters of yarn, fibre or other textile strand.

5. Test methods for identification and characterisation of the PTT fibre

The methods proposed by the applicant for identifying PTT were based on visual and microscopic inspection, Fourier Transform Infrared Spectroscopy (FT-IR), Differential Scanning Calorimetry (DSC) and analysis of mechanical properties. In this section results obtained with these techniques are reported. It has to be highlighted that, due to its chemical composition, the PTT fibre has to be distinguishable in particular from other types of polyester, such as polyethylene terephthalate (PET) and polybutylene terephthalate (PBT).

5.1 Microscopy

The optical microscopic analysis of pure PTT and PET are shown in Figures 1 and 2, respectively. A Zeiss microscope model Axioskop 2 Mat equipped with an Axiocam camera was used and analyses were performed using transmitted light. Glyceryl triacetate (refractive index: 1.158) was used as mounting medium.

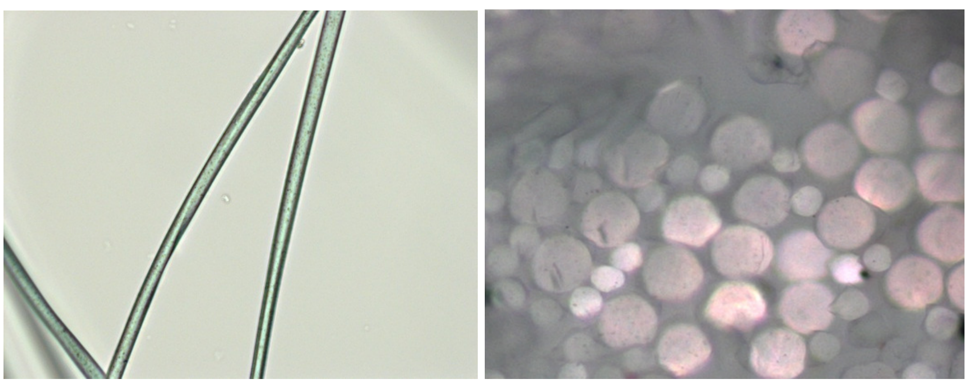


Figure 1: Longitudinal and cross section analysis of PTT 200x and 400x (sample **294**).

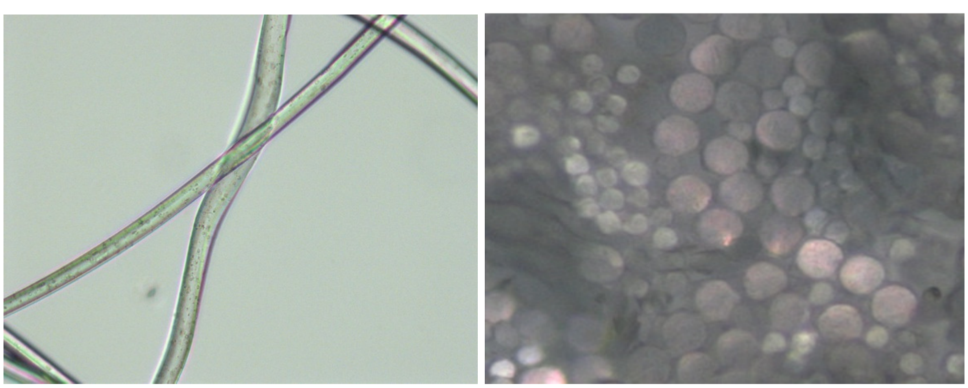


Figure 2: Longitudinal and cross section analysis of PET 400x (sample **015**).

The cross-section of PTT fibre cannot be used for its identification as the fibre can be given a variety of cross section shapes, such as round, delta and trilobal. As evident from the photos reported as an example, PTT cannot be identified by optical microscopy since it has the same appearance as the most common type of polyester (PET), and other man-made fibres in general.

5.2 Fourier transform infrared spectroscopy

The nature of the PTT fibre can be identified by means of Fourier transform infrared spectroscopy (FT-IR). All spectra were acquired using Attenuated Total Reflectance (ATR) mode with a Perkin Elmer instrument (FT-IR spectrometer spectrum 2000). Spectra were acquired in the scan range 4000.00 - 530.00 cm^{-1}, with a resolution of 4.00 cm^{-1} and a total of 4 scans. Samples were analysed without any preparation.

The FT-IR spectra of pure PTT, PET and PBT (Figures 3-5), as well as the overlay of the spectra combination PTT-PET, PTT-PBT and PET-PBT (Figures 6-8) are shown below.

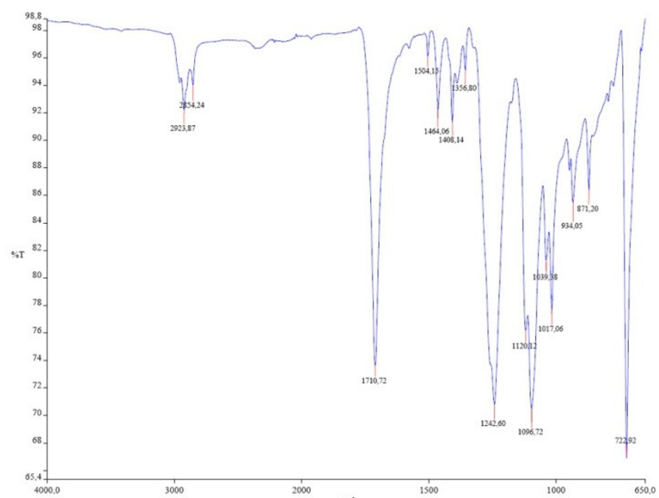


Figure 3: FT-IR spectrum of PTT (sample **297**).

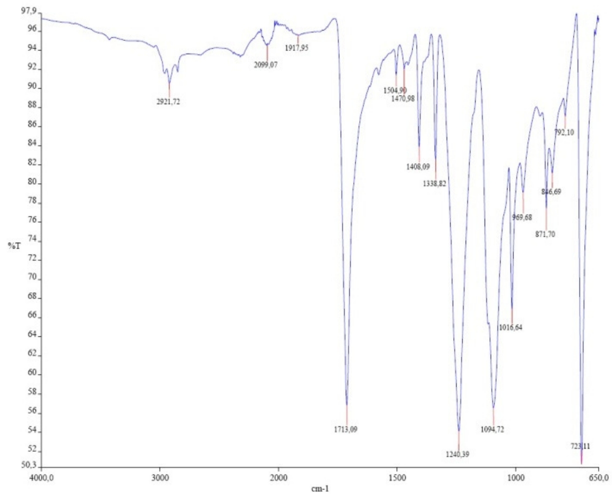


Figure 4: FT-IR spectrum of PET (sample **015**).

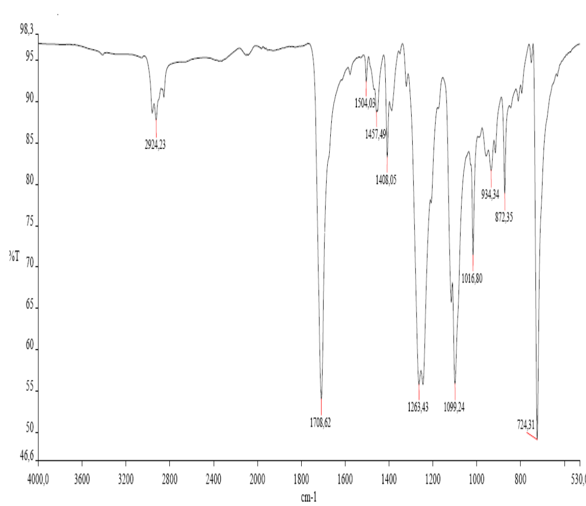


Figure 5: FT-IR spectrum of PBT (sample **086**).

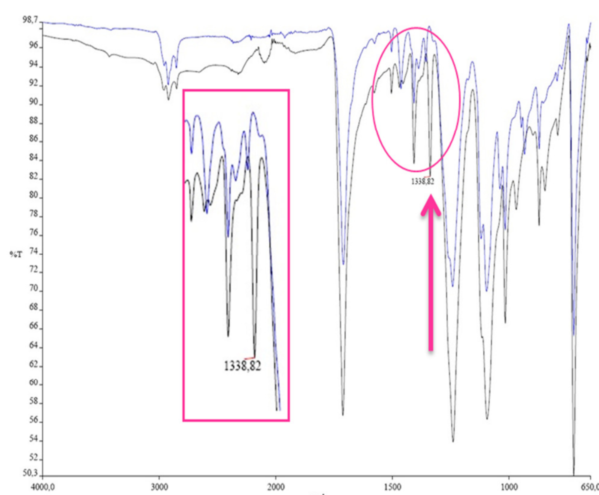


Figure 6: Overlap of FT-IR spectra of PTT (blue – sample **297**) and PET (black - sample **015**).
The pink box reports an expansion of the spectra main differences.

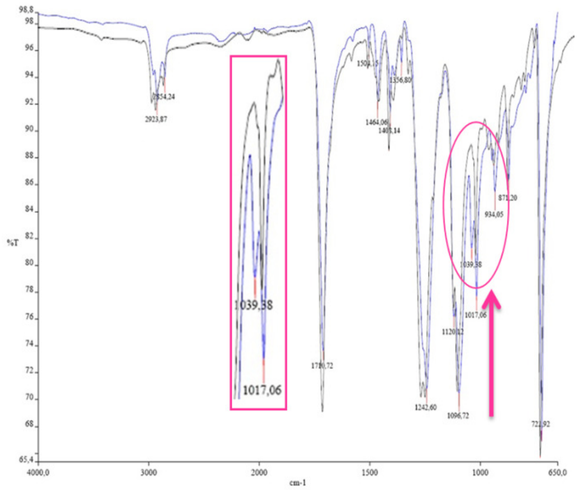


Figure 7: Overlap of FT-IR spectra of PTT (blue – sample **297**) and PBT (black - sample **086**). The pink box reports an expansion of the spectra main differences.

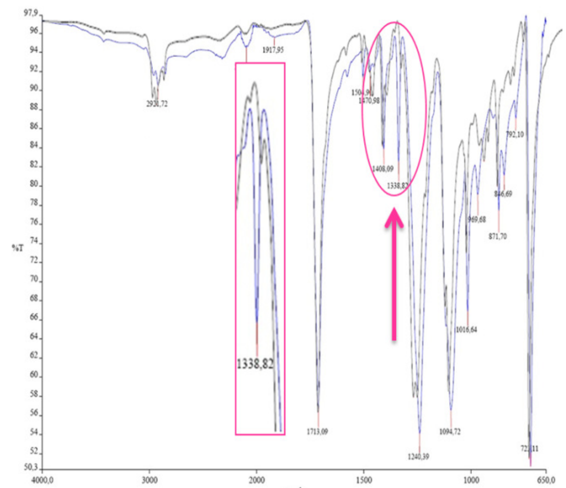


Figure 8: Overlap of FT-IR spectra of PET (blue – sample **015**) and PBT (black – sample **086**) The pink box reports an expansion of the spectra main differences.

Due to the chemical composition of the fibre, PTT spectrum shows one strong peak attributed to its ester group (1711 cm^{-1}), which can also be seen in the spectra of both PET and PBT. Among the other main bands, common also to the other polyesters PET and PBT, there is the one at 1465 cm^{-1}, which corresponds to the bending vibrations of methylene; the band at 1408 cm^{-1}, corresponding to the C-C stretching vibration in the benzene ring and the bands at 1017 and 723 cm^{-1}, which correspond to the bending vibration of the phenylic C-H bonds [3-4].

As evident from Figures 6-8, on one hand, PET can be distinguished from both PTT and PBT due to the presence of one peak at around 1339 cm^{-1} in its spectrum, which corresponds to the O-C-H bending vibration and is typical of the PET *trans* conformer. On the other hand, PTT spectrum shows one peak at 1039 cm^{-1}, which is absent in the PBT and PET spectrum. This peak corresponds to the C-C stretching mode of the PTT's three methylene units, which are arranged in a *gauche-gauche* conformation. As the methylene units of PET and PBT are arranged in different conformations (*trans-trans* and *trans-gauche-trans,* respectively) their spectra lack the 1039 cm^{-1} peak. Thus, FT-IR can be used to differentiate the three types of polyester.

5.3 Differential scanning calorimetry

On the basis of the different melting points, Differential Scanning Calorimetry (DSC) can also be used to distinguish between PTT and PET, whereas it fails to identify PTT in the presence of PBT. However, PTT, PET and PBT can be distinguished on the basis of their crystallization peaks, all of them significantly different.

The equipment used for the analyses was a DSC model Q100 by TA Instruments. A temperature program of 10 °C/min, starting from 42 °C up to 300 °C, with a nitrogen gas flow of 50 ml/min, was employed. The experimental method used consisted in a heating - cooling - heating cycle. Samples weight were in the range 4 - 10 mg. Figure 9 shows the crystallisation and melting peaks of pure PTT, which appear at 161 °C and 224 °C (first heating,) and 227 °C (second heating), respectively. Analogously, the crystallisation and melting peaks of pure PET were measured at 210 °C and 254 °C (first heating) and 255 °C (second heating), respectively (Figure 10). For pure PBT melting peaks were detected at 226 °C (1^{st} heating) and 223 °C (2^{nd} heating), and crystallisation peak at 194 °C (Figure 11).

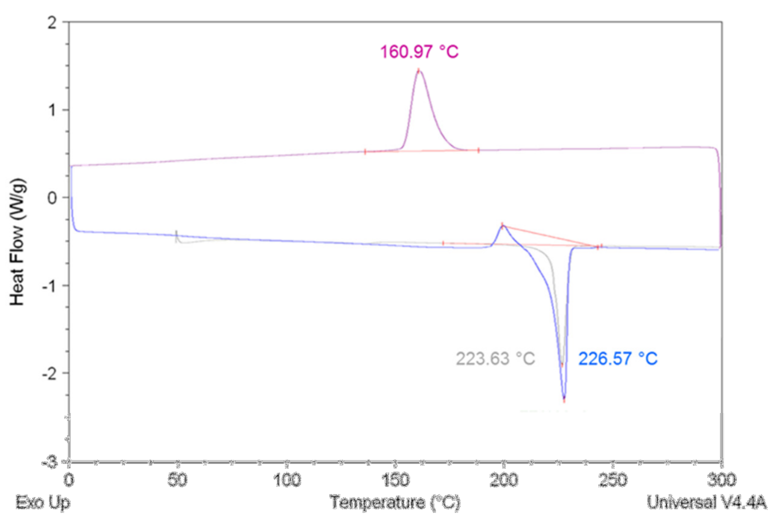


Figure 9: DSC analysis of PTT (sample **296**).

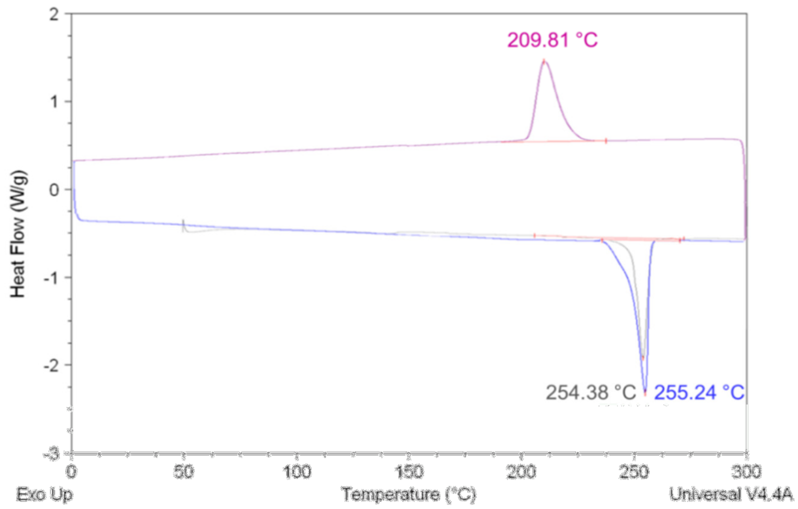


Figure 10: DSC analysis of PET (sample **015**).

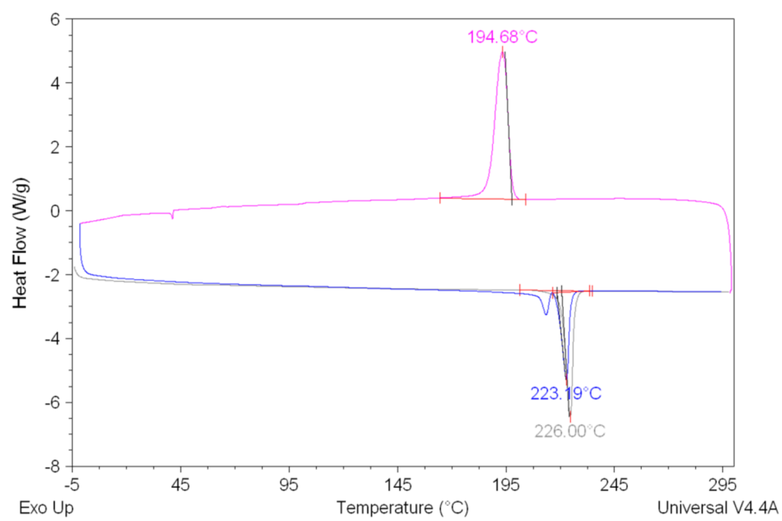


Figure 11: DSC analysis of PBT (sample **086**).

5.4 Elongation at break

The method applied to determine the elongation at break of PTT yarns is described in chapter 6 (tensile properties) of the BISFA manual regarding test methods for bare elastane yarns [5]. The principle of this method foresees to mount a yarn specimen in the clamps of a tensile testing machine and to stretch it, at a constant rate of extension, until rupture. The breaking force, maximum load, per cent elongation at break and per cent elongation at maximum load were measured per each yarn sample on ten replicates.

An Instron dynamometer, model 5544 was used to perform tests with a clamping assembly with both flat jaws made of an alum alloy (Figure 12).

Figure 12: Clamping assembly used with Instron dynamometer.

A load cell of 5 N was used except with sample **297,** for which a load cell of 50 N was needed. The following test conditions were applied: speed of moving clamp 500 mm/min, pretension 0.001 ± 0.0001 cN/dtex, gauge length 50 ± 1.0 mm. Before sampling yarns from bobbins, at least 100 meters from each package were removed and discarded. In the case of fabric samples, some PTT yarns were manually separated from them in order to be analysed. Before testing, all specimens were conditioned without any stress in standard atmosphere (21 ± 1 °C, 65 ± 2 % relative humidity) for at least 16 hours.

Figure 13 shows the trend of a typical curve load versus extension for PTT yarns, where the load continuously increases with extension until rupture.

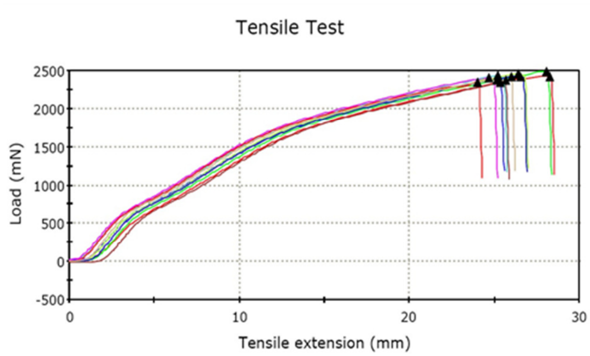


Figure 13: Load versus extension curve for PTT yarns (sample **293**).

Results reported in Table 2 suggested that the load at break of yarns depends on their linear density: the higher the linear density the higher the load at break. In fact, it increased from 1490.8 to 7476.2 mN when the linear density augmented from 56 to 1379 dtex.

Table 2: Elongation at break of pure PTT yarns from bobbin.

JRC code	Composition	dtex	Filaments number	dtex per filament	Pretension mg	Repl.	Load at break mN	Elongation at break %
293	100% PTT	81	72	1.1	86	10	2408.7 ± 33.8	51.9 ± 2.0
294	100% PTT	56	34	1.6	50	10	1490.8 ± 149.6	45.7 ± 3.1
295	100% PTT	78	34	2.3	86	11	2442.7 ± 80.6	32.9 ± 1.4
296	100% PTT	83	72	1.1	86	10	2395.7 ± 135.5	43.4 ± 2.0
297	100% PTT	1379	70	20	1400	10	7476.2 ± 5656.1	129.8 ± 88.5
300	100% PTT	56	24	2.3	50	10	1584.8 ± 52.2	63.7 ± 2.5
317	100% PTT	81	unknown	unknown	86	10	2401.5 ± 60.1	44.8 ± 2.2

Table 3: Elongation at break of pure PTT yarns manually separated from fabric samples.

JRC code	Composition	dtex	Filaments number	dtex per filament	Pretension mg	Repl.	Load at break mN	Elongation at break %
301	100% PTT	81	unknown	unknown	80	10	2489.8 ± 20.8	59.3 ± 0.9
302	100% PTT	81	36	2.3	80	10	2208.8 ± 75.8	71.5 ± 2.9
309	100% PTT	unknown	unknown	unknown	80	10	2781.6 ± 63.4	73.3 ± 1.7
311	100% PTT	unknown	unknown	unknown	80	10	2266.0 ± 89.3	105.2 ± 2.3
312	100% PTT	unknown	unknown	unknown	80	10	2301.9 ± 79.8	95.9 ± 1.8
322	100% PTT	unknown	unknown	unknown	80	10	2142.5 ± 75.0	69.3 ± 1.5

Some issues with repeatability of results were noticed in the case of sample **297**. Experimentally, it was noticed that the break of all the 70 filaments did not happen contemporaneously; on the contrary, some filaments broke first and others subsequently, so that the dynamometer did not record the break always at the same point of extension and load. This could be possibly due to the much higher value of linear density per filament of this sample (20 dtex/filament) in comparison with all the other samples (1.1-2.3 dtex/filament).

The elongation at break for PTT samples was moderate, in the range 33 – 130 %, and apart from sample **297**, which showed large variations, was always lower than 105 %.

5.5 Elastic recovery

The standard method EN 15930:2010 [6] regarding the elasticity of fibres was applied to evaluate the elastic properties of PTT yarns. This method covers the determination of recoverable stretch and permanent deformation of elastic yarns and is applicable to continuous filament yarns.

Both PTT yarns from bobbin and manually separated from fabrics were tested. Also in this case, before sampling yarns from bobbin, at least 100 meters from each package were removed and discarded. In the case of yarns from fabrics, particular attention was paid at the moment of the separation in order to avoid any stress that could have caused alterations in the elastic properties of the samples.

Before testing, specimens were conditioned without any stress in standard atmosphere (21 ± 1 °C, 65 ± 2 % relative humidity) for at least 16 hours. The method was applied using the same equipment (Instron dynamometer, model 5544), clamping assembly and load cell used to test the elongation at break. In addition, also a Favimat tensile tester (Textechno, Germany) was employed. In the last case a load cell of 220 cN was used. The following test conditions were applied: speed of moving clamp 50 mm/min, pretension 0.001 ± 0.0001 cN/dtex, gauge length 50 ± 1.0 mm. In the case of the Instron dynamometer, the load was set to zero after mounting specimens with attached their pretension weights in the clamping assembly. Yarn extension was measured at pretension. Figure 14 shows the clamping assembly of Favimat tester and Figure 15 reports the method profile.

Figure 14: Clamping assembly used with Favimat dynamometer.

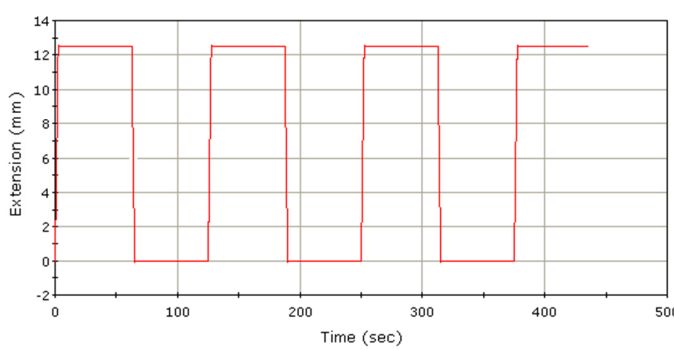


Figure 15: Three-cycle method profile.

Specimens were extended to 25 % elongation and maintained at this elongation for one minute and then they were allowed to relax for one minute, after returning to the initial gauge length. The cycle was repeated two more times; finally specimens were extended again at the same per cent elongation. Such a small elongation (25 %) was selected as the majority of PTT samples broke before reaching 50 % elongation. Specimen extension was measured at pretension load on the fourth load cycle.

Based on this measurement the per cent elastic recovery and permanent deformation (PD) of specimens were calculated (see equations 4.5.1 and 4.5.2).

$$\textit{elastic recovery} = \frac{E_{spec} - E_{rec}}{E_{spec}} \times 100 \qquad 4.5.1$$

$$\textit{elastic recovery} = \frac{E_{rec}}{L_{init}} \times 100 \qquad 4.5.2$$

where:

E_{spec} is the specified extension of the fibre, expressed in mm

E_{rec} is the extension determined at the specified pretension force on the fourth load cycle (recovery extension), expressed in mm

L_{init} is the initial length at the specified pretension on the first cycle, expressed in mm

As an example, the load versus extension curve obtained for a yarn is reported in Figure 16.

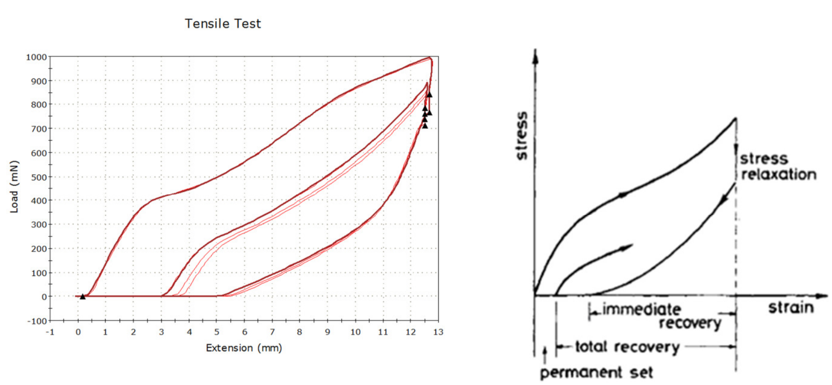


Figure 16: Curve load versus extension for the three-cycle method (sample **300**).

The elastic recovery of PTT yarns from bobbin was in the range 65.7 – 75.0 %, corresponding to permanent deformations ranging from 6.2 to 8.8 %, when measured with the Instron equipment (Table 4). The results obtained with the Favimat tester were in good agreement with the ones measured with the Instron dynamometer, as they were in the range 70.3 -78.1 % (Table 5). It was not possible to measure the elastic properties of sample **297** with Favimat tester, because the load cell was overloaded. When the Favimat equipment was used, a slight modification of the method was applied. In this case, specimens were mounted on the clamping assembly without pretension weights as the pretension was applied directly by the instrument. Before running the three-cycle program, the length at the pretension was used as initial length for calculations.

Table 4: Elastic recovery and permanent deformation of pure PTT yarns from bobbin. Measurements performed using Instron dynamometer.

JRC code	declared dtex	Pretension mg	Repl.	Elastic recovery %	Permanent deformation %
293	81	86	12	69.28 ± 2.27	7.68 ± 0.57
294	56	50	10	74.34 ± 0.86	6.22 ± 0.19
295	78	86	10	73.17 ± 0.71	6.71 ± 0.18
296	83	86	10	72.39 ± 2.82	6.90 ± 0.70
297	1379	1400	10	65.73 ± 4.10	8.84 ± 0.95
300	56	50	10	70.98 ± 1.50	7.26 ± 0.38
317	not known	80	10	75.04 ± 1.55	6.24 ± 0.39

Table 5: Elastic recovery and permanent deformation of pure PTT yarns from bobbin. Measurements performed using Favimat dynamometer.

JRC code	declared dtex	Pretension mg	Repl.	Elastic recovery %	Permanent deformation %
293	81	81	10	74.8 ± 0.55	6.21 ± 0.14
294	56	56	10	77.47 ± 0.43	5.55 ± 0.11
295	78	78	10	78.08 ± 0.37	5.40 ± 0.09
296	83	83	10	74.02 ± 0.75	6.38 ± 0.19
297	1379	1379	-	*cell overloaded*	*cell overloaded*
300	56	56	10	70.32 ± 0.36	7.31 ± 0.09
317	not known	80	-	*not feasible*	*not feasible*

Elongation tests were carried out also on PTT yarns manually separated from fabric samples using the Favimat equipment. As the linear density of these PTTs yarns was not known, it was applied a pretension of 80 mg, in the same range as the one applied to study the pure fibres from bobbins. As shown in Table 6, the elastic recovery of manually separated PTT yarns was in the range 51.3 – 69.0 %, showing 20 to 30 % decrease compared to PTT yarns from bobbin. Consequently, the permanent deformation values were substantially higher, varying between 7.6 and 12.0 % correspondent to an increase of 20 – 35 %. These differences could be due to the fabric manufacture process the fibres undergo, which could slightly alter their elastic properties. Alternatively, another hypothesis could be that the manual separation could have slightly damaged the fibres.

Independently from the dynamometer used, the elastic recovery did not seem to depend on the sample's linear density. In these conditions, PTT showed some elastic properties, however it has to be highlighted that the elongation was set at only 25 %. Generally at EU level a fibre is considered elastic if it shows a permanent deformation lower than 10 % at 50 % elongation; this is not the case for PTT.

Table 6: Elastic recovery and permanent deformation of PTT yarns manually separated from fabrics. Measurements performed using Favimat dynamometer.

JRC code	declared dtex	Pretension mg	Repl.	Elastic recovery %	Permanent deformation %
301	81	80	10	69.00 ± 0.28	7.64 ± 0.07
302	81	80	10	58.83 ± 0.93	10.12 ± 0.72
309	not known	80	10	51.30 ± 0.27	12.00 ± 0.06
311	not known	80	10	53.64 ± 1.50	11.42 ± 0.37
312	not known	80	10	61.75 ± 1.55	9.45 ± 0.39
322	not known	80	-	*not feasible*	*not feasible*

Finally, the linear densities of PTT yarns from bobbins were evaluated with both the gravimetric and the vibroscopic methods [7] using Favimat. The gravimetric method could not be used in the case of PTT yarns manually separated from fabric samples.As shown in Tables 7 and 8, there was a good agreement between the linear densities measured with the two methods and usually also with the declared values.

Table 7: Linear density of pure PTT yarns from bobbin.

JRC code	declared dtex	gravimetric method dtex	vibroscopic method dtex
293	81	88	86
294	56	59	56
295	78	81	78
296	83	85	81
297	1374	1437	not feasible
300	56	56	51
317	not known	89	84

Table 8: Linear density of pure PTT yarns manually separated from fabric samples.

JRC code	declared dtex	vibroscopic method dtex
301	81	87
302	81	78
309	not known	110
311	not known	87
312	not known	71
322	not known	not feasible

6. Test methods for quantification of the PTT fibre

Initially, the JRC verified the applicability of the usual pre-treatment to the PTT fibre and determined both its mass loss due to pre-treatment and its *agreed allowance*. In a second phase, the behaviour of the fibre was studied with all the methods described in the EU Regulation 1007/2011, with the exception of method 12 used for the determination of nitrogen content. The application of the various chemical dissolution methods allowed the determination of the correction factors *d* for PTT's mass loss. In a third phase, the DSC method proposed by the applicant for the quantification of blends PTT/PET was evaluated. Finally, all the samples made by binary and ternary mixtures received from DuPont (**298, 302-315, 321-324**) were analysed, if applicable, by manual separation, chemical analysis and DSC analysis. All results of this section are reported in Annex IV.

6.1 Pre-treatment

Before quantification, samples should be pre-treated in order to eliminate non-fibrous matter. EU Regulation 1007/2011 foresees extracting non-fibrous matter with light petroleum ether and water. The procedure foresees one-hour extraction in Soxhlet with light petroleum ether (boiling range 40 - 60 °C), followed by one-hour extraction in water at room temperature and one-hour extraction in water at 65 ± 5 °C, using a liquor/specimen ratio of 100/1. Both the traditional Soxhlet and an automatic hot-extractor (Soxhtec) were employed for the pre-treatment. No differences, both in terms of FT-IR spectra of the pre-treated samples and of their mass loss, were noticed during preliminary experiments.

In order to evaluate the *b* coefficient for the PTT fibre (mass loss due to pre-treatment), the pre-treatment was carried out on six replicates, two grams each, of pure PTT (samples **296** and **293**). Results (Table 9) showed for sample **296** a mass loss of 0.73 ± 0.05 % and 0.63 ± 0.06 %, using Soxhlet and Soxhtec, respectively (the confidence interval at 95 % probability is reported). The mass loss of sample **293** pre-treated in Soxhtec was 0.61 ± 0.07 %. These values are in line with the content of finishing agents, in the range of 1.0 %, declared by DuPont. Three consecutive pre-treatments were performed in order to evaluate if further loss of mass occurred during the second and third pre-treatment. Sample **296** was used for this test and, as shown in

Table 10, no significant further mass loss was evidenced. These results confirmed that the fibre was insoluble under the conditions of the pre-treatment. Therefore, in agreement with experts from ENNETL, the usual pre-treatment was considered applicable and the *b* coefficient value for PTT was established as 0 %.

In Figure 17 it can be seen a comparison between the FT-IR spectra of a PTT sample (**297**) as received and after pre-treatment. No differences were found between the two spectra.

Table 9: Mass loss due to pre-treatment.

JRC Code	Composition	Description	Repl.	Mass loss %	Conf. limit at 95 %	Equipment
296	100% PTT	yarn from bobbin 83 dtex	6	**0.73**	0.05	**Soxhlet**
296	100% PTT	yarn from bobbin 83 dtex	6	**0.63**	0.06	**Soxhtec**
293	100% PTT	yarn from bobbin 81 dtex	6	**0.61**	0.07	**Soxhtec**

Table 10: Mass loss due to consecutive pre-treatment.

		1st Pre-treatment		2nd Pre-treatment		3rd Pre-treatment		
JRC Code	Replic.	Mass loss %	Conf.limit at 95 %	Mass loss %	Conf.limit at 95 %	Mass loss %	Conf.limit at 95 %	Equipment
296	6	**0.64**	0.08	**0.12**	0.03	**0.13**	0.02	**Soxhtec**

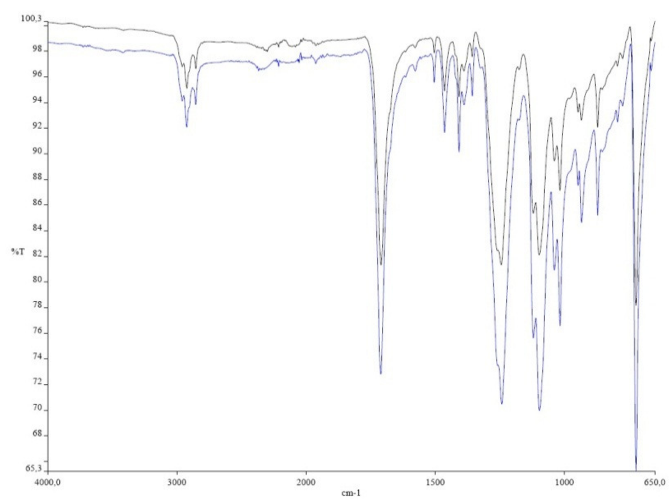


Figure 17: Overlap of the FT-IR spectra of untreated (black) and pre-treated (blue) PTT (sample **297**).

6.2 Agreed allowance

The *agreed allowance* corresponds to the moisture regain in standard atmosphere according to the definition stated in ISO 6348:1980 [8].

A number of experiments were performed on pure PTT, both yarn and staple fibre, (samples **296**, **297** and **299**) with different linear densities, in order to evaluate the fibre's *agreed allowance*. This parameter was calculated both for untreated and pre-treated samples. The procedure described hereafter was applied. Weighing bottles were dried for 5 hours at 105 °C, then cooled in a dessicator for at least 2 hours and weighed. A sample of about 2 g of PTT was placed in each weighing bottle and dried for 16 hours at 105 °C, then cooled in a dessicator for at least 2 hours and weighed. Samples were then conditioned for 72 hours at 20 ± 1 °C and 65 ± 2 % relative humidity and weighed immediately after the conditioning period. The following formulas were used to calculate the *agreed allowance*:

water mass = wet sample mass – dried sample mas 6.2.1

agreed allowance = 100 x (water mass / dried sample mass) 6.2.2

Ten replicates per each sample were analysed (Table 11). The untreated sample **296** was also analysed by DuPont on six replicates (it is reported as **296*** in Table 11). Results were similar for untreated and pre-treated samples and were in the range of 0.28 – 0.40 %, with the average being 0.34 %.

Table 11: *Agreed allowance* (AA) for PTT.

				untreated sample		pre-treated sample	
JRC code	Composition	Description	Repl.	AA %	Conf. limit at 95 %	AA %	Conf. limit at 95 %
296	100% PTT	yarn - 83 dtex	10	0.38	0.04	0.32	0.04
297	100% PTT	yarn - 1379 dtex	10	0.31	0.04	0.28	0.03
299	100% PTT	staple fiber	10	0.40	0.03	0.39	0.03
296*	100% PTT	yarn - 83 dtex	6	0.28	0.03		
		average		0.34		0.33	
		overall average			0.34		

Although the experimental value for PTT was 0.34 %, after discussions with experts from Member States, and considering the established values for the *agreed allowances* of polyester and elastomultiester in the EU regulation 1007/2011 (both equal to 1.50 %) and the value proposed by DuPont (1.50 %), it was agreed to establish the same value of 1.50 % for the *agreed allowance* of PTT.

6.3 Solubility properties

After pre-treatment, the solubility properties of PTT were investigated and the correction factors *d* for mass loss of the insoluble component in the reagents during analysis were evaluated. The correction factors *d* were calculated using the following formula:

$$d = \frac{m}{r} \qquad 6.3.1$$

where:

m is the dry mass of the specimen after pre-treatment

r is the dry mass of the residue

All weighing operations were performed using an analytical balance of weighing capacity of 0.01 mg. The percentages of insoluble component on a clean, dry mass basis, disregarding loss of fibre mass during pre-treatment, were calculated using the following formula:

$$P_1\ \% = \frac{100\ r\ d}{m} \qquad 6.3.2$$

where:

P_1 is the percentage of clean, dry insoluble component

m is the dry mass of the specimen after pre-treatment

r is the dry mass of the residue

d is the correction factor for loss of mass of the insoluble component in the reagent during analysis

In the case of binary mixtures, calculations of percentage of insoluble component on clean, dry mass basis, with adjustment by conventional factors (*agreed allowances*) and, where appropriate, correction factors b for loss of mass during pre-treatment, were performed using the following formula:

$$P_{1A}\ \% = \frac{100\ P_1\left(1+\frac{a_1+b_1}{100}\right)}{P_1\left(1+\frac{a_1+b_1}{100}\right)+\left(100-P_1\right)\left(1+\frac{a_2+b_2}{100}\right)} \qquad 6.3.3$$

where:

P_{1A} is the percentage of insoluble component, adjusted by *agreed allowances* and for loss of mass during pre-treatment

P_1 is the percentage of clean, dry insoluble component as calculated from equation 5.3.2

a_1 is the *agreed allowance* for the insoluble component (listed in Annex IX to the EU Regulation 1007/2011 on textile fibre names and related labelling and marking of the fibre composition of textile products)

a_2 is the *agreed allowance* for the soluble component (listed in Annex IX to the EU Regulation 1007/2011 on textile fibre names and related labelling and marking of the fibre composition of textile products)

b_1 is the percentage mass loss of insoluble component caused by the pre-treatment

b_2 is the percentage mass loss of soluble component caused by the pre-treatment

The percentage of the soluble component (P_{2A} %) was obtained by difference.

The coefficients *b* used in the calculations were: 0 % for polyester, elastane, polyamide, cotton, wool and modal (as pointed out in the EU regulation 1007/2011) and also for PTT. The *agreed allowances* used in the calculations were: 1.50 % for PTT, 1.50 % for elastane, 5.75 % for polyamide, 8.50 % for cotton, 18.25 % for wool, 13.00 % for modal.

Apart from method 12 for nitrogen content, pre-treated specimens of about 1 g of PTT were analysed with all the chemical dissolution methods described in EU Regulation 1007/2011. An overview of all the applied methods is presented in Table 12.

For each sample 10 - 20 replicates were analysed. The data was collected and subjected to statistical evaluation. The results were first examined for evidence of outliers using Grubbs' statistical test, as laid down in ISO 5725-2 [9]. Only very few outliers were found and eliminated out of all measurements. The valid results were then subjected to a further statistical evaluation. The average and standard deviation (SD) of each set of data were calculated, as well as the coefficient of variation (CV). The CV was used to measure the dispersion of the distribution of test results in the JRC laboratory: the lower the value of CV, the better the repeatability of the method. The confidence intervals were calculated at 95 % probability, using the following formula:

$$\mu = x_m \pm \frac{t\ s}{\sqrt{n}} \qquad 6.3.4$$

where:

t is the value listed in the Student's t-distribution for a certain number of degrees of freedom and level of probability

s is the estimated standard deviation

μ is the true value

x_m is the average of experimental results

n is the number of measurements

An overview of results regarding the solubility properties of PTT is shown in Table 13.

Table 12: Overview of the used methods described in the EU regulation 1007/2011.

Method	Solvent	T (ºC)
1	acetone	RT
2	sodium hypochlorite	20
3	formic acid/zinc chloride	40
4	formic acid, 80%	RT
5	benzylalcohol	52
6	dichlorometane	RT
7	sulfuric acid, 75%	50
8	dimethylformamide	100
9	carbon disulfide/acetone	RT
10	acetic acid	RT
11	sulfuric acid, 75%	RT
13	xylene	boiling
14	sulfuric acid, conc.	RT
15	cyclohexanone	boiling
16	formic acid	90

Table 13: Solubility properties of PTT.

JRC code	Method	Repl.	PTT %	Conf. limit at 95 %	JRC results *d* factor	Conf. limit at 95 %	rounded *d* factors
296	1	20	99.15	0.09	1.009	0.001	1.01
296	2	20	100.18	0.11	0.998	0.001	1.00
296	3	20	99.67	0.07	1.003	0.001	1.00
296	4	20	99.31	0.11	1.007	0.001	1.01
296	5	20	98.91	0.12	1.011	0.001	1.01
296	6	20	97.18	0.04	1.029	0.0004	1.03
296	7	20	99.74	0.07	1.003	0.001	1.00
296	8	20	96.99	0.06	1.031	0.001	1.03
296	9	20	98.95	0.16	1.011	0.002	1.01
296	10	20	99.16	0.09	1.008	0.001	1.01
296	11	20	99.59	0.08	1.004	0.001	1.00
296	13	20	98.09	0.04	1.020	0.0005	1.02
301	14	10	99.96	0.12	soluble		soluble
296	15	10	95.06	0.50	1.052	0.006	1.05
296	16	20	97.26	0.07	1.028	0.001	1.03

Table 14 reports the results obtained for PTT in the DuPont's laboratories. The comparison among results obtained at the JRC and DuPont plus the already established *d* correction factors for polyester and elastomultiester are reported in Table 15.

The correction factors *d* obtained by the JRC and DuPont were in good agreement. Out of 14 *d* factors newly determined, 6 were equal, 7 differed by 0.01 and only one differed by 0.02. Comparing the solubility properties of PTT, polyester and elastomultiester, PTT showed from slightly to moderate higher solubility in methods 1, 4, 6, 8 and 9.

Table 14: Solubility properties of PTT (DuPont results).

JRC code	Method	Repl.	PTT %	Conf. limit at 95 %	DuPont results *d* factor	Conf. limit at 95 %	rounded d factors
296	1	6	99.75	0.13	1.002	0.001	1.00
296	2	6	99.76	0.34	1.002	0.003	1.00
296	3	6	99.08	0.33	1.009	0.003	1.01
296	4	6	98.99	0.09	1.010	0.001	1.01
296	5	6	98.22	0.33	1.018	0.003	1.02
296	6	6	98.23	0.22	1.018	0.002	1.02
296	7	6	99.27	0.30	1.007	0.003	1.01
296	8	6	97.05	0.11	1.030	0.001	1.03
296	9	6	99.12	0.34	1.009	0.003	1.01
296	10	6	99.14	0.65	1.009	0.007	1.01
296	11	6	98.09	0.60	1.020	0.006	1.02
296	13	6	97.09	0.15	1.030	0.002	1.03
301	14	6			soluble		soluble
296	15	6	96.49	0.32	1.036	0.003	1.04
296	16	6	96.92	0.28	1.032	0.003	1.03

Table 15: Solubility properties of PTT, polyester and elastomultiester.

	JRC	Dupont		
	experimental *d* factors		*d* factors in Reg. 1007/2011	
Method	PTT	PTT	polyester	elastomultiester
1	1.01	1.00	1.00	1.00
2	1.00	1.00	1.00	1.00
3	1.00	1.01	-	-
4	1.01	1.01	1.00	1.00
5	1.01	1.02	-	-
6	1.03	1.02	1.01	1.01
7	1.00	1.01	1.00	1.00
8	1.03	1.03	1.01	1.01
9	1.01	1.01	1.00	1.00
10	1.01	1.01	-	-
11	1.00	1.02	-	-
13	1.02	1.03	-	-
14	soluble	soluble	soluble	soluble
15	1.05	1.04	-	-
16	1.03	1.03	-	-

PTT is completely insoluble in methods 2, 3, 7 and 11 (d = 1.00); it can be considered insoluble also in methods 1, 4-6, 8-10, 13 and 16 (d = 1.01, 1.01, 1.01, 1.03, 1.03, 1.01, 10.1, 1.02, 1.03, respectively); whereas it is partially soluble in method 15 (d = 1.05). PTT is completely soluble only in method 14 (concentrated sulphuric acid).

Applying method 14 some difficulties were experienced when pure PTT yarns were analysed. In this case, when the acid was added the yarn sample was contracted and formed aggregates that could not be dissolved completely, unless a strong mechanical agitation was applied for the entire duration of the contact time. This was a deviation to the procedure described for method 14 which foresees to stir occasionally by hand. However, no problems were experienced when the method was applied to a pure PTT fabric, as in this case the normal procedure was able to dissolve completely the sample. Considering that, in general, enforcement laboratories would have to analyse the final consumer products, such as clothing that are in the form of fabric, no difficulties should be expected by the application of method 14.

The possible influence of various linear densities and production sites on the solubility properties of PTT was studied using methods 7 and 16, because they were considered the "worst case scenario" due their strong acid conditions (75 % sulphuric acid and 90 % formic acid, respectively) and high temperatures (50 °C and 90 °C, respectively). Method 7 was applied to all pure PTT samples received (yarn, staple fibre and fabric). Results can be found on Table 16.

Table 16: Influence of linear density and production site on solubility properties of PTT.

JRC code	Linear density dtex	Method	Repl.	PTT %	Conf. limit at 95 %	*d* factors	Conf. limit at 95 %	Rounded *d* factors
293	81	**7**	10	98.49	0.21	**1.015**	0.002	**1.02**
294	56	**7**	5	99.79	0.09	**1.002**	0.001	**1.00**
295	78	**7**	5	99.58	0.21	**1.004**	0.002	**1.00**
296	83	**7**	10	99.71	0.09	**1.003**	0.001	**1.00**
297	1379	**7**	5	99.54	0.09	**1.005**	0.001	**1.00**
299	1.7	**7**	5	99.46	0.12	**1.005**	0.001	**1.01**
300	56	**7**	5	99.77	0.14	**1.002**	0.001	**1.00**
301	81	**7**	5	100.26	0.14	**0.997**	0.001	**1.00**
317	unknown	**7**	5	99.25	0.13	**1.008**	0.001	**1.01**
			average			**1.004**		**1.00**
296		**16**	10	97.35	0.03	**1.027**	0.0003	**1.03**
293		**16**	10	97.12	0.04	**1.030**	0.0004	**1.03**
			average			**1.028**		**1.03**

In conclusion, blends containing PTT could be analysed with methods 1-11, 13 and 16 in which PTT remained as residue or with method 14 in which PTT is soluble. On the contrary, method 15 cannot be applied to mixtures containing PTT, due to its partial solubility.

The solubility properties of elastane were studied using methods 3, 4, 7, 8 and 14 since they were not established in the EU Regulation 1007/201, with the aim to find methods that could be used to quantify the binary mixture PTT/elastane (sample **305**) and the ternary mixtures modal/PTT/elastane (samples **304** and **324**). In the case of samples containing elastane, the normal pre-treatment with light petroleum ether and water was not applicable. The pre-treatment applied foresaw the following procedure: one-hour extraction in Gyrowash at 40 °C with an aqueous solution containing 5 g/L of the standard soap (Heal's standard soap, without optical brightening agent suitable for ISO 105 Parts C01-C05), followed by rinsing with water, using a liquor/specimen ratio of 100/1. Table 17 summarizes the determined solubility properties of elastane.

Table 17: Solubility properties of elastane.

JRC code	Method	Repl.	PTT %	Conf. limit at 95 %	*d* factor	Conf. limit at 95 %	Rounded *d* factors
95	**3**	10	99.57	0.08	**soluble**		
95	**4**	20	98.40	0.13	**1.016**	0.001	1.02
95	**7**	10	99.96	0.05	**soluble**		
95	**8**	10	100.03	0.04	**soluble**		
95	**14**	10	99.94	0.04	**soluble**		

Results showed that elastane was soluble in methods 3, 7, 8 and 14, while it was insoluble in method 4, with a *d* correction factor of 1.02.

6.4 Quantification of binary and ternary mixtures containing PTT

6.4.1 *Manual separation*

When feasible, manual separation was performed on binary and ternary mixtures in order to determine reference values against which results obtained with alternative methods were compared. In fact, manual separation is unanimously considered the most accurate one for the quantification of fibre blends.

Out of the ten binary mixtures PTT/PET available, six woven and one knitted fabrics could be analysed by manual separation. However, despite the declared composition, one of them - sample **308** – turned out to be made of pure PTT. On the following binary blends PTT/PET (samples **310**, **313** and **315**), PTT/elastane (**305**) and PTT/cotton (**314**, **321** and **323**) manual separation was not applicable, either because of the fabric construction or because the separated yarns were made by intimate mixture of different fibres. The values of *agreed allowance* used for the calculation of results were 1.50 % for PTT and polyester, 18.25 % for wool and 5.75 % for polyamide.

Table 18 reports the quantification obtained by the JRC on six, ten or twenty replicates, and by DuPont on three replicates. Results were in excellent agreement, confirming that the manual separation was a very accurate and reproducible method.

In addition the values of confidence limits calculated at 95 % probability were generally very low showing that this method is also repeatable.

For several samples, such as **302**, **303** and **311**, results showed that the declared composition was not correct.

Table 18: Quantification of binary mixtures by manual separation.

JRC code	Declared Composition	Sample type	JRC results Repl.	JRC results PTT %	JRC results Conf. limit at 95 %	DuPont results Repl.	DuPont results PTT %	DuPont results Conf. limit at 95 %
298	66% PTT - 34% PET	woven fabric	6	**66.33**	0.13			
302	65% PTT - 35% PET	woven fabric	20	**74.09**	0.07	3	**74.26**	0.17
303	50% PTT - 50% PET	knitted fabric	20	**28.50**	0.30			
309	75% PTT - 25% PET	woven fabric	20	**76.04**	0.03	3	**76.11**	0.04
311	60% PTT - 40% PET	woven fabric	20	**70.32**	0.08	3	**70.34**	0.33
312	60% PTT - 40% PET	woven fabric	20	**62.91**	0.11	3	**62.89**	0.03
306	58% polyamide - 41% PTT	woven fabric	6	**42.24**	0.39			
322	76% PTT - 24% wool	knitted fabric	10	**77.38**	0.15			
307	76% PTT - 17% PET - 7% polyamide	woven fabric	6	**76.47**	0.08			

6.4.2 *Chemical analysis*

Binary mixtures of PTT/PET could not be quantified by any chemical methods as the solubility properties of these two fibres were practically identical. Method 7 was used to quantify the binary mixtures PTT/cotton and PTT/elastane, method 4 for the blends PTT/polyamide and method 2 for PTT/wool. In all these methods, PTT was insoluble.

Results are reported in Table 19, together with the trueness of method 4 and 2 (the bias values lower than 1% were highlighted in green). The trueness of method 4 and 2 (expressed in terms of bias) was calculated for samples **306** and **322**, as the difference between the content of PTT, obtained by method 4 or 2, and its reference value, obtained by manual separation. In fact, trueness, as defined in ISO 5725 – Part 1 [10], is the closeness of agreement between the average value obtained from a large series of test results and an accepted reference value. It is usually expressed in terms of bias, which is the difference between the expectation of the test results and an accepted reference value. The quantification made by the chemical methods 4 and 2 was in very good agreement with the reference values obtained via manual separation, thus confirming the accuracy of these methods. In the case of sample **305**, the quantification carried out using method 7 showed that the declared composition was not correct, that is out of the 3 % manufacturing tolerance established in the EU regulation 1007/2011.

Table 19: Quantification of binary mixtures by chemical methods.

JRC code	Declared composition	PTT (MS) %	Method	Repl.	PTT (CM) %	Conf. limit at 95 %	Bias %
305	80% PTT - 20% elastane	-	7	10	**84.10**	0.14	-
306	42% PTT - 58% polyamide	**42.24**	4	6	**43.11**	0.33	**0.87**
322	76% PTT - 24% wool	**77.38**	2	10	**77.39**	0.13	**0.01**
314=321	30% PTT - 70% cotton	-	7	10	**29.30**	0.06	-
323	40% PTT - 60% cotton	-	7	10	**42.05**	0.15	-

Sample **307**, ternary mixture of PTT, polyester and polyamide, could not be fully quantified, as no chemical method is available for blends made by PTT and PET. In this case, only the percentage of polyamide could be determined by dissolving it with method 4, based on 80 % formic acid aqueous solution (Table 20).

Table 20: Partial quantification of sample **307** by chemical method.

JRC code	Declared composition	Method	PTT + PET %	Polyamide %
307	76% PTT - 17% PET - 7% polyamide	4	92.98	7.02

Samples **304** and **324**, ternary mixtures of PTT with modal and elastane, were quantified with two different variants as recommended by the EU Regulation 1007/2011, nonetheless the accuracy of the methods used could not be measured as the manual separation of these two samples was not feasible (Table 21). For these samples, however, the measured PTT content was in the range of the declared composition. Variant 4 foresees that two components of the mixture (A and B) are dissolved one after the other from the same specimen by applying two different methods consecutively. Variant 2 was also applied for confirmation. In this case, two specimens need to be analysed; one component (A) is removed from the first specimen, leaving as residue the other two components (BC), while two components (A and B) are dissolved contemporaneously from the second test specimen, leaving as residue the third component (C).

The *agreed allowances* used in the calculations were: 1.50 % for PTT, 1.50 % for elastane, 5.75 % for polyamide, 8.50 % for cotton, 18.25 % for wool and 13.00 % for modal. According to the analysis performed on PTT samples (Table 9), the *d* factor used for methods 2, 4 and 7 were 1.00, 1.01 and 1.00, respectively.

Table 21: Quantification of ternary mixtures by chemical methods.

JRC code	Declared composition	Method	Variant	PTT %	Modal %	Elastane %
304	68% modal - 28% PTT - 5% elastane	8 + 7	4	**24.93**	69.90	5.17
304	68% modal - 28% PTT - 5% elastane	7 and 8	2	**25.45**	69.38	5.17
324	58% modal - 37% PTT - 5% elastane	8 + 7	4	**39.20**	57.96	2.85
324	58% modal - 37% PTT - 5% elastane	7 and 8	2	**39.16**	57.99	2.85

6.4.3 *DSC method*

The analyses were performed using a Differential Scanning Calorimeter from TA Instruments, model Q100, equipped with an auto sampler. The initial analysis consisted in heating-cooling-heating cycles with a temperature program starting at 42 °C – 10 °C/min – 300 °C – 10 °C/min – 0 °C – 10 °C/min – 300 °C. The nitrogen gas flow was set at 50 ml/min, and the weight of samples was in the range 2-10 mg.

Usually the DSC first heating cycle is used to erase the thermal history of samples and the melting peaks are integrated on the second heating. However, with the PTT samples the melting peaks in the second heating cycle were misshaped and their integration was not repeatable; for this reason it was decided to integrate the melting peak on the first heating cycle, as the PTT melting peaks were much more defined and the fusion enthalpies values more repeatable. For the quantification of PTT/PET binary mixtures calibration curves were built up using handmade independent mixtures containing various percentages of PTT and PET. The samples used as standards were **296** for PTT and **015** for PET. The calibration curves were linear and showed good correlation factors (like the one showed in Figure 18).

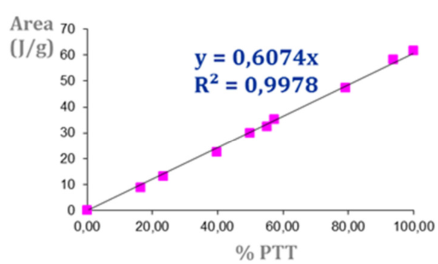


Figure 18: Calibration curve prepared with samples **296** (PTT) and **015** (PET).

Table 22 reports the quantification results of binary and ternary mixtures obtained using manual separation, chemical methods and the DSC method initially used. A color code was used to emphasise the bias results on the table: values below 1% highlighted in green (considered as good results), between 1 and 2 % in yellow (considered as acceptable results), and higher than 2 % in red (considered as unacceptable results). This code was used in all the tables presented from here onwards. The values obtained via manual separation or, when not feasible, *via* chemical methods, were considered reference values against which the trueness of the DSC method was measured. The differences between the PTT content measured by the DSC method and the reference method (manual separation when possible) correspond to the *bias*. The higher the bias the farther the results obtained via DSC are from the true values. These initial quantifications generally showed bias values higher than 1 % and often higher than 2 %, meaning that the DSC method was not accurate enough.

Table 22: Quantification by DSC method (calibration curve **296/015**).

JRC code	Declared Composition	PTT (MS) %	PTT (CM) %	PTT (DSC) %	Bias %
298	66% PTT - 34% PET	66.33	-	65.81	-0.52
302	65% PTT - 35% PET	74.09	-	76.07	1.98
303	50% PTT - 50% PET	28.50	-	29.53	1.03
308	55% PTT - 45% PET	100.00	-	102.63	2.63
309	75% PTT - 25% PET	76.04	-	78.66	2.62
310	30% PET - 70% PTT	-	-	70.00	
311	60% PTT - 40% PET	70.32	-	73.75	3.43
312	60% PTT - 40% PET	62.91	-	66.52	3.61
313	52% PET - 48% PTT	-	-	45.97	
315	79% PET - 21% PTT	-	-	58.89	
305	80% PTT - 20% elastane	-	84.10	80.63	-3.47
314	30% PTT - 70% cotton	-	29.30	31.00	1.70
322	76% PTT - 24% wool	77.38	77.39	-	
323	40% PTT - 60% cotton	-	42.05	-	
304	68% modal - 28% PTT - 5% elastane	-	25.31	26.80	1.49
324	58% modal - 37% PTT - 5% elastane	-	41.98	-	

In order to understand if the high bias values observed were caused by structural differences among the pure PTT fibres present in the binary mixtures and in the calibration curves, all available PTT samples were analysed and their melting peaks integrated on the first heating cycle. As reported in Table 23, various PTT samples showed different fusion enthalpies and the samples seemed to belong to three different groups: group I, with an average area of the melting peak equal to 64.32 J/g, group II with 61.78 J/g and group III with 59.59 J/g.

Table 23: Enthalpy of fusion for 100 % PTT samples.

Group	JRC code	Repl.	Enthalpy J/g	SD J/g	Conf. limit at 95 %
I	**293**	3	64.37	0.11	0.27
	294	3	64.21	0.09	0.21
	295	3	64.36	0.06	0.14
II	**296**	8	61.78	0.51	0.43
	297	3	61.43	0.09	0.23
	299	3	61.90	0.08	0.20
	317	3	61.94	0.,05	0.13
III	**300**	3	59.59	0.16	0.40

The *t*-Student test was applied to the average fusion enthalpies of the three groups to evaluate if they could be considered equal or not. The null hypothesis assumed that the three groups (compared two by two each time) showed the same average fusion enthalpy. First of all, the standard deviations of the two independent sets of measurements under evaluation (s_1 and s_2, with the number of replicates $n_1 = n_2$) were analysed with the *F*-test (two-sided test) to determine if they differed significantly [11].

To check the variances, the statistic *F* was calculated:

$$F = \frac{s_1^2}{s_2^2} \qquad 6.4.3.1$$

where s_1^2 is the biggest variance, as *F* must be higher than 1.

Taking into consideration the degrees of freedom for each set of measurements and the confidence level required (95 % probability), *F* values were compared with the critical value $F_{n1-1,\ n2-1}$ (P=0.05) reported in tables. When the *F* value was higher than *F* critical, it was assumed that there was a statistically significant difference between the two variances.

To judge whether the averages of two independent sets of measurements differed significantly, in the case of non-significant difference between variances, the statistic *t* was calculated as follows:

$$t = \frac{(\bar{x}_1 - \bar{x}_2)}{s\sqrt{\frac{1}{n_1} + \frac{1}{n_2}}} \qquad 6.4.3.2$$

where $\bar{x}_1$ and $\bar{x}_2$ are the sample means and n_1 and n_2 the number of replicates for the two sets of measurements. The degrees of freedom of t are $n_1 + n_2 - 2$.

The standard deviation was calculated with the following formula:

$$s^2 = \frac{(n_1 - 1)s_1^2 + (n_2 - 1)s_2^2}{(n_1 + n_2 - 2)} \qquad 6.4.3.3$$

When the difference between variances was significant, then the statistic t was calculated as follows:

$$t = \frac{(\bar{x}_1 - \bar{x}_2)}{\sqrt{\frac{s_1^2}{n_1} + \frac{s_2^2}{n_2}}} \qquad 6.4.3.4$$

with the degrees of freedom estimated using the Welch-Satterthwaiteu approximation:

$$\nu = \frac{\left(\frac{s_1^2}{n_1} + \frac{s_2^2}{n_2}\right)^2}{\left(\frac{s_1^4}{n_1^2(n_1 - 1)} + \frac{s_2^4}{n_2^2(n_1 - 1)}\right)} \qquad 6.4.3.5$$

When necessary, the calculated value of v was rounded down to the nearest integer.

Finally, t values were compared with the critical value t (P=0.05) reported in the Student's t-distribution tables. According to the t-test, the difference between the two averages could be considered not significant when the calculated $|t|$ value did not exceed the critical one.

The overview of the results is presented in Tables 24 and 25. The statistical analysis confirmed, at 95 % probability, that the average melting peak areas of the three groups of PTT samples were significantly different.

Table 24: Average enthalpies of fusion for the three groups of pure PTT.

Groups	Repl.	Enthalpy J/g	SD J/g
I	9	64.32	0.11
II	17	61.78	0.38
III	3	59.59	0.16

Table 25: Comparison of enthalpies of fusion for the three groups of pure PTT.

Group	F	F crit at 95 %	SD	T	T crit at 95 %	average
I-II	12.67	4.07	≠	-25.694	2.086	≠
I-III	2.23	6.06	=	-59.248	2.228	≠
II-III	5.69	39.43	=	9.552	2.101	≠

Given the fact that statistically significant differences were observed among samples of pure PTT analysed as received on the first heating cycle, the samples were heat-treated for 16 hours at 105 °C in a ventilated oven in order to try to erase their thermal history and to homogenise their degree of crystallinity. Sample **293** from the first group and samples **296** and **317** from the second one were studied. Results are reported in Table 26.

Table 26: Enthalpies of fusion of 100 % PTT samples before and after heat treatment.

JRC code	Replicates	Enthalpy J/g	SD J/g
293	3	64.37	0.11
293 heat treated	3	63.41	0.04
296	8	61.78	0.51
296 heat treated	3	59.92	1.61
317	3	61.94	0.05
317 heat treated	3	61.85	0.12

The *t*-Student test was applied to check if the average melting peak areas before and after heat treatment could be considered equal or not. As reported in Table 27, the null hypothesis (meaning that the results obtained before and after heat treatment would be equivalent) could be assumed for samples **296** and **317** at 95 % probability. However, this was not the case for sample **293**.

Table 27: Comparison of enthalpies of fusion before and after heat treatment of samples.

JRC code	F	F crit. at 95 %	SD	T	T crit. at 95 %	average
293	7.15	39.00	=	14.615	2.775	≠
296	9.97	6.54	≠	1.434	4.303	=
317	4.75	39.00	=	1.228	2.775	=

Results were not conclusive, as they did not clearly show whether the heat-treatment had an influence on the enthalpy of fusion of PTT samples. Due to this uncertainty, three calibration curves were then built up using handmade independent mixtures containing various percentages of PTT and PET. The samples used to calibrate (**293**, **296** and **317** for PTT, and **316** for PET) were heat-treated for 16 hours at 105 °C in a ventilated oven (Table 28).

Table 28: DSC calibration curves made with heat treated PTT and PET samples.

PTT code	PET code	m	R^2	Calibration points
296	316	0.5910	0.9968	8
317	316	0.6034	0.9979	8
293	316	0.6150	0.9962	7

Although the three calibration curves were considered linear (with correlation factors higher than 0.995), they showed different angular coefficients, which was probably the reason for the different quantification obtained for the mixtures containing PTT (Table 29). Results proved that, even applying the heat-treatment to both standards and samples under quantification, the differences between the PTT content obtained via manual separation (or chemical methods) and the DSC method – expressed in terms of bias - were, again, generally much higher than 1 %, up to 12 %, meaning that the DSC method was still not accurate.

Table 29: Quantification by DSC method (heat treated samples and standards).

			Cal. curve 296/316		Cal. curve 317/316		Cal. curve 293/316	
JRC code	**PTT (MS) %**	**PTT (CM) %**	**PTT (DSC) %**	**Bias %**	**PTT (DSC) %**	**Bias %**	**PTT (DSC) %**	**Bias %**
298	66.33	-	68.00	1.67	66.60	0.27	65.40	-0.93
302	74.09	-	70.15	-3.94	68.71	-5.38	67.41	-6.68
303	28.50	-	20.93	-7.57	20.50	-8.00	20.11	-8.39
308	100.00	-	102.98	2.98	100.86	0.86	98.96	-1.04
309	76.04	-	72.76	-3.28	71.26	-4.78	69.62	-6.42
310	-	-	62.62		61.34		60.18	
311	70.32	-	68.88	-1.44	67.47	-2.85	66.20	-4.12
312	62.91	-	59.46	-3.45	58.24	-4.67	57.14	-5.77
313	-	-	44.57		43.65		42.83	
315	-	-	57.46		56.28		55.22	
305	-	84.10	78.00	-6.10	76.40	-7.70	74.96	-9.14
314	-	29.30	29.75	0.45	29.13	-0.17	28.59	0.71
322	77.38	77.39	67.17	-10.21	65.79	-11.59	64.55	-12.83
323	-	42.05	42.89		42.01		41.22	
304	-	25.31	25.48	0.17	24.96	-0.35	24.49	-0.82
324	-	41.98	37.60		36.82		36.13	

All results presented so far indicated the existence of various groups of PTT samples showing different enthalpies of fusion. To check if these differences were real or if they could be explained by lack of repeatability of the analysis and/or peak integration, one sample from each group was analysed in five different days over three weeks, after heat-treatment for 16 hours at 105 °C in a ventilated oven. The samples chosen were **293**, **296** and **300**, representing the three groups. Statistics (*F* and *t*-test) were used once more to compare the melting peak average area for each sample on different days. These results are reported below in Tables 30-35.

Table 30: Average enthalpies of fusion of the heat treated sample **293** in different days.

JRC code	Day	Repl.	Enthalpy J/g	SD J/g
	1-Wed week 39	10	60.89	0.57
	2-Mon week 40	10	59.91	0.67
293	3-Tue week 40	10	61.21	0.79
	4-Wed week 40	10	60.34	0.96
	5-Mon week 41	10	59.57	0.44

Table 31: Average enthalpies of fusion of the heat treated sample **296** in different days.

JRC code	Day	Repl.	Enthalpy J/g	SD J/g
	1-Wed week 39	10	56.54	0.45
	2-Mon week 40	10	56.74	0.93
296	3-Tue week 40	8	57.04	0.69
	4-Wed week 40	9	56.28	0.52
	5-Mon week 41	10	56.14	0.51

Table 32: Average enthalpies of fusion of the heat treated sample **300** in different days.

JRC code	Day	Repl.	Enthalpy J/g	SD J/g
	1-Wed week 39	10	56.64	0.61
	2-Mon week 40	10	56.50	0.30
300	3-Tue week 40	10	56.08	0.62
	4-Wed week 40	10	56.61	0.51
	5-Mon week 41	10	55.75	0.84

Table 33: Comparison of average enthalpies of fusion of the heat treated sample **293** in different days.

JRC Code	Day	F	F crit at 95 %	SD	T	T crit at 95 %	Average
	1-2	1.39	4.03	=	3.503	2.101	≠**
	2-3	1.39	4.03	=	-3.960	2.101	≠**
	3-4	1.31	4.03	=	2.607	2.101	≠*
	4-5	4.80	4.03	≠	2.319	2.160	≠*
293	1-3	1.94	4.03	=	-1.058	2.101	=
	1-4	2.82	4.03	=	1.543	2.101	=
	1-5	1.70	4.03	=	5.791	2.110	≠**
	2-4	2.02	4.03	=	-1.171	2.101	=
	2-5	2.37	4.03	=	1.333	2.101	=
	3-5	3.31	4.03	=	5.727	2.145	≠**

Table 34: Comparison of average enthalpies of fusion of the heat treated sample **296** in different days.

JRC Code	Day	F	F crit at 95 %	SD	T	T crit at 95 %	Average
	1-2	1.15	4.03	=	-1.039	2.101	=
	2-3	2.77	4.36	=	-1.132	2.120	=
	3-4	1.75	4.53	=	2.544	2.131	≠*
	4-5	1.05	4.10	=	0.607	2.110	=
296	1-3	-1.85	4.20	=	2.417	2.120	≠*
	1-4	1.14	4.43	=	1.377	2.120	=
	1-5	1.86	4.03	=	1.309	2.101	=
	2-4	2.11	4.43	=	1.579	2.110	=
	2-5	2.87	4.03	=	1.500	2.101	=
	3-5	3.16	4.20	=	1.847	2.120	=

Table 35: Comparison of average enthalpies of fusion of the heat treated sample **300** in different days.

JRC Code	Day	F	F crit at 95 %	SD	T	T crit at 95 %	Average
	1-2	4.27	4.03	≠	0.649	2.160	=
	2-3	4.32	4.03	≠	1.929	2.160	=
	3-4	1.44	4.03	=	-2.075	2.101	=
	4-5	2.69	4.03	=	2.757	2.101	≠*
300	1-3	1.01	4.03	=	2.027	2.101	=
	1-4	1.43	4.03	=	0.122	2.101	=
	1-5	1.89	4.03	=	2.705	2.120	≠*
	2-4	2.99	4.03	=	-0.581	2.101	=
	2-5	8.06	4.03	≠	2.660	2.201	≠**
	3-5	1.86	4.03	=	1.011	2.101	=

In the case of samples **296** and **300**, a good repeatability was observed, as respectively in 80 % and 70 % of the comparisons the enthalpies could be considered statistically equivalent at 95 % probability. These numbers rose to 100 % and 90 % of comparisons respectively, at 99 % probability. However, for sample **293** the repeatability was lower; in fact, only in 4 or 6 comparisons out of 10 the enthalpies could be considered statistically equivalent at 95 % or 99 % probability, respectively. In Tables 32-34, the single asterisk means that the test would pass at 99 % probability, while in the opposite case a double asterisk was used.

Based on the generally good repeatability of the instrumental analysis and integration, the general mean of the enthalpies of fusion for the 3 different samples were calculated and statistically compared (Tables 36 and 37). For the heat-treated samples statistics indicated that the PTT samples could be grouped in 2 different categories. In fact, sample **293** showed a significantly higher melting peak area when compared to the ones of both samples **296** and **300**.

Table 36: General mean of enthalpies of fusion of the heat treated samples **293**, **296** and **300** analysed in different days.

JRC code	Repl.	Enthalpy J/g	SD J/g
293	50	60.39	0.91
296	47	56.53	0.69
300	50	56.31	0.68

Table 37: Comparison of average enthalpies of fusion for the three groups of pure heat treated PTT.

JRC code	F	F crit at 95 %	SD	T	T crit at 95 %	Average
293-300	1.73	1.78	=	23.29	1.985	≠
293-296	1.82	1.76	≠	25.34	1.987	≠
296-300	1.05	1.77	=	1.569	1.985	=

The conclusion of all these experiments was that PTT samples were different in terms of enthalpies of fusion on the first heating cycle, independently if before the DSC analysis they were heat-treated, for 16 hours at 105 °C in a ventilated oven, or not. Furthermore this difference could not be explained by a lack of repeatability of the DSC analysis or melting peak integration.

In order to get an accurate quantification of fibre mixtures containing PTT another approach was then tested. Whenever possible, the PTT and PET yarns separated from the fabric sample under evaluation were used to build up the calibration curve. On the one hand, this approach showed two advantages: 1) to use, as standards for the calibration curves, yarns that had gone through the same thermal process as the sample under evaluation, thus having the same thermal history and possible the same degree of crystallinity; 2) to need just a few milligrams of those standards. On the other hand, it also exhibited two drawbacks: 1) the need of building up one calibration curve for each sample to be quantified; 2) the necessity to be able to manually separate at least some milligrams of pure PTT and PET yarns from the fabric sample under evaluation.

Five calibration curves were then constructed using PTT and PET yarns manually separated from the fabric samples **298**, **302**, **309**, **311** and **312**. As reported in Table 38, good correlation factors were obtained, ranging from 0.9946 to 0.9973. In addition, the quantification results of binary mixtures PTT/PET were in very good agreement with the reference ones obtained via manual separation. The differences, expressed in terms of bias, were in all cases lower than 1 %. Furthermore they were both positive and negative, thus not showing a trend that could have been caused by a systematic error.

Table 38: Quantification by DSC method
(calibration curves prepared with PTT and PET separated from the samples under evaluation).

	Decl. comp.	MS	DSC		Calibration curve	
JRC code	**PTT %**	**PTT %**	**PTT %**	**Bias %**	**m**	**R^2**
298	66	66.33	66.81	**-0.48**	0.6017	0.9952
302	65	74.09	74.81	**-0.72**	0.6198	0.9952
309	75	76.04	75.70	**0.34**	0.5841	0.9973
311	60	70.32	71.20	**-0.88**	0.5984	0.9955
312	60	62.91	62.74	**0.17**	0.5989	0.9946

Based on the results of the five PTT/PET binary mixtures to which the approach was applicable, it could be concluded that an accurate quantification could be obtained using the DSC method, integrating the melting peak of PTT on the first heating cycle and preparing the calibration curve with PTT and PET yarns manually separated from the fabric sample under evaluation.

On 30th November 2012, these preliminary results were presented in the 12th Meeting of the European Network of National Experts on Textile Labelling (ENNETL). The discussion was about how to find a way to develop an accurate DSC method that was independent from the PTT and PET used for the calibration curve. It was decided that some more experiments should be conducted before adopting the dependent method. The aim of those experiments was to try to improve the shape of the melting peak of PTT on the second heating cycle, thus allowing its repeatable integration, and/or to erase the thermal history of samples by annealing before analysing them via DSC.

The first tests had the objective of trying to obtain statistically equivalent fusion enthalpies for the various pure PTT samples. Different cooling rates were tested, both slow and quick in order to either give time to the melted sample to form crystals or to let it solidify in an amorphous phase. Being 10 °C/min the cooling rate used for the results presented so far, the following cooling rates were additionally tested: 2.5, 5, 20 and 30 °C/min. The cooling system did not allow temperature to decrease faster than 30 °C/min. The temperature program applied was 0 °C – 20 °C/min – 300 °C – 2.5, 5, 20 or 30 °C/min – 0 °C – 20 °C/min – 300 °C. Table 38 shows the fusion enthalpies obtained integrating both the first and the second melting peak. The average values were based on three replicates.

As it can be seen in Figure 19, independently from the cooling rate used, the PTT melting peak on the second heating cycle was not easily integrated as it was always partly overlapped to an either exothermic or endothermic peak placed at lower temperature. In addition, the fusion enthalpy values, both on the first and on the second heating cycle (Table 39), varied greatly among samples of pure PTT. This implied the impossibility to get accurate quantifications of PTT in mixtures with PET based on a generic calibration curve prepared with any PTT sample.

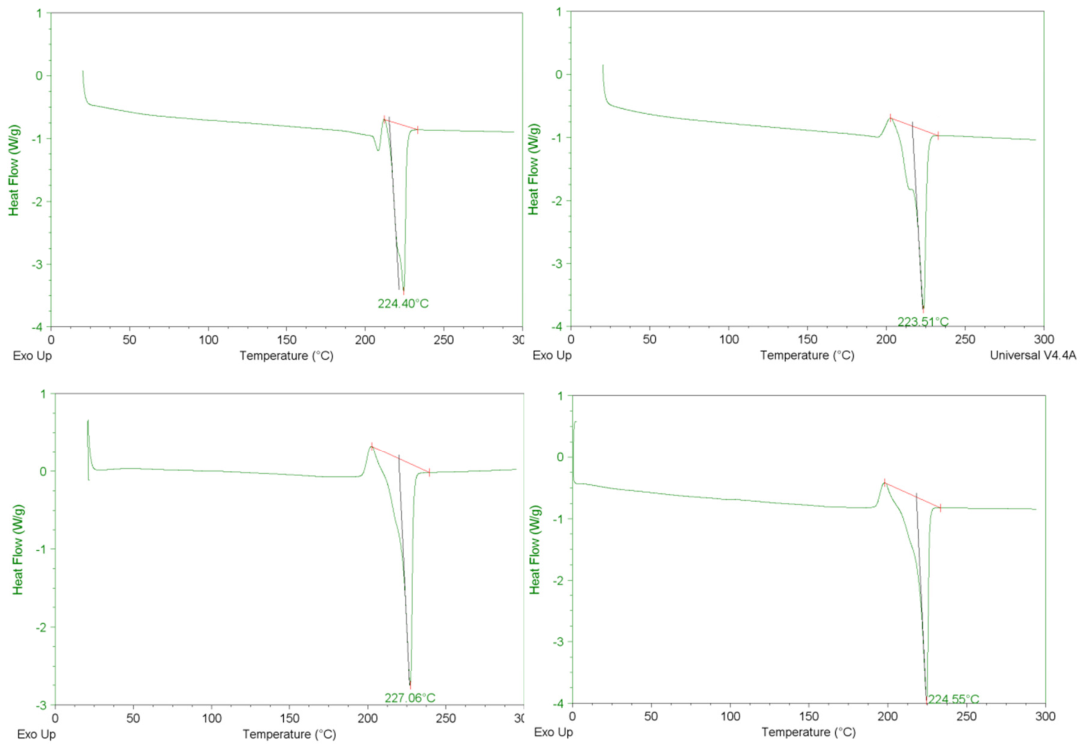


Figure 19: PTT's melting peaks on 2nd heating cycle obtained with different cooling rates (sample **296**). From left to right, top to bottom: 2.5, 5, 20 and 30 °C/minute, respectively.

Table 39: Influence of different cooling rates on enthalpies of fusion of pure PTT.

				1st heating		2nd heating	
Cooling rates ºC/min	**JRC code**	**Composition**	**Repl.**	**Enthalpy J/g**	**SD J/g**	**Enthalpy J/g**	**SD J/g**
	295	100% PTT	3	74.84	0.67	58.39	0.55
2.5	**296**	100% PTT	3	68.01	0.64	57.42	1.15
	300	100% PTT	3	67.45	1.32	57.40	1.28
	295	100% PTT	3	69.87	1.28	65.54	0.98
5	**296**	100% PTT	3	69.39	1.13	75.42	1.08
	300	100% PTT	3	70.11	0.96	76.68	0.72
	295	100% PTT	3	72.38	1.86	73.96	1.68
20	**296**	100% PTT	3	68.01	1.03	70.10	1.25
	300	100% PTT	3	69.60	0.95	72.33	1.76
	295	100% PTT	3	69.12	1.35	77.82	1.23
30	**296**	100% PTT	3	65.92	0.90	71.55	1.29
	300	100% PTT	3	66.67	0.97	73.64	1.20

An annealing method was also tested for the same purpose of achieving equivalent fusion enthalpies for different samples of pure PTT by erasing their thermal history. Selected samples were heat-treated in a ventilated oven for 16 hours at different temperatures below the melting point (130, 150 and 200 °C) prior to the DSC analysis (Figure 20). The DSC program was 0 °C – 20 °C/min – 300 °C – 30 °C/min – 0 °C – 20 °C/min – 300 °C. The results are reported in Table 40 as the averages of three replicates. Also after annealing, the fusion enthalpies of the various pure PTT samples analysed differed significantly both in the first and in the second heating cycle of each sample. Consequently, even the use of an annealing method at temperatures in the range 130 – 200 °C did not allow the use of an independent calibration curve for the quantification of binary mixtures PTT/PET.

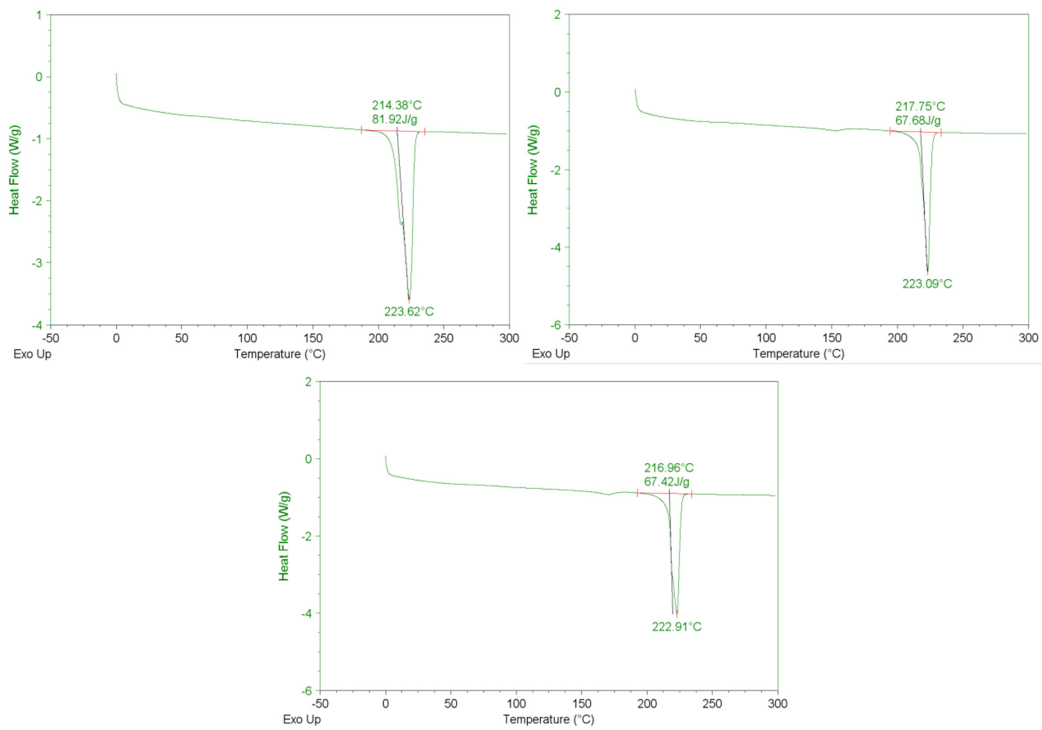


Figure 20: PTT melting peaks on 1st heating cycle of sample **296** heat-treated at different temperatures. From left to right, top to bottom: 130, 150 and 200 °C, respectively.

Table 40: Influence of different annealing temperatures on enthalpies of fusion of pure PTT.

Temperature ºC	JRC code	Repl.	1st heating Enthalpy J/g	1st heating SD J/g	2nd heating Enthalpy J/g	2nd heating SD J/g
130	**295**	3	74.02	1.40	86.77	1.61
	296	3	69.48	0.65	83.86	0.82
	300	3	69.95	0.91	82.21	1.43
150	**295**	3	70.58	2.13	80.22	2.25
	296	3	68.28	1.40	77.64	1.20
	300	3	68.39	0.97	81.92	0.64
200	**295**	3	81.01	1.06	73.31	1.00
	296	3	81.95	1.59	75.47	1.28
	300	3	78.26	1.05	71.49	1.29

The final attempt to develop a DSC quantification method based on independent calibration curves involved the preparation of PTT samples in an amorphous phase. This was obtained by melting the samples in their aluminium pans either in a muffle or in an oven for a given time and then quenching them with liquid nitrogen. The very low temperatures of liquid nitrogen (around -200 °C) allowed an extremely rapid sample cooling. This process, known as *quenching,* allowed the samples to solidify in an amorphous phase after having erased their thermal history by melting them.

The influence of the temperature and time of the heat treatment and the time of the quenching step with liquid nitrogen were evaluated. The heat treatment was performed at 245, 265 and 300 °C, immerging pans in the liquid nitrogen for 1, 3, 5 or 10 minutes. Several combinations of heating temperature, heating time and quenching time were tested. After quenching, the samples were analysed via DSC going from 0 °C to 300 °C with a heating rate of 20 °C/minute.

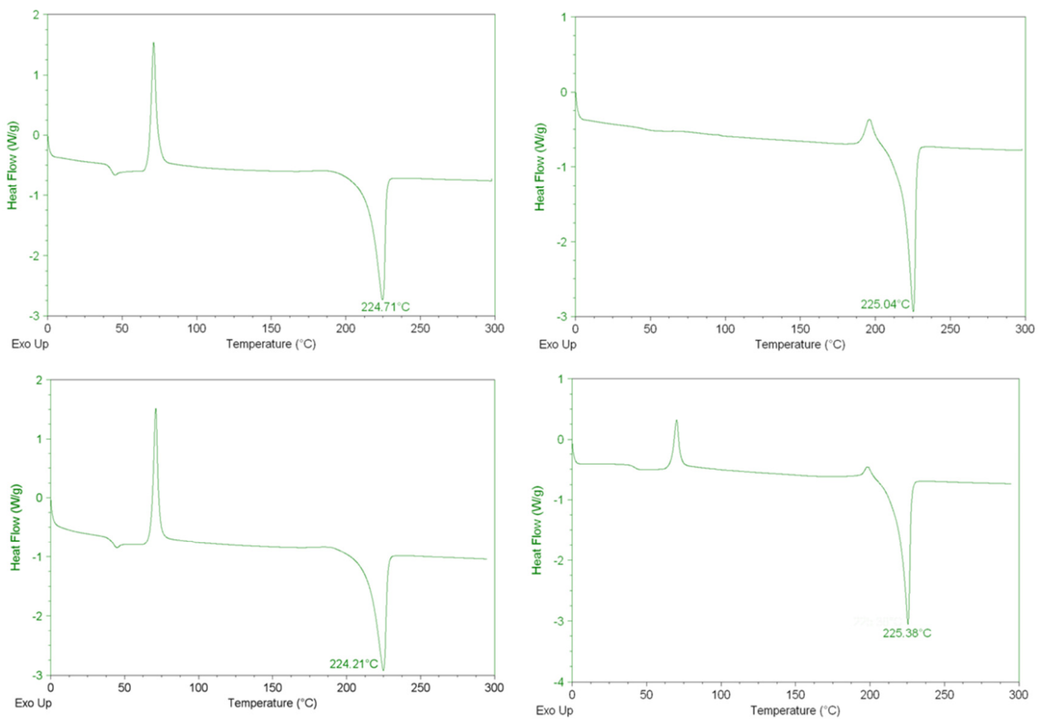


Figure 21: Melting peak areas for sample **317** heat-treated and quenched at various conditions. From left to right, top to bottom: 300 °C 10 min, N_2 1 min; 265 °C 3 min, N_2 1 min; 245 °C 10 min, N_2 5min; 235 °C 10 min, N_2 10 min.

Depending on the temperature, two types of heating system were used, muffle (T > 260 °C) and ventilated oven. Compared to the previous methods, in the case of quenching the glass transition and the cold crystallisation peak of PTT became much easier to identify. The shape of the PTT melting peaks depended on the combination of parameters used: in some cases the peaks were sharp and well defined, while in others they were broad and misshaped (Figure 21). The enthalpies also seemed to highly depend on the used conditions. The repeatability of the measured enthalpies of fusion was checked by performing 3 or 5 replicates for each combination of heating temperature/heating time/quenching time used and comparing the obtained values. These results are reported in Table 41, expressed in terms of coefficients of variation. They showed that the use of lower temperatures for the heat-treatment step corresponded to an improvement in repeatability. The quenching conditions showing the best results were normally also the ones showing the best peak shape.

Table 41: Influence of different heat treatments and quenching times on PTT and PET enthalpies of fusion.

JRC code	Composition	Heating system	T ºC	Heating time min	Quenching time min	Repl.	Enthalpy J/g	SD J/g	CV %
295	PTT	muffle	300	10	1	3	69.23	2.28	3.30
296	PTT	muffle	300	10	1	3	65.20	1.97	3.02
300	PTT	muffle	300	10	1	3	65.32	1.67	2.47
316	PET	muffle	300	10	1	3	51.16	3.03	5.93
298	PTT-PET	muffle	300	10	1	3	31.56	1.03	3.28
302	PTT-PET	muffle	300	10	1	3	38.07	0.50	1.32
309	PTT-PET	muffle	300	10	1	3	40.36	0.38	0.94
311	PTT-PET	muffle	300	10	1	3	35.26	1.16	3.30
312	PTT-PET	muffle	300	10	1	3	30.40	0.53	1.73
316	PET	muffle	300	10	1	5	41.26	1.64	3.98
317	PTT	muffle	300	10	1	5	54.92	1.53	2.79
311	PTT-PET	muffle	300	10	1	5	31.93	1.13	3.54
312	PTT-PET	muffle	300	10	1	4	26.14	1.31	5.01
316	PET	muffle	265	3	1	5	48.87	4.51	9.23
317	PTT	muffle	265	3	1	5	52.51	1.69	3.22
312	PTT-PET	muffle	265	3	1	5	30.72	1.21	3.93
296	PTT	oven	245	10	1	5	53.71	0.69	1.28
312	PTT-PET	oven	245	10	1	5	31.77	0.58	1.83
312	PTT-PET	oven	245	10	3	5	30.57	1.04	3.40
296	PTT	oven	245	10	5	5	53.58	0.53	0.98
317	PTT	oven	245	10	5	5	54.43	0.73	1.35
311	PTT-PET	oven	245	10	5	5	36.05	0.55	1.52
312	PTT-PET	oven	245	10	5	5	29.60	0.93	3.13
295	PTT	oven	245	10	10	5	54.83	0.82	1.49
296	PTT	oven	245	10	10	5	50.08	0.38	0.75
317	PTT	oven	245	10	10	5	54.43	0.73	1.35
295	PTT	oven	235	10	10	5	54.30	0.44	0.87
296	PTT	oven	235	10	10	5	50.99	0.72	1.40
316	PET	oven	235	10	10	5	55.21	0.46	0.84
317	PTT	oven	235	10	10	5	51.72	0.81	1.52
311	PTT-PET	oven	235	10	10	5	34.04	0.57	1.68
312	PTT-PET	oven	235	10	10	5	32.80	1.15	3.50

The first results were obtained maintaining for 10 minutes the samples, closed in the aluminium pans, at 300 °C in a muffle and then quenching them for 1 minute in liquid nitrogen before analysing them with DSC. Even though in several cases the coefficient of variations calculated on 3 and 5 replicates were higher than 3 %, the quantification of five binary mixtures PTT/PET (samples **298**, **302**, **309**, **311** and **312**) were carried using the above mentioned conditions for sample preparations (Table 42). The quantification was based on the integration of the PTT melting peak and on five calibration curves. The PET (sample **316**), selected to prepare the first four calibration curves, is the yarn used to produce the two blend samples **311** and **312**. Four different samples of pure PTT (**295**, **296**, **300** and **317**) were used to build up the first four calibration curves. The PTT yarn sample **317** was employed in the production of the two blend samples **311** and **312**. Finally, the fifth calibration was prepared using PTT and PET yarns manually separated from the binary mixture **312**. Both the blends under quantification and all the calibrations points were treated in the same way before the DSC analysis. As the correlation coefficient (R^2) using a linear interpolation was lower than 0.995, the calibration curves were fitted with a quadratic equation ($y = ax^2 + bx$, a parabolic curve crossing the axis origin).

Table 42: Quantification by DSC method
(samples and standards heat-treated at 300 °C for 10 min and quenched for 1 min in liquid nitrogen).

Calib. PET code	Calib. PTT code	a	b	R^2	JRC code	PTT (MS) %	Repl.	PTT (DSC) %	SD %	CV %	Bias %
316	**300**	0.0033	0.3407	0.9949	**298**	66.33	3	58.95	1.41	2.39	-7.38
					302	74.09	3	67.55	0.64	0.95	-6.54
					309	76.04	3	70.42	0.47	0.67	-5.62
					311	70.32	3	63.91	1.53	2.39	-6.41
					312	62.91	3	57.35	0.73	1.28	-5.56
316	**296**	0.0033	0.3158	0.9984	**298**	66.33	3	61.02	1.43	2.35	-5.31
					302	74.09	3	69.73	0.65	0.93	-4.36
					309	76.04	3	72.64	0.48	0.66	-3.40
					311	70.32	3	66.05	1.55	2.35	-4.27
					312	62.91	3	59.39	0.74	1.25	-3.52
316	**295**	0.0045	0.2375	0.9960	**298**	66.33	3	61.41	1.30	2.12	-4.92
					302	74.09	3	69.30	0.58	0.84	-4.79
					309	76.04	3	71.92	0.43	0.59	-4.12
					311	70.32	3	65.97	1.40	2.13	-4.35
					312	62.91	3	59.93	0.68	1.13	-2.98
316	**317**	0.0025	0.2993	0.9970	**311**	70.32	5	68.02	1.77	2.60	-2.30
					312	62.91	4	58.62	2.21	3.77	-4.29
Man. sep. from 312	**Man. sep. from 312**	0.0042	0.2342	0.9971	**311**	70.32	5	63.65	1.47	2.32	-6.67
					312	62.91	4	55.78	1.86	3.34	-7.13

In all cases, the quantification results were not acceptable because not accurate. In fact, the difference between the PTT content obtained by DSC and the reference value measured by manual separation (bias) was in the range -2.30 and -7.34 %. In has to be noted that the bias was always negative, thus indicating that most probably a systematic error was involved. Surprisingly, bad quantification results were obtained not only with generic calibration curves (the first three), but also when they were prepared with the PTT and PET yarns used in the production of the blends under evaluation (the fourth one) or with the PTT and PET yarns manually extracted from the blend under evaluation (the fifth one).

In order to understand the underlying causes of these bad results, thermogravimetric analyses were carried out on PTT sample **296** to evaluate if some degradation happened in the applied heat treatment conditions. The first experiment, a classical weight versus temperature curve was obtained increasing the temperature at 20 °C/min from room temperature to 900 °C. It exhibited a mass loss of about 1.0 % around 300 °C (Figure 22). A second experiment was recorded using the following temperature programme: RT – 50 °C/min – 300 °C (20 min). In this case the mass loss of sample **296** after 10 minutes at 300 °C was 4.3 % (Figure 23 and Table 43).

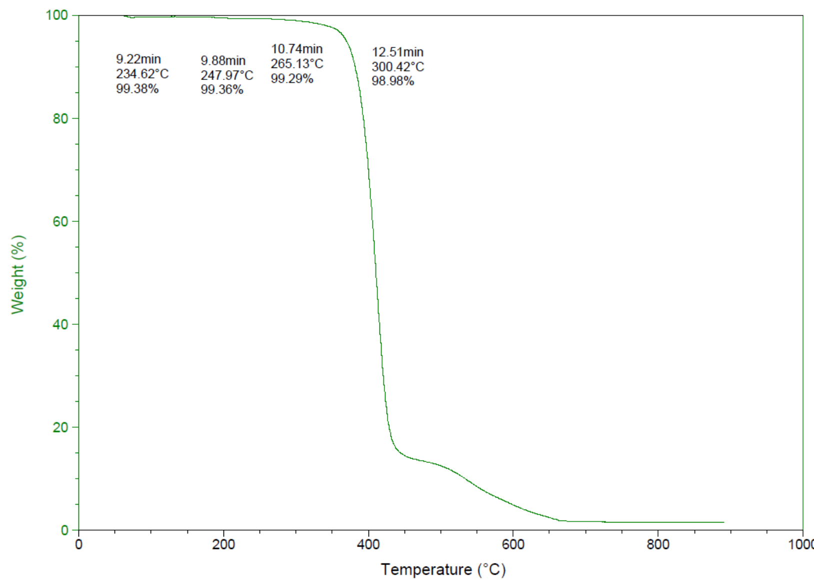


Figure 22: TGA analysis of sample **296**.

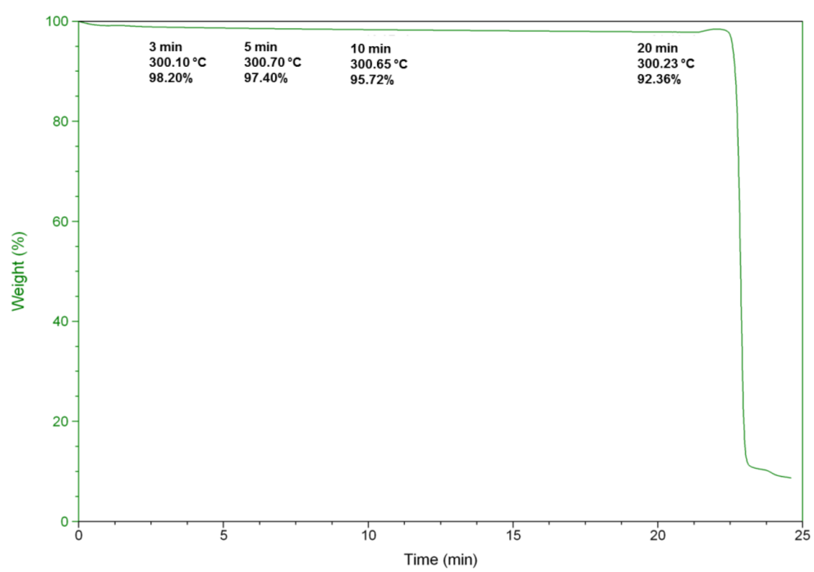


Figure 23: TGA analysis of sample **296** heat treated at 300 °C for 20 min.

Table 43: Mass loss of pure PTT (sample **296**) due to heat treatment.

Heating time min	Mass loss at 235 ºC %	Mass loss 245 ºC %	Mass loss 265 ºC %	Mass loss 300 ºC %
3	0.31	1.37	1.40	1.80
5	0.70	1.52	2.20	2.60
10	0.97	1.81	2.93	4.28
20	1.41	2.19	3.71	7.64

To avoid the partial mass loss of samples, the following tests were performed heating at 265 °C for 3 minutes and then quenching for 1 minute (Table 40). In this case, the coefficients of variation of the measured enthalpies of fusion were larger than in the previous case, in the range 2 – 9 %. Again, thermogravimetric analysis showed a mass loss of 0.7 % around 265 °C, when measured during the temperature scan, and of 1.4 % after heat treatment at 265 °C for 3 minutes.

Considering that the principal aim of the quenching method was to erase the thermal history of PTT fibres and obtaining them in an amorphous phase and in order to avoid any degradation of samples, further experiments were made heat treating at 245 °C for 10 minutes and then quenching for 1, 3, 5 or 10 minutes (Table 41). The selected temperature allowed melting PTT fibres but not PET ones. Apart from two exceptions, the coefficients of variation for the enthalpies of fusion were all lower than 2 %. In this case, a mass loss of about 0.6 % around 245 °C was recorded during the temperature scan, and of 1.8 % after heat treatment at 245 °C for 10 minutes.

The quantification of PTT/PET binary mixtures **311** and **312** were carried out in these conditions, using the PTT and PET yarns used in the production of the blends (sample **317** and **316**, respectively). The results showed a very low bias for sample **311** but still an unacceptable accuracy for sample **312** (Table 44).

Table 44: Quantification by DSC method
(samples and standards heat-treated at 245 °C for 10 min and quenched for 1 min in liquid nitrogen).

Calib. PET code	Calib. PTT code	a	b	R^2	JRC code	PTT (MS) %	Repl.	PTT (DSC) %	SD %	CV %	Bias %
316	**317**	0.0010	0.4442	0.9991	**311**	70.32	5	70.09	0.94	1.34	-0.23
					312	62.91	5	58.83	1.65	2.81	-4.08

Following the encouraging results, the last conditions tested were heat treatment at 235 °C for 10 minutes and then quenching for 10 minutes. The mass losses measured during the temperature scan and after heat treatment were 0.6 % and 1.0 %, respectively, and the coefficients of variation for PTT enthalpy of fusion were in general lower than 2 %.

Several quantification tests were performed using the three heat treatments and quenching conditions reported in Table 45. Binary mixtures PTT/PET were quantified by using independent different calibration curves built up with various types of PTT (**295**, **296** and **317**) and one PET (**316**). Heat treatments were done either at 245 or 235 °C; both temperatures are between the melting point of PTT (around 225 °C) and PET (around 250 °C). The PTT quantification was based both on the integration of the PTT and the PET peaks, and the results can be seen in Tables 46 - 51. The standards for the calibration curves were heat treated and quenched in the same conditions used for samples under evaluation. The calibration curves were always linear (R^2 higher than 0.995), apart from the case of heat treatment at 245 °C and the integration of the PTT melting peak.

Table 45: Heat treatments and quenching conditions used in the following quantification tests.

T ºC	Heating time min	Quenching time min
245	10	5
235	10	10
235	20	10

Table 46: Quantification by DSC method based on PTT peak integration.
Heat treatment 245 °C (10 min); quenching with liquid nitrogen (5 min).

Calib. PET code	Calib. PTT code	JRC code	PTT (MS) %	a	b	R^2	Repl.	PTT (DSC) %	SD %	CV %	Bias %
		298	66.33				5	64.63	1.08	1.67	**-1.70**
		302	74.09				5	70.43	2.66	3.78	**-3.66**
316	**317**	**309**	76.04	0.0019	0.3349	0.9984	5	78.98	2.01	2.54	**2.94**
		311	70.32				5	71.39	1.20	1.68	**1.07**
		312	62.91				5	65.87	1.23	1.87	**2.96**
		298	66.33				5	61.88	1.12	1.81	**-4.45**
		302	74.09				5	67.94	2.80	4.12	**-6.15**
316	**296**	**309**	76.04	0.0013	0.3976	0.9975	5	76.96	2.14	2.78	**0.92**
		311	70.32				5	69.29	0.78	1.13	**-1.03**
		312	62.91				5	63.17	1.28	2.03	**0.26**
		298	66.33				5	59.86	1.09	1.82	**-6.47**
		302	74.09				5	65.72	2.70	4.11	**-8.37**
316	**295**	**309**	76.04	0.0014	0.4103	0.9982	5	74.44	2.06	2.77	**-1.60**
		311	70.32				5	67.03	0.75	1.12	**-3.29**
		312	62.91				5	61.11	1.24	2.03	**-1.80**

Table 47: Quantification by DSC method based on PTT peak integration. Heat treatment 235 °C (10 min); quenching with liquid nitrogen (10 min).

Calib. PET code	Calib. PTT code	JRC code	PTT (MS) %	m	R^2	Repl.	PTT (DSC) %	SD %	CV %	Bias %
		298	66.33			5	62.55	1.96	3.13	-3.78
		302	74.09			5	75.73	0.55	0.73	1.64
316	317	309	76.04	0.5104	0.9990	5	75.09	0.82	1.09	-0.95
		311	70.32			5	66.69	1.12	1.68	-3.63
		312	62.91			5	64.26	2.25	3.50	1.35
		298	66.33			5	62.63	1.97	3.15	-3.70
		302	74.09			5	75.83	0.56	0.74	1.74
316	296	309	76.04	0.5097	0.9985	5	75.19	0.83	1.10	-0.85
		311	70.32			5	66.78	1.12	1.68	-3.54
		312	62.91			5	64.34	2.25	3.50	1.43
		298	66.33			5	61.26	1.92	3.13	-5.07
		302	74.09			5	74.01	0.30	0.41	-0.08
316	295	309	76.04	0.5211	0.9951	5	73.77	0.52	0.70	-2.27
		311	70.32			5	65.32	1.10	1.68	-5.00
		312	62.91			5	62.94	2.20	3.50	0.03

Table 48: Quantification by DSC method based on PTT peak integration. Heat treatment 235 °C (20 min); quenching with liquid nitrogen (10 min).

Calib. PET code	Calib. PTT code	JRC code	PTT (MS) %	m	R^2	Repl.	PTT (DSC) %	SD %	CV %	Bias %
		302	74.09			5	72.76	0.83	1.14	-1.33
316	317	309	76.04	0.5295	0.9994	5	73.53	1.20	1.63	-2.51
		311	70.32			6	68.97	0.67	0.97	-1.35
		312	62.91			6	60.56	0.92	1.52	-2.35
		302	74.09			5	73.33	0.84	1.15	-0.76
316	296	309	76.04	0.5254	0.9964	5	74.11	1.21	1.63	-1.93
		311	70.32			6	69.51	0.67	0.96	-0.81
		312	62.91			6	61.03	0.93	1.52	-1.88
		302	74.09			5	74.15	0.85	1.15	0.06
316	295	309	76.04	0.5196	0.9973	5	74.93	1.23	1.64	-1.11
		311	70.32			6	70.29	0.68	0.97	-0.03
		312	62.91			6	61.71	0.94	1.52	-1.20

Table 49: Quantification by DSC method based on PET peak integration. Heat treatment 245 °C (10 min); quenching with liquid nitrogen (5 min).

Calib. PET code	Calib. PTT code	JRC code	PTT (MS) %	m	R^2	Repl.	PTT (DSC) %	SD %	CV %	Bias %
		309	76.04	0.3957	0.9984	5	68.47	2.14	3.13	-7.57
316	317	311	70.32			5	72.60	0.74	1.02	2.28
		312	62.91			4	68.59	2.37	3.46	5.68
		309	76.04	0.3994	0.9966	5	68.95	2.06	2.99	-7.09
316	296	311	70.32			5	72.94	0.71	0.97	2.62
		312	62.91			4	69.07	2.28	3.30	6.16
		309	76.04	0.4112	0.9958	5	70.39	2.03	2.88	-5.65
316	295	311	70.32			5	74.30	0.70	0.94	3.98
		312	62.91			4	70.50	2.25	3.19	7.59

Table 50: Quantification by DSC method based on PET peak integration.
Heat treatment 235 °C (10 min); quenching with liquid nitrogen (10 min).

Calib. PET code	Calib. PTT code	JRC code	PTT (MS) %	m	R^2	Repl.	PTT (DSC) %	SD %	CV %	Bias %
316	317	**298**	66.33	0.5571	0.9998	5	69.34	0.81	1.17	3.01
		302	74.09			5	82.96	0.78	0.94	8.87
		309	76.04			5	75.92	0.62	0.82	-0.12
		311	70.32			5	72.57	1.09	1.50	2.25
		312	62.91			5	69.62	1.86	2.67	6.71
316	296	**298**	66.33	0.5574	0.9997	5	69.35	0.81	1.17	3.02
		302	74.09			5	82.97	0.78	0.94	8.88
		309	76.04			5	75.93	0.62	0.82	-0.11
		311	70.32			5	72.59	1.09	1.50	2.27
		312	62.91			5	69.63	1.85	2.66	6.72
316	295	**298**	66.33	0.5568	0.9995	5	69.32	0.81	1.17	2.99
		302	74.09			5	82.95	0.78	0.94	8.86
		309	76.04			5	75.91	0.62	0.82	-0.13
		311	70.32			5	72.56	1.09	1.50	2.24
		312	62.91			5	69.60	1.86	2.67	6.69

Table 51: Quantification by DSC method based on PET peak integration.
Heat treatment 235 °C (20 min); quenching with liquid nitrogen (10 min).

Calib. PET code	Calib. PTT code	JRC code	PTT (MS) %	m	R^2	Repl.	PTT (DSC) %	SD %	CV %	Bias %
316	317	**302**	74.09	0.5832	0.9996	5	79.93	0.35	0.44	5.84
		309	76.04			5	75.76	0.44	0.58	-0.28
		311	70.32			6	70.88	0.34	0.48	0.56
		312	62.91			6	64.39	0.54	0.84	1.48
316	296	**302**	74.09	0.5807	0.9996	5	79.85	0.35	0.44	5.76
		309	76.04			5	75.65	0.44	0.58	-0.39
		311	70.32			6	70.76	0.34	0.48	0.44
		312	62.91			6	64.24	0.54	0.84	1.33
316	295	**302**	74.09	0.5789	0.9998	5	79.77	0.35	0.44	5.68
		309	76.04			5	75.56	0.44	0.58	-0.48
		311	70.32			6	70.64	0.34	0.48	0.32
		312	62.91			6	64.10	0.54	0.84	1.19

Results were evaluated in terms of *bias*, which gives the difference between the PTT contents obtained via DSC and the values that are considered as reference values, obtained through manual separation. In general, the trueness was better when the samples were quantified based on PTT enthalpy of fusion. Comparing the various sample preparations, the best results were achieved heat-treating the samples and the standards at 235 °C for 20 minutes followed by a 10 minutes immersion in liquid nitrogen. Nevertheless, the bias values were still too high as in several cases higher than 1 %; therefore, the method was considered not acceptable as not enough accurate.

The conclusion based on the entire set of data just presented is that it is impossible to develop an accurate DSC quantitative method that makes use of independent calibration curves, meaning calibration curves prepared using whatever PTT and PET samples available in a laboratory. Consequently, a DSC method using calibration curves built up with PTT and PET yarns separated from each fabric sample under evaluation is the only alternative to obtain accurate results.

The following part of the experimentation was dedicated to the optimisation of both the integration step and the DSC method conditions together with the evaluation of the in-house method repeatability.

Initially, the quantification of four PTT/PET binary mixtures (samples **302**, **309**, **311** and **312**) were carried out using both multipoint calibration curves and single point calibration based on PTT peak in three different days, with the following DSC temperature program 0 °C – 10 °C/min – 300 °C, using five different kinds of integration. The linear integration consisted in connecting the two baselines before and after the melting peak, the parallel integration was obtained using a line parallel to the x axis, the linear fixed range integration foresaw the selection for each set of analysis (sample plus its calibration curve) of an optimal range of temperature to be used in the integration of all thermograms for that set of analysis, the sigmoidal fixed range integration used a sigmoidal curve connecting the two baselines before and after the melting peak in a fixed range of temperatures optimised for each set of analysis, and finally the linear perpendicular drop fixed range integration allowed to separate two consecutive peaks by a line perpendicular to the x axis (Figure 24).

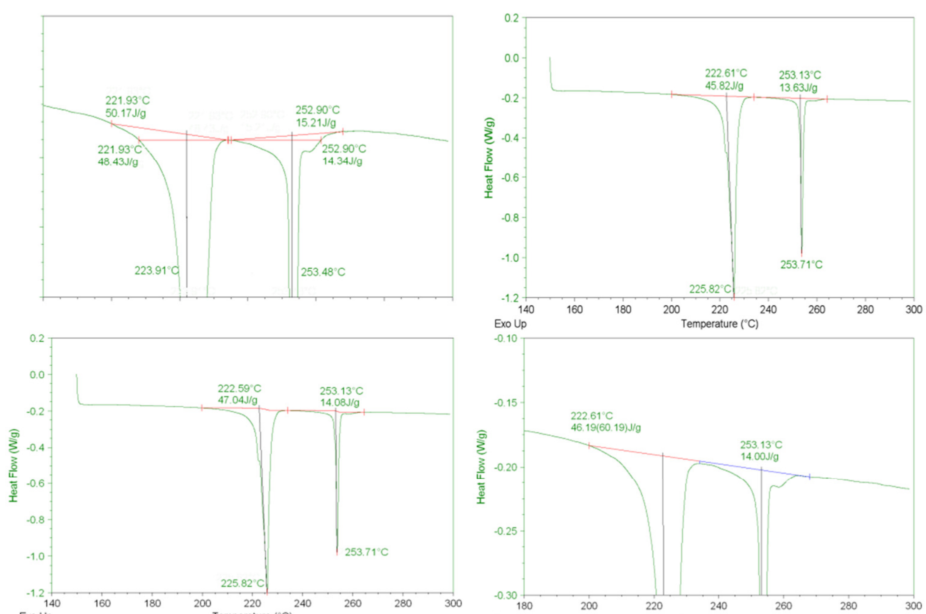


Figure 24: Various types of integrations (sample **311**).From left to right, top to bottom: linear and parallel, linear fixed range, sigmoidal fixed range and perpendicular drop fixed range.

From the thermograms it can be seen that the changes on the type of integration implied also a considerable change on the enthalpies values. Three replicates were analysed on day one and four replicates on days two and three. The results in terms of PTT content, standard deviation, coefficient of variation and bias obtained applying the five types of integrations are reported in Tables 52 and 53.

Table 52: Quantification by DSC method applying various types of integration. (multipoint calibration curves based on PTT peak)

				Linear integration							
	JRC code	PTT (MS) %	Repl.	a	b	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09	3			0.6198	0.9952	74.81	0.93	1.24	0.72
	309	76.04	3			0.5841	0.9973	75.70	2.79	3.69	-0.34
	311	70.32	3			0.5984	0.9955	71.20	2.73	3.83	0.88
	312	62.91	3			0.5989	0.9946	62.74	1.43	2.28	-0.17
Day 2	302	74.09	4	0.0013	0.4868		0.9999	76.59	0.57	0.74	2.50
	309	76.04	4	0.0010	0.5307		0.9978	77.01	0.93	1.21	0.97
	311	70.32	4			0.6153	0.9983	72.58	0.42	0.58	2.26
	312	62.91	4			0.5942	0.9983	65.13	2.23	3.42	2.22
Day 3	302	74.09	4	0.0012	0.4940		0.9921	77.63	0.65	0.84	3.54
	309	76.04	4			0.6085	0.9977	77.03	0.13	0.17	0.99
	311	70.32	4			0.6251	0.9974	71.88	0.27	0.38	1.56
	312	62.91	4			0.5920	0.9968	64.07	0.54	0.84	1.16

				Parallel integration							
	JRC code	PTT (MS) %	Repl.	a	b	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09	3			0.5821	0.9945	74.92	0.95	1.27	0.83
	309	76.04	3			0.5677	0.9949	74.36	0.75	1.01	-1.68
	311	70.32	3			0.5928	0.9955	72.02	3.17	4.40	1.70
	312	62.91	3			0.5991	0.9946	64.10	1.37	2.14	1.19
Day 2	302	74.09	4	0.0016	0.4293		0.9995	77.18	0.51	0.66	3.09
	309	76.04	4	0.0009	0.4952		0.9980	78.71	1.01	1.28	2.67
	311	70.32	4			0.5852	0.9975	72.82	0.45	0.62	2.50
	312	62.91	4			0.5633	0.9960	65.07	2.42	3.72	2.16
Day 3	302	74.09	4	0.0015	0.4438		0.9935	77.28	0.58	0.75	3.19
	309	76.04	4			0.5748	0.9956	77.54	0.11	0.14	1.50
	311	70.32	4			0.5909	0.9960	72.55	0.11	0.15	2.23
	312	62.91	4			0.5582	0.9950	64.29	0.46	0.72	1.38

				Linear fixed range integration							
	JRC code	PTT (MS) %	Repl.	a	b	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09	3			0.5901	0.9990	75.29	0.85	1.13	1.20
	309	76.04	3			0.5818	0.9954	74.14	1.47	1.98	-1.90
	311	70.32	3			0.6207	0.9980	70.85	3.12	4.40	0.53
	312	62.91	3			0.6219	0.9973	64.45	0.55	0.85	1.54
Day 2	302	74.09	4			0.5686	0.9958	78.61	0.58	0.74	4.52
	309	76.04	4			0.5935	0.9983	77.05	0.47	0.61	1.01
	311	70.32	4			0.6057	0.9990	73.00	0.4	0.55	2.68
	312	62.91	4			0.5822	0.9992	65.95	2.39	3.62	3.04
Day 3	302	74.09	4	0.0014	0.4815		0.9968	77.91	1.15	1.48	3.82
	309	76.04	4			0.6029	0.9974	76.35	0.59	0.77	0.31
	311	70.32	4			0.6088	0.9963	73.20	0.06	0.08	2.88
	312	62.91	4			0.5835	0.9961	64.12	0.16	0.25	1.21

	JRC code	PTT (MS) %	Repl.	a	b	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
				Sigmoidal fixed range integration							
Day 1	**302**	74.09	3			0.5919	0.9994	75.46	0.85	1.13	**1.37**
	309	76.04	3			0.5890	0.9950	73.47	1.52	2.07	**-2.57**
	311	70.32	3			0.6400	0.9992	70.01	3.08	4.40	**-0.31**
	312	62.91	3			0.6380	0.9981	64.35	0.08	0.12	**1.44**
Day 2	**302**	74.09	4			0.6051	0.9999	76.88	0.48	0.62	**2.79**
	309	76.04	4			0.6047	0.9997	76.75	0.72	0.94	**0.71**
	311	70.32	4			0.6207	0.9986	72.85	0.33	0.45	**2.53**
	312	62.91	4			0.5932	0.9990	66.61	2.31	3.47	**3.70**
Day 3	**302**	74.09	4			0.6169	0.9966	76.07	2.03	2.67	**1.98**
	309	76.04	4			0.6097	0.9998	76.18	1.66	2.18	**0.14**
	311	70.32	4	-0.0004	0.6538		0.9951	73.13	0.16	0.22	**2.81**
	312	62.91	4			0.5948	0.9964	64.65	0.21	0.32	**1.74**

	JRC code	PTT (MS) %	Repl.	a	b	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
				Perpendicular drop fixed range integration							
Day 1	**302**	74.09	3			0.6059	0.9945	74.73	0.70	0.94	**0.64**
	309	76.04	3			0.5860	0.9949	77.79	1.26	1.62	**1.75**
	311	70.32	3			0.6365	0.9977	70.93	3.02	4.26	**0.61**
	312	62.91	3			0.6359	0.9967	64.61	0.38	0.59	**1.70**
Day 2	**302**	74.09	4	-0.0017	0.7376		0.9991	79.38	0.42	0.53	**5.29**
	309	76.04	4			0.6017	0.9998	76.35	0.72	0.94	**0.31**
	311	70.32	4			0.6128	0.9972	73.12	0.39	0.53	**2.80**
	312	62.91	4			0.5949	0.9974	66.41	2.59	3.90	**3.50**
Day 3	**302**	74.09	4	-0.0006	0.6759		0.9959	76.29	0.78	1.02	**2.20**
	309	76.04	4			0.6082	0.9989	76.13	0.78	1.02	**0.09**
	311	70.32	4	-0.0005	0.6563		0.9889	73.10	0.53	0.73	**2.78**
	312	62.91	4	-0.0004	0.6311		0.9945	63.58	0.22	0.35	**0.67**

Table 53: Quantification by DSC method applying various types of integration. (single point calibration based on PTT peak)

	JRC code	PTT (MS) %	Repl.	Linear integration: PTT (DSC) %	SD %	CV %	Bias %	Parallel integration: PTT (DSC) %	SD %	CV %	Bias %
Day 1	**302**	74.09	3	72.10	0.89	1.23	**-1.99**	73.50	0.93	1.27	**-0.59**
	309	76.04	3	74.77	2.76	3.69	**-1.27**	70.71	0.72	1.02	**-5.33**
	311	70.32	3	70.93	2.72	3.83	**0.61**	71.31	3.14	4.40	**0.99**
	312	62.91	3	63.07	1.44	2.28	**0.16**	63.92	1.37	2.14	**1.01**
Day 2	**302**	74.09	4	73.13	0.64	0.88	**-0.96**	72.30	0.58	0.80	**-1.79**
	309	76.04	4	74.52	1.01	1.36	**-1.52**	76.05	1.10	1.45	**0.01**
	311	70.32	4	72.65	0.42	0.58	**2.33**	72.60	0.45	0.62	**2.28**
	312	62.91	4	64.79	2.22	3.43	**1.88**	64.28	2.39	3.72	**1.37**
Day 3	**302**	74.09	4	71.97	0.69	0.96	**-2.12**	71.85	0.65	0.90	**-2.24**
	309	76.04	4	75.79	0.12	0.16	**-0.25**	75.81	0.11	0.15	**-0.23**
	311	70.32	4	71.50	0.27	0.38	**1.18**	71.69	0.11	0.15	**1.37**
	312	62.91	4	63.74	0.54	0.85	**0.83**	63.13	0.45	0.71	**0.22**

	JRC code	PTT (MS) %	Repl.	Linear fixed range integration PTT (DSC) %	SD %	CV %	Bias %	Sigmoidal fixed range integration PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09	3	73.79	0.83	1.12	-0.30	74.43	0.84	1.13	0.34
	309	76.04	3	71.04	1.41	1.98	-5.00	70.62	1.47	2.08	-5.42
	311	70.32	3	71.09	3.13	4.40	0.77	71.03	3.13	4.41	0.71
	312	62.91	3	65.37	0.56	0.86	2.46	65.90	0.09	0.14	2.99
Day 2	302	74.09	4	78.22	0.58	0.74	4.13	76.94	0.49	0.64	2.85
	309	76.04	4	76.10	0.46	0.60	0.06	76.74	0.72	0.94	0.70
	311	70.32	4	73.00	0.40	0.55	2.68	72.85	0.33	0.45	2.53
	312	62.91	4	66.34	2.41	3.63	3.43	67.65	2.35	3.47	4.74
Day 3	302	74.09	4	74.40	1.30	1.75	0.31	74.99	2.12	2.83	0.90
	309	76.04	4	74.43	0.59	0.79	-1.61	76.53	1.67	2.18	0.49
	311	70.32	4	72.16	0.06	0.08	1.84	72.70	0.15	0.21	2.38
	312	62.91	4	64.66	0.17	0.26	1.75	65.83	0.22	0.33	2.92

	JRC code	PTT (MS) %	Repl.	Perpendicular drop fixed range integration PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09	3	75.20	0.71	0.94	1.11
	309	76.04	3	75.07	1.22	1.63	-0.97
	311	70.32	3	72.92	3.11	4.26	2.60
	312	62.91	3	67.01	0.39	0.58	4.10
Day 2	302	74.09	4	81.99	0.44	0.54	7.90
	309	76.04	4	76.45	0.72	0.94	0.41
	311	70.32	4	73.12	0.39	0.53	2.80
	312	62.91	4	68.25	2.66	3.90	5.34
Day 3	302	74.09	4	77.22	0.74	0.96	3.13
	309	76.04	4	74.87	0.77	1.03	-1.17
	311	70.32	4	73.36	0.50	0.68	3.04
	312	62.91	4	66.55	0.22	0.33	3.64

In each day the four samples and their corresponding multipoint calibration curves and single point calibrations were analysed and the thermograms integrated using five different approaches. The outcome of these tests highlighted the strong influence that the integration approach used can have on final results. Considering the biases obtained, in the case of multipoint calibration curves, the parallel integration showed the worst results and the linear integration the best ones. In the case of single point calibrations, the linear integration was confirmed to be the approach giving the best results, while the worst were shown by the perpendicular drop fixed range integration. Almost all results seemed to point out that some problems had occurred on day two.

Similar analyses were carried out also in DuPont laboratories on the same samples in three days applying a slightly different DSC program (-20 °C – 10 °C/min – 280 °C) and three types of integration: the linear fixed range, the sigmoidal fixed range and the linear perpendicular drop. Three replicates were analysed on day one and four replicates on days two and three. Results are reported in Tables 54 and 55.

Table 54: DuPont's quantification by DSC method applying various types of integration. (multipoint calibration curves based on PTT peak)

				Linear fixed range integration							
	JRC code	PTT (MS) %	Repl.	a	b	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09	3	0.0030	0.3477		0.9888	79.23	1.2	1.51	5.14
	309	76.04	3	0.0016	0.4975		0.9959	78.83	0.41	0.52	2.79
	311	70.32	3	0.0014	0.5311		0.9962	72.42	0.74	1.02	2.10
	312	62.91	3			0.6277	0.9961	64.62	0.35	0.54	1.71
Day 2	302	74.09	4	0.0016	0.4536		0.9982	77.21	2.16	2.80	3.12
	309	76.04	4			0.6033	0.9964	78.85	0.64	0.81	2.81
	311	70.32	4			0.6218	0.9980	71.60	0.74	1.03	1.28
	312	62.91	4			0.6013	0.9954	64.27	0.51	0.79	1.36
Day 3	302	74.09	4	0.0023	0.4027		0.9919	79.07	0.28	0.35	4.98
	309	76.04	4	0.0011	0.4891		0.9992	80.21	0.66	0.82	4.17
	311	70.32	4			0.5882	0.9956	71.86	2.98	4.15	1.54
	312	62.91	4			0.6067	0.9955	63.66	0.9	1.41	0.75

				Sigmoidal fixed range integration					
	JRC code	PTT (MS) %	Repl.	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09	3	0.6559	0.9982	78.16	0.78	1.00	4.07
	309	76.04	3	0.6742	0.9986	78.41	0.47	0.60	2.37
	311	70.32	3	0.7053	0.9979	71.77	0.61	0.85	1.45
	312	62.91	3	0.6566	0.9998	66.26	0.37	0.56	3.35
Day 2	302	74.09	4	0.6111	0.9990	77.57	2.07	2.67	3.48
	309	76.04	4	0.6324	0.9990	78.93	0.58	0.73	2.89
	311	70.32	4	0.6444	0.9992	72.41	0.64	0.88	2.09
	312	62.91	4	0.6110	0.9969	65.87	0.39	0.59	2.96
Day 3	302	74.09	4	0.6328	0.9979	79.29	0.31	0.39	5.20
	309	76.04	4	0.6162	0.9977	81.66	0.82	1.00	5.62
	311	70.32	4	0.6153	0.9985	72.85	2.62	3.60	2.53
	312	62.91	4	0.6223	0.9980	64.97	0.22	0.34	2.06

				Linear perpendicular drop integration							
	JRC code	PTT (MS) %	Repl.	a	b	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09	3	-0.0015	0.7714		0.9956	75.38	0.32	0.42	1.29
	309	76.04	3			0.6388	0.9955	76.41	0.36	0.47	0.37
	311	70.32	3			0.6440	0.9975	72.59	0.91	1.25	2.27
	312	62.91	3			0.6749	0.9985	63.38	0.17	0.27	0.47
Day 2	302	74.09	4	-0.0017	0.7762		0.9966	73.41	3.66	4.99	-0.68
	309	76.04	4	-0.0006	0.6691		0.9961	76.65	0.91	1.19	0.61
	311	70.32	4			0.6409	0.9966	71.91	1.57	2.18	1.59
	312	62.91	4			0.6205	0.9976	65.00	0.82	1.26	2.09
Day 3	302	74.09	4	-0.0018	0.7991		0.9977	75.85	0.4	0.53	1.76
	309	76.04	4			0.5908	0.9974	81.10	0.8	0.99	5.06
	311	70.32	4	-0.0007	0.6579		0.9962	70.61	1.26	1.78	0.29
	312	62.91	4	-0.0007	0.6892		0.9969	62.92	0.28	0.45	0.01

The experiments carried out in DuPont's laboratories consistently indicated that, among the three approaches of integration tested, the sigmoidal fixed range was the worst one. The best results were obtained, in the case of multipoint calibration curves, with the linear perpendicular drop and, for the single point calibration, with the linear fixed range. DuPont results were particularly good when samples were quantified using a single point calibration and a linear fixed range integration. In fact, in these conditions nine biases out of twelve were lower than 1 %, indicating a good accuracy of the method. The quantification results obtained using single point calibration seemed to be particularly influenced by the type of integration.

Table 55: DuPont's quantification by DSC method applying various types of integration. (single point calibration based on PTT peak)

	JRC code	PTT (MS) %	Repl.	Linear fixed range integration PTT (DSC) %	SD %	CV %	Bias %	Sigmoidal fixed range integration PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09	3	73.58	1.56	2.12	-0.51	78.36	0.78	1.00	4.27
	309	76.04	3	76.29	0.47	0.62	0.25	78.40	0.47	0.60	2.36
	311	70.32	3	70.60	0.84	1.19	0.28	73.13	0.62	0.85	2.81
	312	62.91	3	63.96	0.35	0.55	1.05	66.53	0.37	0.56	3.62
Day 2	302	74.09	4	73.00	2.48	3.40	-1.09	76.50	2.04	2.67	2.41
	309	76.04	4	77.59	0.63	0.81	1.55	79.49	0.58	0.73	3.45
	311	70.32	4	71.22	0.74	1.04	0.90	73.29	0.65	0.89	2.97
	312	62.91	4	62.69	0.50	0.80	-0.22	64.37	0.38	0.59	1.46
Day 3	302	74.09	4	74.56	0.35	0.47	0.47	78.85	0.31	0.39	4.76
	309	76.04	4	76.80	0.73	0.95	0.76	80.85	0.81	1.00	4.81
	311	70.32	4	71.28	2.95	4.14	0.96	73.66	2.65	3.60	3.34
	312	62.91	4	63.03	0.89	1.41	0.12	65.05	0.22	0.34	2.14

	JRC code	PTT (MS) %	Repl.	Perpendicular drop fixed range integration PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09	3	78.41	0.28	0.36	4.32
	309	76.04	3	76.53	0.36	0.47	0.49
	311	70.32	3	72.17	0.94	1.30	1.85
	312	62.91	3	62.00	0.17	0.27	-0.91
Day 2	302	74.09	4	78.30	3.16	4.04	4.21
	309	76.04	4	79.53	0.91	1.14	3.49
	311	70.32	4	73.73	1.38	1.87	3.41
	312	62.91	4	65.43	0.82	1.25	2.52
Day 3	302	74.09	4	80.48	0.33	0.41	6.39
	309	76.04	4	79.46	0.78	0.98	3.42
	311	70.32	4	72.45	1.18	1.63	2.13
	312	62.91	4	66.24	0.27	0.41	3.33

The evaluation of the whole group of thermograms allowed noticing that for same sample and calibration points the melting peaks of PTT and PET were partly overlapped. A further optimisation of the DSC method was then planned.

In order to reach an optimal separation between the PTT and PET melting peaks, thus facilitating their integration, the ramp of temperature was halved to 5 °C/min. At the same time, to keep the time of analysis relatively short and considering that the PTT's glass transition, occurring at approximately 50 °C, was not of interest, the initial temperature of the analysis was set at 150 °C. The amount of sample used for analyses was selected in the range 5 – 8 mg to allow the preparation of accurate calibration points and contemporaneously being a quantity that was still possible to close into the DSC pans. The nitrogen flow, which prevents any oxidative reaction, was kept at 50 ml/min. As already done in all previous experiments, in order to have contact between the samples under measurement and the inert atmosphere the lids of the sample pans were pierced with an extremely thin needle to produce three holes on each lid. Finally, the quantification was based on the integration of both PTT and PET peaks using multipoint calibration curves and on the integration of PTT peak using single point calibration. With these modifications the DSC method had a total run time of approximately 40 minutes.

The final SOP of the DSC method was tested on six days on four binary mixtures PTT/PET with various levels of PTT: sample **302** (74.09 %), **309** (76.04 %), **311** (70.32 %) and **312** (62.91 %). All these samples were manually separable, thus allowing comparison between the reference values and the DSC ones. In all cases four replicate analyses were performed. These experiments allowed the evaluation of the influence of the type of integration and of the repeatability. The types of integration that were allowed by the software were tested and compared. Results are reported in Tables 56 - 58.

Table 56: Quantification by the optimised DSC method applying various types of integration. (multipoint calibration curves based on PTT peak)

			Linear integration							
	JRC code	**PTT (MS) %**	**a**	**b**	**m**	**R^2**	**PTT (DSC) %**	**SD %**	**CV %**	**Bias %**
Day 1	**302**	74.09	0.0012	0.5175		0.9983	75.29	0.37	0.49	**1.20**
	309	76.04			0.6178	0.9977	76.18	0.51	0.67	**0.14**
	311	70.32			0.6098	0.9970	70.41	2.09	2.97	**0.09**
	312	62.91			0.6028	0.9985	64.32	0.50	0.78	**1.41**
Day 2	**302**	74.09	0.0011	0.5120		0.9981	75.48	0.80	1.06	**1.39**
	309	76.04	0.0003	0.5797		0.9945	76.63	1.40	1.83	**0.59**
	311	70.32			0.6341	0.9984	71.29	0.23	0.32	**0.97**
	312	62.91	0.0009	0.5979		0.9903	64.86	1.17	1.80	**1.95**
Day 3	**302**	74.09	0.0018	0.5364		0.9973	77.10	0.71	0.92	**3.01**
	309	76.04			0.7028	0.9964	73.19	1.16	1.58	**-2.85**
	311	70.32			0.7246	0.9992	68.69	1.03	1.50	**-1.63**
	312	62.91			0.7031	0.9950	68.37	0.48	0.70	**5.46**
Day 4	**302**	74.09	0.0012	0.5259		0.9995	73.54	0.68	0.92	**-0.55**
	309	76.04			0.6232	0.9977	75.47	0.81	1.07	**-0.57**
	311	70.32			0.6370	0.9968	70.77	0.60	0.85	**0.45**
	312	62.91			0.6344	0.9966	63.84	1.12	1.75	**0.93**
Day 5	**302**	74.09	0.0013	0.4923		0.9974	76.11	0.32	0.42	**2.02**
	309	76.04			0.6335	0.9963	76.84	0.42	0.55	**0.80**
	311	70.32			0.6518	0.9968	71.84	0.50	0.70	**1.52**
	312	62.91	0.0007	0.5784		0.9956	63.94	0.93	1.45	**1.03**
Day 6	**302**	74.09	0.0019	0.4495		0.9979	75.35	0.48	0.64	**1.26**
	309	76.04	0.0013	0.4972		0.9962	77.16	0.80	1.04	**1.12**
	311	70.32			0.6437	0.9991	70.99	0.75	1.06	**0.67**
	312	62.91	0.0017	0.4469		0.9979	63.88	1.36	2.13	**0.97**

			Parallel integration							
	JRC code	PTT (MS) %	a	b	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
Day 1	**302**	74.09	0.0015	0.4511		0.9987	76.09	0.53	0.70	**2.00**
	309	76.04			0.5838	0.9961	76.44	0.36	0.47	**0.40**
	311	70.32			0.5813	0.9977	70.26	1.91	2.72	**-0.06**
	312	62.91	0.0008	0.5138		0.9994	64.68	0.51	0.79	**1.77**
Day 2	**302**	74.09	0.0016	0.4420		0.9985	75.15	0.64	0.85	**1.06**
	309	76.04	0.0006	0.5236		0.9950	77.08	1.07	1.39	**1.04**
	311	70.32			0.6026	0.9962	71.25	0.22	0.31	**0.93**
	312	62.91	0.0013	0.5508		0.9934	63.67	1.26	1.98	**0.76**
Day 3	**302**	74.09	0.0021	0.4730		0.9988	77.45	0.64	0.83	**3.36**
	309	76.04	0.0006	0.6192		0.9970	73.35	0.93	1.27	**-2.69**
	311	70.32			0.6879	0.9974	69.02	0.94	1.36	**-1.30**
	312	62.91	0.0011	0.5828		0.9993	68.45	1.56	2.28	**5.54**
Day 4	**302**	74.09	0.0017	0.4532		0.9998	74.21	0.75	1.01	**0.12**
	309	76.04			0.6009	0.9962	75.63	0.94	1.24	**-0.41**
	311	70.32	0.0008	0.5536		0.9977	70.75	0.69	0.98	**0.43**
	312	62.91			0.6086	0.9953	63.37	1.08	1.70	**0.46**
Day 5	**302**	74.09	0.0018	0.4275		0.9984	75.76	0.60	0.79	**1.67**
	309	76.04			0.6030	0.9956	76.67	0.68	0.89	**0.63**
	311	70.32	0.0050	0.5841		0.9970	71.93	0.50	0.70	**1.61**
	312	62.91	0.0010	0.5269		0.9969	63.73	0.98	1.54	**0.82**
Day 6	**302**	74.09	0.0021	0.3888		0.9971	75.91	0.26	0.34	**1.82**
	309	76.04	0.0015	0.4477		0.9970	77.69	1.30	1.67	**1.65**
	311	70.32			0.6114	0.9973	71.63	1.01	1.41	**1.31**
	312	62.91	0.0017	0.4080		0.9954	64.24	1.90	2.96	**1.33**

			Linear fixed range integration							
	JRC code	PTT (MS) %	a	b	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
Day 1	**302**	74.09	0.0008	0.5316		0.9981	76.22	0.39	0.51	**2.13**
	309	76.04			0.5917	0.9991	76.71	0.78	1.02	**0.67**
	311	70.32			0.5819	0.9991	71.21	1.83	2.57	**0.89**
	312	62.91	0.0016	0.4645		0.9989	65.29	1.26	1.93	**2.38**
Day 2	**302**	74.09	0.0008	0.5278		0.9946	75.77	0.81	1.07	**1.68**
	309	76.04	0.0005	0.5389		0.9905	77.87	1.11	1.43	**1.83**
	311	70.32			0.6031	0.9965	72.04	0.22	0.31	**1.72**
	312	62.91	0.0007	0.5956		0.9888	64.35	1.23	1.91	**1.44**
Day 3	**302**	74.09	0.0013	0.5645		0.9994	77.96	0.66	0.85	**3.87**
	309	76.04			0.6695	0.9962	73.44	1.16	1.58	**-2.60**
	311	70.32			0.7001	0.9993	68.62	1.06	1.54	**-1.70**
	312	62.91			0.6906	0.9971	68.20	1.44	2.11	**5.29**
Day 4	**302**	74.09	0.0008	0.5464		0.9996	74.74	0.46	0.62	**0.65**
	309	76.04			0.6006	0.9975	76.03	1.17	1.54	**-0.01**
	311	70.32			0.6035	0.9953	70.40	1.23	1.75	**0.08**
	312	62.91			0.6079	0.9967	64.89	1.00	1.54	**1.98**
Day 5	**302**	74.09	0.0009	0.5080		0.9995	78.59	0.64	0.81	**4.50**
	309	76.04			0.5963	0.9969	77.43	0.69	0.89	**1.39**
	311	70.32			0.6350	0.9969	72.70	1.20	1.65	**2.38**
	312	62.91	0.0001	0.5885		0.9922	64.48	1.17	1.81	**1.57**
Day 6	**302**	74.09	0.0014	0.4738		0.9950	77.07	0.31	0.40	**2.98**
	309	76.04	0.0013	0.4649		0.9953	80.61	0.91	1.13	**4.57**
	311	70.32			0.6167	0.9990	70.53	3.09	4.38	**0.21**
	312	62.91	0.0014	0.4507		0.9898	69.04	2.11	3.06	**6.13**

			Sigmoidal fixed range integration							
	JRC code	PTT (MS) %	a	b	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09			0.6452	0.9978	74.13	0.42	0.57	0.04
	309	76.04			0.6268	0.9988	76.98	0.78	1.01	0.94
	311	70.32			0.6235	0.9946	70.25	1.90	2.70	-0.07
	312	62.91			0.6022	0.9995	66.39	0.87	1.31	3.48
Day 2	302	74.09			0.6194	0.9974	74.92	0.98	1.31	0.83
	309	76.04	-0.0005	0.6013		0.9931	75.37	1.10	1.46	-0.67
	311	70.32			0.6350	0.9979	72.46	0.31	0.43	2.14
	312	62.91	0.0004	0.6272		0.9888	65.15	1.26	1.93	2.24
Day 3	302	74.09			0.6915	0.9959	77.58	1.12	1.44	3.49
	309	76.04			0.7024	0.9957	73.33	1.25	1.70	-2.71
	311	70.32			0.7355	0.9989	68.29	1.11	1.63	-2.03
	312	62.91			0.5940	0.9976	68.44	0.53	0.77	5.53
Day 4	302	74.09			0.6414	0.9959	73.03	0.49	0.67	-1.06
	309	76.04			0.6310	0.9985	75.84	0.96	1.27	-0.20
	311	70.32			0.6513	0.9991	69.38	1.25	1.80	-0.94
	312	62.91			0.6284	0.9968	65.58	0.97	1.48	2.67
Day 5	302	74.09			0.6142	0.9969	77.77	0.71	0.91	3.68
	309	76.04			0.6336	0.9979	77.35	0.56	0.72	1.31
	311	70.32	-0.0010	0.7614		0.9979	72.79	1.16	1.59	2.47
	312	62.91			0.6222	0.9949	64.83	1.03	1.59	1.92
Day 6	302	74.09	0.0008	0.5594		0.9955	76.29	0.38	0.50	2.20
	309	76.04			0.6196	0.9980	80.02	0.54	0.67	3.98
	311	70.32	-0.0007	0.7105		0.9996	70.30	3.56	5.06	-0.02
	312	62.91	0.0008	0.5095		0.9979	70.54	2.38	3.37	7.63

			Perpendicular drop fixed range integration							
	JRC code	PTT (MS) %	a	b	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09	-0.0014	0.7548		0.9905	74.51	0.45	0.60	0.42
	309	76.04			0.6149	0.9992	76.97	0.59	0.77	0.93
	311	70.32	-0.0070	0.6684		0.9986	70.16	1.97	2.81	-0.16
	312	62.91			0.5963	0.9994	67.07	0.86	1.28	4.16
Day 2	302	74.09	-0.0012	0.7233		0.9943	74.54	1.01	1.35	0.45
	309	76.04			0.6019	0.9949	77.26	1.24	1.60	1.22
	311	70.32			0.6349	0.9982	72.61	0.60	0.83	2.29
	312	62.91	0.0004	0.6367		0.9919	65.18	2.07	3.18	2.27
Day 3	302	74.09	-0.0008	0.7789		0.9868	77.13	0.83	1.08	3.04
	309	76.04			0.6897	0.9973	74.18	1.35	1.82	-1.86
	311	70.32			0.7259	0.9992	68.57	1.06	1.55	-1.75
	312	62.91			0.6946	0.9970	69.77	1.47	2.11	6.86
Day 4	302	74.09	-0.0010	0.7296		0.9959	72.49	0.63	0.87	-1.60
	309	76.04			0.6257	0.9974	75.49	0.91	1.21	-0.55
	311	70.32			0.6430	0.9971	69.90	1.43	2.05	-0.42
	312	62.91			0.6242	0.9963	66.77	1.04	1.56	3.86
Day 5	302	74.09	-0.0014	0.7277		0.9967	78.33	0.99	1.26	4.24
	309	76.04			0.6251	0.9984	77.71	0.49	0.63	1.67
	311	70.32	-0.0005	0.6801		0.9982	72.89	1.08	1.48	2.57
	312	62.91	-0.0005	0.6245		0.9946	64.92	0.93	1.43	2.01
Day 6	302	74.09	-0.0008	0.6926		0.9990	75.49	0.39	0.52	1.40
	309	76.04	0.0006	0.5501		0.9947	80.21	2.10	2.62	4.17
	311	70.32	-0.0008	0.7045		0.9980	70.79	3.47	4.90	0.47
	312	62.91	0.0012	0.4868		0.9888	64.46	2.18	3.38	1.55

Table 57: Quantification by the optimised DSC method applying various types of integration. (multipoint calibration curves based on PET peak)

			Linear integration							
	JRC code	PTT (MS) %	a	b	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09	0.0010	0.3428		0.9986	73.21	0.44	0.61	-0.88
	309	76.04			0.5496	0.9998	77.89	0.20	0.25	1.85
	311	70.32			0.5131	0.9946	70.18	2.37	3.37	-0.14
	312	62.91			0.5163	0.9995	61.89	0.65	1.05	-1.02
Day 2	302	74.09	0.0008	0.3453		0.9991	73.29	0.53	0.72	-0.80
	309	76.04			0.5421	0.9976	75.38	0.42	0.56	-0.66
	311	70.32			0.5236	0.9975	69.44	0.21	0.30	-0.88
	312	62.91			0.5583	0.9987	60.36	0.80	1.32	-2.55
Day 3	302	74.09	0.0011	0.3809		0.9983	72.35	0.64	0.89	-1.74
	309	76.04			0.6264	0.9985	76.31	0.32	0.42	0.27
	311	70.32			0.5981	0.9990	70.36	0.53	0.75	0.04
	312	62.91			0.5904	0.9962	64.29	0.48	0.74	1.38
Day 4	302	74.09			0.3868	0.9958	75.08	0.65	0.86	0.99
	309	76.04			0.5601	0.9980	75.96	0.86	1.13	-0.08
	311	70.32			0.5395	0.9983	71.01	1.02	1.43	0.69
	312	62.91			0.5179	0.9985	62.94	0.51	0.81	0.03
Day 5	302	74.09	0.0010	0.3264		0.9992	72.06	0.95	1.32	-2.03
	309	76.04	0.0011	0.5020		0.9977	73.97	0.05	0.07	-2.07
	311	70.32			0.5463	0.9987	69.53	0.53	0.76	-0.79
	312	62.91			0.5323	0.9992	62.25	0.60	0.97	-0.66
Day 6	302	74.09	0.0009	0.3417		0.9965	72.95	0.08	0.10	-1.14
	309	76.04	-0.0008	0.5631		0.9994	75.15	0.28	0.38	-0.89
	311	70.32			0.5402	0.9974	71.42	0.79	1.10	1.10
	312	62.91			0.4989	0.9952	64.26	0.59	0.92	1.35

			Parallel integration							
	JRC code	PTT (MS) %	a	b	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09	0.0009	0.3145		0.9982	72.81	0.74	1.02	-1.28
	309	76.04			0.5211	0.9988	76.68	0.21	0.27	0.64
	311	70.32	0.0006	0.4504		0.9911	69.98	2.62	3.74	-0.34
	312	62.91			0.4933	0.9995	60.85	1.50	2.46	-2.06
Day 2	302	74.09	0.0006	0.3207		0.9989	73.33	0.78	1.06	-0.76
	309	76.04			0.5105	0.9947	74.20	0.32	0.43	-1.84
	311	70.32			0.5011	0.9979	70.15	0.23	0.33	-0.17
	312	62.91			0.5504	0.9975	60.82	1.41	2.31	-2.09
Day 3	302	74.09	0.0009	0.3453		0.9946	71.41	0.88	1.23	-2.68
	309	76.04	0.0012	0.5258		0.9962	74.96	0.21	0.27	-1.08
	311	70.32			0.5756	0.9974	70.38	0.51	0.72	0.06
	312	62.91	0.0013	0.4880		0.9990	61.16	0.39	0.64	-1.75
Day 4	302	74.09	0.0006	0.3122		0.9955	73.78	0.73	0.99	-0.31
	309	76.04			0.5380	0.9975	76.37	1.09	1.42	0.33
	311	70.32			0.5277	0.9954	71.41	1.21	1.69	1.09
	312	62.91			0.5092	0.9968	63.14	0.59	0.93	0.23
Day 5	302	74.09	0.0011	0.2774		0.9941	70.45	1.06	1.50	-3.64
	309	76.04	0.0018	0.4420		0.9966	72.94	0.68	0.93	-3.10
	311	70.32	0.0009	0.4885		0.9966	69.23	0.35	0.50	-1.09
	312	62.91			0.5256	0.9995	62.01	0.38	0.61	-0.90
Day 6	302	74.09	0.0007	0.3175		0.9877	72.40	0.05	0.07	-1.69
	309	76.04			0.4837	0.9957	74.22	0.08	0.11	-1.82
	311	70.32	0.0014	0.4442		0.9999	69.87	0.20	0.28	-0.45
	312	62.91			0.4732	0.9978	61.89	1.11	1.79	-1.02

	JRC code	PTT (MS) %	a	b	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
			Linear fixed range integration							
Day 1	302	74.09	0.0008	0.3528		0.9995	73.41	0.35	0.48	-0.68
	309	76.04			0.5479	0.9996	75.07	0.27	0.36	-0.97
	311	70.32			0.5242	0.9981	69.85	0.25	0.36	-0.47
	312	62.91	0.0017	0.5232		0.9946	62.46	0.62	0.99	-0.45
Day 2	302	74.09			0.3836	0.9963	74.20	0.43	0.58	0.11
	309	76.04			0.5391	0.9985	75.10	0.44	0.59	-0.94
	311	70.32			0.5262	0.9964	69.42	0.31	0.45	-0.90
	312	62.91			0.5567	0.9988	61.01	1.03	1.69	-1.90
Day 3	302	74.09			0.4169	0.9986	72.70	0.68	0.94	-1.39
	309	76.04			0.6168	0.9974	75.70	0.27	0.36	-0.34
	311	70.32			0.5990	0.9988	70.53	0.76	1.08	0.21
	312	62.91			0.5908	0.9971	64.31	0.48	0.75	1.40
Day 4	302	74.09			0.3855	0.9964	74.88	0.74	0.99	0.79
	309	76.04			0.5556	0.9989	75.87	0.89	1.17	-0.17
	311	70.32			0.5397	0.9986	70.80	0.92	1.30	0.48
	312	62.91			0.5152	0.9974	62.81	0.54	0.86	-0.10
Day 5	302	74.09			0.3782	0.9946	73.92	1.02	1.38	-0.17
	309	76.04			0.5559	0.9956	75.20	0.10	0.13	-0.84
	311	70.32			0.6090	0.9986	69.84	0.59	0.84	-0.48
	312	62.91			0.5286	0.9984	61.97	0.59	0.95	-0.94
Day 6	302	74.09	0.0008	0.3458		0.9969	72.71	0.50	0.69	-1.38
	309	76.04	-0.0012	0.5880		0.9992	75.69	0.29	0.38	-0.35
	311	70.32			0.5409	0.9986	70.49	1.44	2.04	0.17
	312	62.91	0.0013	0.4558		0.9909	61.26	1.48	2.42	-1.65

	JRC code	PTT (MS) %	a	b	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
			Sigmoidal fixed range integration							
Day 1	302	74.09	0.0007	0.3524		0.9994	73.05	0.34	0.47	-1.04
	309	76.04			0.5587	0.9989	74.65	0.25	0.33	-1.39
	311	70.32			0.5172	0.9963	69.23	0.27	0.39	-1.09
	312	62.91	0.0007	0.5741		0.9882	63.25	0.55	0.87	0.34
Day 2	302	74.09			0.3822	0.9960	73.94	0.43	0.58	-0.15
	309	76.04			0.5529	0.9989	75.01	0.43	0.57	-1.03
	311	70.32			0.5312	0.9956	69.50	0.31	0.45	-0.82
	312	62.91			0.5568	0.9989	61.00	1.15	1.89	-1.91
Day 3	302	74.09			0.4149	0.9986	72.40	0.72	0.99	-1.69
	309	76.04			0.6343	0.9990	76.04	0.23	0.30	0.00
	311	70.32			0.6017	0.9987	70.57	0.76	1.08	0.25
	312	62.91			0.7027	0.9977	64.49	0.13	0.20	1.58
Day 4	302	74.09			0.3839	0.9964	74.54	0.76	1.02	0.45
	309	76.04			0.5644	0.0000	75.72	0.69	0.91	-0.32
	311	70.32			0.5414	0.9989	70.89	0.91	1.28	0.57
	312	62.91			0.5166	0.9979	62.96	0.35	0.56	0.05
Day 5	302	74.09	0.0008	0.3297		0.9997	72.19	1.14	1.58	-1.90
	309	76.04			0.5642	0.9963	75.13	0.16	0.21	-0.91
	311	70.32			0.5547	0.9979	69.93	0.61	0.87	-0.39
	312	62.91			0.5299	0.9984	62.05	0.45	0.73	-0.86
Day 6	302	74.09	0.0008	0.3432		0.9960	72.46	0.50	0.69	-1.63
	309	76.04	-0.0013	0.5997		0.9960	75.22	0.38	0.51	-0.82
	311	70.32			0.5444	0.9994	70.47	1.46	2.07	0.15
	312	62.91	-0.0007	0.5342		0.9919	61.13	1.59	2.60	-1.78

	JRC code	PTT (MS) %	Perpendicular drop fixed range integration							
			a	b	m	R^2	PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09			0.4634	0.9956	74.48	0.20	0.27	0.39
	309	76.04			0.5614	0.9996	74.80	0.33	0.44	-1.24
	311	70.32	0.0005	0.5259		0.9926	70.21	2.58	3.67	-0.11
	312	62.91			0.5217	0.9995	60.50	1.19	1.97	-2.41
Day 2	302	74.09			0.4547	0.9981	73.81	0.40	0.54	-0.28
	309	76.04			0.5574	0.9982	75.13	0.44	0.59	-0.91
	311	70.32			0.5412	0.9950	68.69	0.87	1.27	-1.63
	312	62.91			0.5744	0.9983	59.50	2.00	3.36	-3.41
Day 3	302	74.09			0.5156	0.9955	73.46	0.75	1.02	-0.63
	309	76.04			0.6462	0.9984	75.66	1.49	1.97	-0.38
	311	70.32			0.6156	0.9982	70.52	0.76	1.08	0.20
	312	62.91	0.0012	0.5282		0.9997	61.13	0.68	1.11	-1.78
Day 4	302	74.09			0.4702	0.9979	74.77	0.36	0.48	0.68
	309	76.04	-0.0002	0.5896		0.9926	76.92	1.54	2.00	0.88
	311	70.32			0.5617	0.9981	71.10	1.09	1.53	0.78
	312	62.91			0.5323	0.9957	61.23	0.79	1.29	-1.68
Day 5	302	74.09			0.4581	0.9988	73.17	0.33	0.45	-0.92
	309	76.04	0.0011	0.5077		0.9972	73.48	0.23	0.31	-2.56
	311	70.32			0.5619	0.9961	69.60	0.59	0.85	-0.72
	312	62.91			0.5449	0.9998	60.95	0.96	1.58	-1.96
Day 6	302	74.09			0.4672	0.9965	74.37	0.69	0.93	0.28
	309	76.04	-0.0013	0.5999		0.9989	75.58	0.50	0.66	-0.46
	311	70.32			0.5620	0.9952	70.19	1.58	2.25	-0.13
	312	62.91			0.5072	0.9957	62.78	1.67	2.66	-0.13

Table 58: Quantification by the optimised DSC method applying various types of integration. (single point calibration based on PTT peak)

	JRC code	PTT (MS) %	Linear integration				Parallel integration			
			PTT (DSC) %	SD %	CV %	Bias %	PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09	71.66	0.41	0.57	-2.43	71.15	0.60	0.84	-2.94
	309	76.04	75.57	0.51	0.67	-0.47	74.77	0.35	0.47	-1.27
	311	70.32	69.97	2.08	2.97	-0.35	69.41	1.88	2.71	-0.91
	312	62.91	63.45	0.49	0.77	0.54	61.88	0.53	0.86	-1.03
Day 2	302	74.09	72.47	0.87	1.20	-1.62	70.91	0.73	1.03	-3.18
	309	76.04	75.71	1.44	1.90	-0.33	75.24	1.14	1.52	-0.80
	311	70.32	70.78	0.22	0.31	0.46	70.33	0.22	0.31	0.01
	312	62.91	62.96	1.24	1.97	0.05	60.86	1.37	2.25	-2.05
Day 3	302	74.09	72.05	0.80	1.11	-2.04	71.40	0.74	1.04	-2.69
	309	76.04	72.78	1.16	1.59	-3.26	72.71	0.98	1.35	-3.33
	311	70.32	68.17	1.02	1.50	-2.15	68.01	0.92	1.35	-2.31
	312	62.91	66.24	0.46	0.69	3.33	64.80	1.64	2.53	1.89
Day 4	302	74.09	69.77	0.74	1.06	-4.32	68.60	0.84	1.22	-5.49
	309	76.04	74.51	0.80	1.07	-1.53	74.41	0.93	1.25	-1.63
	311	70.32	70.21	0.59	0.84	-0.11	69.09	0.73	1.06	-1.23
	312	62.91	64.32	1.13	1.76	1.41	62.60	1.06	1.69	-0.31
Day 5	302	74.09	71.09	0.35	0.49	-3.00	70.06	0.68	0.97	-4.03
	309	76.04	75.91	0.42	0.55	-0.13	75.70	0.67	0.89	-0.34
	311	70.32	71.24	0.50	0.70	0.92	70.47	0.52	0.74	0.15
	312	62.91	62.48	0.97	1.55	-0.43	60.68	1.25	2.06	-2.23
Day 6	302	74.09	70.44	0.56	0.80	-3.65	69.53	0.31	0.45	-4.56
	309	76.04	72.98	0.88	1.21	-3.06	72.88	1.47	2.02	-3.16
	311	70.32	71.35	0.75	1.05	1.03	70.15	1.03	1.47	-0.17
	312	62.91	58.15	1.48	2.55	-4.76	58.56	2.10	3.59	-4.35

	JRC code	PTT (MS) %	Linear fixed range integration: PTT (DSC) %	SD %	CV %	Bias %	Sigmoidal fixed range integration: PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09	72.31	0.46	0.64	-1.78	73.13	0.41	0.56	-0.96
	309	76.04	75.97	0.77	1.01	-0.07	76.79	0.78	1.02	0.75
	311	70.32	72.22	1.86	2.58	1.90	71.63	1.83	2.55	1.31
	312	62.91	64.25	1.24	1.93	1.34	66.91	1.25	1.87	4.00
Day 2	302	74.09	72.34	0.93	1.29	-1.75	75.53	0.99	1.31	1.44
	309	76.04	79.08	1.17	1.48	3.04	76.45	1.06	1.39	0.41
	311	70.32	71.63	0.21	0.29	1.31	72.79	0.31	0.43	2.47
	312	62.91	63.52	1.31	2.06	0.61	64.93	1.31	2.02	2.02
Day 3	302	74.09	72.28	0.77	1.07	-1.81	77.69	1.20	1.54	3.60
	309	76.04	74.13	1.17	1.58	-1.91	74.82	1.28	1.71	-1.22
	311	70.32	68.52	1.06	1.55	-1.80	68.97	1.12	1.62	-1.35
	312	62.91	66.73	1.41	2.11	3.82	67.89	0.53	0.78	4.98
Day 4	302	74.09	70.21	0.52	0.74	-3.88	71.10	0.48	0.68	-2.99
	309	76.04	75.93	1.17	1.54	-0.11	76.47	0.97	1.27	0.43
	311	70.32	70.74	1.24	1.75	0.42	69.70	1.26	1.81	-0.62
	312	62.91	65.49	1.01	1.54	2.58	66.70	0.98	1.47	3.79
Day 5	302	74.09	74.19	0.74	1.00	0.10	76.21	0.70	0.92	2.12
	309	76.04	77.63	70.00	90.17	1.59	78.58	0.56	0.71	2.54
	311	70.32	73.58	0.90	1.22	3.26	74.91	1.11	1.48	4.59
	312	62.91	64.42	1.20	1.86	1.51	65.39	1.04	1.59	2.48
Day 6	302	74.09	74.62	0.36	0.48	0.53	73.75	0.40	0.54	-0.34
	309	76.04	79.64	0.90	1.13	3.60	78.56	0.53	0.67	2.52
	311	70.32	71.12	3.12	4.39	0.80	72.83	3.41	4.68	2.51
	312	62.91	65.10	2.34	3.59	2.19	67.48	2.50	3.70	4.57

	JRC code	PTT (MS) %	Perpendicular drop fixed range integration: PTT (DSC) %	SD %	CV %	Bias %
Day 1	302	74.09	77.74	0.40	0.51	3.65
	309	76.04	76.66	0.59	0.77	0.62
	311	70.32	73.07	1.86	2.55	2.75
	312	62.91	67.47	0.86	1.27	4.56
Day 2	302	74.09	77.54	0.93	1.20	3.45
	309	76.04	77.49	1.25	1.61	1.45
	311	70.32	73.07	0.60	0.82	2.75
	312	62.91	65.37	2.15	3.29	2.46
Day 3	302	74.09	77.23	0.76	0.98	3.14
	309	76.04	75.01	1.37	1.83	-1.03
	311	70.32	69.07	1.06	1.53	-1.25
	312	62.91	68.44	1.44	2.10	5.53
Day 4	302	74.09	74.47	0.57	0.77	0.38
	309	76.04	76.08	0.92	1.21	0.04
	311	70.32	71.38	1.46	2.05	1.06
	312	62.91	67.98	0.99	1.46	5.07
Day 5	302	74.09	81.04	0.85	1.05	6.95
	309	76.04	75.75	0.48	0.63	-0.29
	311	70.32	74.34	1.04	1.40	4.02
	312	62.91	63.84	0.92	1.44	0.93
Day 6	302	74.09	77.45	0.36	0.46	3.36
	309	76.04	78.40	2.21	2.82	2.36
	311	70.32	72.52	3.24	4.47	2.20
	312	62.91	61.43	2.36	3.84	-1.48

By analysing the results it was evident that the two approaches for integration, linear and linear fixed range, were the best ones to obtain accurate quantification of binary blends PTT/PET. Concerning the calibration, the best results were obtained using multipoint calibration curves based on PET peak, followed by the results calculated with multipoint calibration curves based on PTT peak and the ones measured with single point calibration based on PTT peak. In the case of the quantification done with multipoint calibration curves based on PET peak and the linear fixed range integration, the biases were always lower than 2 % and in 19 samples out of 24 lower than 1 %.

Such small biases confirmed the adequate accuracy and repeatability of the optimised DSC method that was validated with exactly the same SOP in collaboration with laboratories from Member States, DuPont and the JRC. The type of integration selected was the linear fixed range as it appeared less influenced by the decisions of each operator. It was decided to further test and compare, in the validation study, the accuracy of the quantification done using multipoint calibration curves and single point calibration based on both PTT and PET peak.

7. Method validation

During the 12th ENNETL meeting, held at the JRC premises in Ispra (Italy) on 30th November 2012, it was decided that the optimised DSC method would have needed to be fully validated through a collaborative exercise.

The study was organised by the JRC according to the standard ISO 5725-2:1994 [9], as a balanced uniform–level experiment, i.e. with the same number of test results in each laboratory and with each laboratory analysing the same levels of test samples. Twelve laboratories from Member States were involved in the validation exercise, as well as DuPont and JRC laboratories. One of the aims of the validation study was to judge which one, among the four following integration approaches, was the most accurate: calibration curve based on the integration of either the PTT or the PET melting peak and quantification based on single point calibration using the area of either the pure PTT or the pure PET melting peak.

The preparation, analyses and results of the method validation are described in the following sections.

7.1 Sample preparation

Six PTT/PET binary mixtures (samples **302**, **303**, **309**, **311**, **312** and **315**) with different levels of PTT were selected for the validation study. For the collaborative study, they were respectively encoded with the letters **A - F**. Apart from sample **303**, which was knitted fabric, the other blends were all woven fabrics. The reference values for the PTT content were measured via manual separation for all samples, except sample F for which manual separation was not feasible because one of the yarns of the woven fabric was an intimate mixture of PTT and PET. The PTT percentages thus calculated for samples **A**, **B**, **C**, **D** and **E** were 74.04, 28.50, 76.04, 70.32 and 62.91 %, respectively.

The preparation of the samples started with the cut of specimens in form of squares of approximately 10 cm^2 (about one gram) taken at random on the entire surface of each woven sample. Specimens were then randomly numbered, mixed, packed and labelled. Each package, made of polypropylene, contained one fabric square, either to be analysed or to be considered as spare sample. Ten packages, casually selected, were used for the homogeneity study and the others were prepared to be sent to the participants. Additionally, for the preparation of the calibration curves, laboratories were provided with pure PTT and PET yarns manually separated, by the JRC, from each of the six samples under evaluation. In order to homogenise the extracted yarns, they were put into a 2 L glass bottle and mixed by means of a rotator drive (Stuart STR4, Staffordshire, UK) for 1 hour at 60 rpm (Figure 25). The homogeneised PTT and PET yarns extracted from each fabric sample were then packed individually and put in a bigger package containing also the corresponding fabric and spare fabric samples.

Packages were marked with codes indicating the laboratories' code (LC01-LC20), the sample code (**A-F**), a random number (1-20) and the nature of the sample (fabric, spare fabric, PTT or PET) (Figure 26).

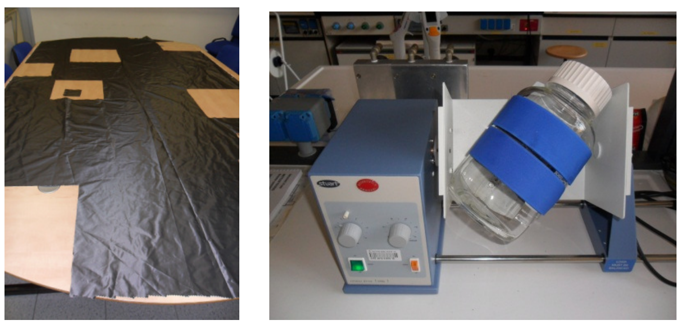


Figure 25: left) Preparation and cutting of woven fabric; right) Rotator drive device for sample homogenisation.

Figure 26: Samples packages sent to participants.

7.2 Homogeneity assessment

The JRC team carried out the homogeneity assessment on each sample, according to the test for 'sufficient homogeneity' established in the IUPAC harmonised protocol for proficiency testing [12]. Ten packages for each fabric sample were randomly selected and two test portions from each package were analysed in a random order under repeatability conditions. Each sample was quantified with calibration curves based on both PTT and PET enthalpies of fusion and the results are reported in Table 59.

Table 59: Homogeneity study results.

		Based on PTT peak		Based on PET peak				Based on PTT peak		Based on PET peak	
		Portion a	Portion b	Portion a	Portion b			Portion a	Portion b	Portion a	Portion b
	Package	PTT %	PTT %	PTT %	PTT %		Package	PTT %	PTT %	PTT %	PTT %
Sample A	1	77.96	77.16	74.42	73.93	Sample B	1	29.32	29.02	27.97	28.60
	2	76.33	76.67	74.56	74.09		2	28.41	27.19	27.87	31.29
	3	78.77	78.10	77.13	73.51		3	28.96	28.83	29.42	28.97
	4	76.42	77.18	73.11	74.98		4	29.32	30.07	26.94	30.48
	5	78.02	77.53	73.84	73.91		5	30.39	27.42	29.07	26.29
	6	77.79	75.83	73.84	75.01		6	28.64	29.09	29.25	28.97
	7	77.51	75.26	73.18	73.86		7	28.36	28.71	29.25	29.03
	8	76.58	76.15	74.51	74.65		8	29.48	30.15	29.23	29.15
	9	76.25	75.72	74.58	73.67		9	27.93	30.45	27.31	29.22
	10	76.42	75.96	74.02	74.00		10	28.87	29.74	30.44	29.22
Sample C	1	73.60	77.31	75.01	75.33	Sample D	1	70.42	71.12	70.16	70.99
	2	80.88	81.41	74.44	72.94		2	71.70	72.06	69.39	69.77
	3	78.88	79.28	75.01	74.63		3	73.17	68.86	69.18	71.58
	4	78.36	81.50	74.67	74.91		4	70.64	72.81	70.05	69.90
	5	80.43	80.43	74.42	75.37		5	71.53	72.62	71.05	69.11
	6	78.85	79.40	75.03	74.72		6	68.15	72.87	71.82	69.46
	7	81.09	82.92	74.15	72.89		7	70.82	67.25	68.91	71.69
	8	79.28	78.05	75.05	74.88		8	73.28	70.26	69.15	71.29
	9	74.17	78.92	75.66	74.72		9	70.36	72.34	71.19	69.72
	10	77.70	81.31	74.91	73.25		10	71.56	72.57	69.85	70.05
Sample E	1	63.38	64.14	61.86	64.80	Sample F	1	60.19	59.88	55.88	55.28
	2	62.76	65.39	62.04	60.09		2	62.39	59.97	55.70	54.75
	3	60.87	62.69	60.17	61.12		3	59.73	61.47	56.42	56.03
	4	64.22	65.92	60.44	64.02		4	60.14	60.14	55.86	55.26
	5	63.53	65.26	60.56	63.34		5	59.80	60.50	55.30	55.51
	6	62.97	64.11	63.65	59.45		6	59.22	60.63	55.61	56.64
	7	62.32	62.08	61.71	61.32		7	59.37	60.26	56.09	55.84
	8	63.02	62.86	60.83	61.67		8	59.08	61.40	56.62	56.03
	9	64.44	65.18	59.63	60.85		9	58.79	60.96	55.98	55.22
	10	64.37	65.01	59.32	60.07		10	59.90	60.62	56.15	55.57

Table 60: Samples' homogeneity evaluation.

		outliers	s_{sam}^2	c	Homogeneity test	W	p	Normality test
Sample A	% PTT (PTT peak)	no	0.33	1.02	OK	0.97	0.668	OK
	% PTT (PET peak)	yes	-0.26	1.41	OK	0.83	0.002	NO
Sample B	% PTT (PTT peak)	no	-0.14	1.03	OK	0.97	0.733	OK
	% PTT (PET peak)	no	-0.51	1.99	OK	0.93	0.152	OK
Sample C	% PTT (PTT peak)	no	2.29	3.73	OK	0.92	0.09	OK
	% PTT (PET peak)	no	0.15	0.86	OK	0.85	0.005	NO
Sample D	% PTT (PTT peak)	no	-0.97	4.12	OK	0.92	0.092	OK
	% PTT (PET peak)	no	-0.66	1.91	OK	0.91	0.069	OK
Sample E	% PTT (PTT peak)	no	0.77	1.27	OK	0.97	0.836	OK
	% PTT (PET peak)	no	-0.28	3.04	OK	0.92	0.119	OK
Sample F	% PTT (PTT peak)	no	-0.41	1.44	OK	0.96	0.583	OK
	% PTT (PET peak)	no	0.03	0.46	OK	0.98	0.864	OK

For the statistical evaluation the unrounded figures were used. First, the normality of the distribution of each data set was evaluated using the Shapiro-Wilk test [13]. The null hypothesis for this test was that the data were normally distributed. For a chosen alpha level of 0.05, the null hypothesis was accepted when the p-value was greater than 0.05. As reported in Table 60, almost all set of data could be considered normally distributed.

Data were then examined visually for pathologies (see Annex VII). No trends or non-random distribution of differences between first and second test results were observed. Out of the 120 couples of results only one was considered an outlier after applying the Cochran's test to the set of measurements. The estimates of sampling variance (s^2_{sam}, between variance) and analytical variance (s^2_{an}, within variance) were calculated with a single-factor analysis of variance (ANOVA). According to the test, samples can be considered 'sufficiently homogeneous' if the sampling variance is lower than a critical value *c*, where $c = 1.88\ \sigma^2_{all} + 1.01\, s^2_{an}$, considering ten testing specimens. The allowable variance (σ^2_{all}) is calculated following the formula σ^2_{all} = 0.09 x σ^2_p, where σ^2_p is the target variance, an estimation of the expected variability of the trial.

As the reproducibility limit R of the DSC method was unknown, the target coefficient of variation was calculated on the basis of the Horwitz equation PCV_R (%) = $2*C^{-0.15}$, using the PTT content obtained during the homogeneity test [14]. The target standard deviation σ_p was then calculated multiplying the obtained value for the mean of the data set and dividing by 100 ($\sigma_p = CV_R$ (%) * mean / 100). All six samples could be considered 'sufficiently homogeneous' as their sampling variances were smaller than *c* (Table 59).

7.3 Distribution and instructions

Samples were dispatched from 16^{th} to 29^{th} July 2013 to 15 participants, listed in Table 61. Twelve laboratories based on Member States took part in the validation; in addition also the petitioner (DuPont) and two different operators in the JRC joined the study. The analyses made at the JRC could be considered as obtained by two different participants as they were conducted in different days on different samples and by two independent operators. Each participant received:

- six binary mixtures (**A**, **B**, **C**, **D**, **E** and **F**) made of PTT/PET, plus one spare sample for each mixture;

- six yarn samples (**A**, **B**, **C**, **D**, **E** and **F**) made of pure PTT manually separated from the correspondent fabric sample;
- six yarn samples (**A**, **B**, **C**, **D**, **E** and **F**) made of pure PET manually separated from the correspondent fabric sample;
- an accompanying letter with instructions (Annex VII);
- standard operating procedure to be carefully and strictly applied (Annex VII);
- Excel sheets containing calculation formulas for reporting results (Annex VII);
- a form that had to be sent back after the sample reception to confirm their arrival (Annex VII);

Table 61: Participants enrolled in the validation study.

Laboratory	Country
BWZ DS-Hamburg	Germany
C.N.R. (Consiglio Nazionale delle Ricerche) ISMAC Institute	Italy
DuPont de Nemours and Company Inc.	USA
European Commission, JRC, Chemical Assessment and Testing Unit-1	Italy
European Commission, JRC, Chemical Assessment and Testing Unit-2	Italy
General Chemical State Laboratory	Greece
IFTH (Institut Francais Textile-Habillement)-Tourcoing	France
INCDTP-National Research and Development Institute for Textile and Leather	Romania
INTEXTER - Universitat Politecnica de Catalunya	Spain
Intitute of Biopolymers and Chemical Fibres, Special Fibres Team	Poland
Shirley Technologies Ltd.	United Kingdom
Tampere University of Technology	Finland
Teknologisk Institute	Denmark
Textile Institute of CPST	Lithuania
Textile Testing Institute	Czech Republic

Instructions were sent to all participants. For the precision experiments the laboratories were asked to analyse three replicates for each of the six samples (**A-F**) with the provided method. The DSC analysis had to be conducted in repeatability conditions, and in case of any problem during the analysis of some replicates all the three replicates of the sample had to be repeated. Calibration curves, with seven experimental points each, had to be prepared for each sample with the PTT and PET yarns separated from the sample under quantification and provided by the JRC.

The results had to be reported by the end of August 2013, in the Excel templates provided by the JRC team. By 13th September 2013, all laboratories reported their results.

7.4 Results of collaborative trial

Participants were asked to strictly follow the SOP of method developed by the JRC, fully reported in Annex VII. The integration had to be performed drawing a linear baseline between fixed ranges of temperature. For each sample under evaluation, laboratories had to analyse three replicates of the sample, three replicates of both the pure PTT and the pure PET yarns manually separated from the sample, plus five binary mixtures prepared using the PTT and PET yarns just mentioned to build the two calibration curves based on the integration of either the PTT or the PET melting peak.

Laboratories were encoded to avoid identification. Results were received from all laboratories by 13th September 2013. The participants obtained all the results requested under repeatability conditions, i.e. within a short interval of time and by the same operator. In total, 1980 test results were collected.

Some deviations from the instructions were reported. In particular, Laboratory 11 used air instead of a nitrogen atmosphere during the DSC analyses and Laboratory 13 did not make holes in pan lids and used a slightly different temperature program (room temperature - 30 °C/min – 140 °C – 5 °C/min – 285 °C) stopping the DSC analysis at 285 °C instead of 300 °C and starting the ramp of 5 °C/min at 140 °C instead of 150 °C. The participants' comments are reported in Annex VII. Table 62 reports the information regarding the equipment used in the laboratories.

Table 62: Equipment, cooling system, reference standards for temperature and heat capacity calibration used in the collaborative trial.

	DSC manufacturer	Model	Autosampler (yes/no)	Software used for the integration
LAB 1	Perkin Elmer	DSC-7	no	Pyris
LAB 2	Netzsch	DSC 200 F3	no	Netzsch Proteus Thermal Analysis
LAB 3	Mettler Toledo	DSC 821	no	STARe SW 9.3
LAB 4	TA Instruments	Q10	no	TA Advantage Software for Thermal Analysis
LAB 5	PerkinElmer	Diamond	no	Pyris
LAB 6	Mettler Toledo	DSC 1	no	STAR Software Version 10.0
LAB 7	Perkin Elmer	DSC 7	no	PYRIS version 4
LAB 8	Perkin Elmer	DSC 4000	no	Pyris
LAB 9	Perkin Elmer	Diamond DSC	no	PyrisManager
LAB 10	Netzsch	DSC 204 F1	yes	Netzsch Proteus Analysis
LAB 11	Perkin Elmer	DSC 7	no	PC Series Thermal Analysis System
LAB 12	Mettler Toledo	823	yes	STARe
LAB 13	TA Instruments	Q-1000	yes	Universal analysis 2001
LAB 14	TA Instruments	DSC-Q100	yes	Universal analysis 2000
LAB 15	TA Instruments	DSC-Q100	yes	Universal analysis 2000

	Cooling system	Reference standards for temperature calibration	Reference standards for heat capacity calibration
LAB 1	Cool water	In and Sn	In (enthalpy calibration)
LAB 2	Liqui Nitrogen	In, Sn, Bi, Hg, Zn	In, Sn, Bi, Hg, Zn, CsCl
LAB 3	intra cooler HAAKE EK 90 MT	Indium	
LAB 4	Refrigerated Cooling System (RCS)	Indium Metal	Indium Metal
LAB 5	water	Indium, zinc	Indium, zinc
LAB 6	Cryostat Cooling, HUBER TC 100 RC	In, Sn	In, Sn
LAB 7	NO	Tin	Tin
LAB 8	Closed system water cooler	Tin reference standard	Tin reference standard
LAB 9	Intracooler	156.6(In), 419.47(Zn)	28.45(In), 108.37 (Zn)
LAB 10	Netzsch CC 200 F1 with liquid nitrogen	Indium and Tin	Indium and Tin
LAB 11	air cooling	In, Sn	In
LAB 12	Huber	In-Zn-H2O	In
LAB 13	Mechanical Cooler	Hg, In, Sn, Pb, Zn	Heat capacity not calibrated for these experiments. Heat of fusion calibrated by Indium
LAB 14	Refrigerated Cooling System (RCS)	Indium	Sapphire
LAB 15	Refrigerated Cooling System (RCS)	Indium	Sapphire

The results were statistically evaluated to determine the consensus value, the repeatability and reproducibility, following the rules laid down in ISO 5725-5 and 5725-2 [15, 9]. The software Prolab [16] was used to perform the statistic calculations. For the statistical evaluation the unrounded figures were used. All participants provided their results in the templates developed and provided by the JRC.

Some samples, for example **A**, **C** and **F** and the corresponding pure PET yarns manually separated from them, showed either melting peak for PET with shoulders, or more than one peak corresponding to the PET melting. The method protocol requested to integrate all these as if it was a unique peak.

At a first look, it was immediately evident that in several cases the calibration curves could not be considered linear, as the corresponding correlation coefficients R^2 were too far from the unity. In order to harmonise as much as possible the data treatment, all calibration curves showing a correlation coefficient higher than 0.995 were considered linear, whereas the others were either recalculated eliminating the outlier point (when it existed) or fitting the points with quadratic curves which were later used to calculate the final content of PTT.

After a first evaluation of the results, some doubts arose regarding the accuracy of the integration step. For this reason, laboratories were asked to send, for each sample, the DSC thermograms of the fabric, the pure PTT and one point of the calibration curve in order to verify the integration of the PTT and PET melting peaks. The received information was evaluated and, for example, it was noted that in some cases the integration of the melting peaks were not done between fixed ranges of temperature for the same set of analyses. Certain laboratories were then requested to reintegrate some set of thermograms and the new results were reported by the end of September 2013 (Table 63).

Table 63: Requests of reintegration.

Lab code	Samples reintegrated	Reason
LAB 7	B/C/E/F	the same range of temperature was not used for the integration of the set of data
LAB 9	A/B/C/D/E/F	
LAB 12	B/C/F	
LAB 4	E	
LAB 3	A	not all the PET peaks were integrated as a unique peak
LAB 8	A	
LAB 8	C/D/E	the integration range for PTT had to be expanded

The percentage of PTT in the mixtures was determined on the basis of four different approaches: two multipoint calibration curves built up considering the fusion enthalpies of PTT or PET, respectively; and two single point calibrations based on the enthalpies of fusion of 100 % PTT or PET. The PTT and PET yarns used for calibrating were manually separated from the samples under evaluation.

The statistical analysis was performed according to ISO 5725-5 on the corrected results which are presented in Annex VII and summarised in Tables 66-71. For each of the four integration approaches, tables A, B and C (representing the original data, the mean values and the standard deviation, respectively) are reported in the Annex.

In order to guarantee that the test results are normally distributed, the standard ISO 5725 assumes that the materials to be tested are homogeneous and that all laboratories (apart from very few outlier ones) have the same analytical performance.

The normality of the distribution of each set of data was evaluated by the Shapiro-Wilk test [13]. As shown in Table 64, out of the 24 data sets only 4 could be considered as being part of a normal distribution at 95 % probability level in the case of multipoint calibration and one in the case of single point calibration.

It has to be noticed that the statistical evaluation of results should be used with due care, in case of absence of normality of data sets. In fact, the statistical treatment of data described in ISO 5725 is based on the assumption that the data come from normal distributions. However, the distribution of the mean values, reported in Table

65, was found normal in the majority of cases (14 data sets considered normal out of 24).

Table 64: Results of Shapiro-Wilk test for normality on original corrected data.

		Multipoint calibration			Single point calibration		
		W	p	Normality test	W	p	Normality test
Sample A	% PTT (PTT peak)	0.90	0.001	NO	0.89	0.001	NO
	% PTT (PET peak)	0.77	<0.0001	NO	0.93	0.007	NO
Sample B	% PTT (PTT peak)	0.78	<0.0001	NO	0.71	<0.0001	NO
	% PTT (PET peak)	0.79	<0.0001	NO	0.8	<0.0001	NO
Sample C	% PTT (PTT peak)	0.93	0.007	NO	0.87	0.000	NO
	% PTT (PET peak)	0.96	0.175	OK	0.94	0.020	NO
Sample D	% PTT (PTT peak)	0.98	0.536	OK	0.92	0.005	NO
	% PTT (PET peak)	0.86	<0.0001	NO	0.96	0.099	OK
Sample E	% PTT (PTT peak)	0.89	0.001	NO	0.91	0.002	NO
	% PTT (PET peak)	0.91	0.001	NO	0.95	0.039	NO
Sample F	% PTT (PTT peak)	0.96	0.129	OK	0.94	0.015	NO
	% PTT (PET peak)	0.96	0.087	OK	0.94	0.022	NO

Table 65: Results of Shapiro-Wilk test for normality on original mean values.

		Multipoint calibration			Single point calibration		
		W	p	Normality test	W	p	Normality test
Sample A	% PTT (PTT peak)	0.96	0.730	OK	0.94	0.432	OK
	% PTT (PET peak)	0.78	0.002	NO	0.94	0.358	OK
Sample B	% PTT (PTT peak)	0.73	0.000	NO	0.67	0.000	NO
	% PTT (PET peak)	0.75	0.001	NO	0.77	0.001	NO
Sample C	% PTT (PTT peak)	0.91	0.148	OK	0.76	0.001	NO
	% PTT (PET peak)	0.92	0.217	OK	0.9	0.107	OK
Sample D	% PTT (PTT peak)	0.89	0.069	OK	0.86	0.021	NO
	% PTT (PET peak)	0.84	0.012	NO	0.94	0.359	OK
Sample E	% PTT (PTT peak)	0.78	0.002	NO	0.89	0.075	OK
	% PTT (PET peak)	0.86	0.024	NO	0.92	0.200	OK
Sample F	% PTT (PTT peak)	0.88	0.044	OK	0.92	0.165	OK
	% PTT (PET peak)	0.94	0.371	OK	0.89	0.064	OK

Results, in terms of assigned values and precision parameters, were calculated using Part-5 of the standard ISO 5725 [15]. This part describes methods, alternative to the one explained in Part 2, for the determination of precision of a standard measurement method. In ISO 5725 - Part 2, Mandel's h and k statistics and Cochran's and Grubbs' tests are performed to identify statistical outliers and stragglers; it is up to the statistician then to decide which results to discard and which not. These decisions will influence the calculated standard mean values and standard deviations for repeatability and reproducibility. The impact can be strong, in particular considering that quite often results from a precision experiment are at the border between stragglers and statistical outliers. The robust method proposed in Part 5 allows elaborating data without the need to take such decisions as no data are rejected. In fact, by assigning different weights to the data coming from each laboratory, the method calculates results in a way that they are not influenced much by data of low quality.

Tables 66-71 summarise the outcome of the validation study when results are treated following ISO 5725-5. Tables 66-69 report the PTT contents calculated on the basis of the quantification done respectively *via* multipoint calibration curves based on either PTT or PET peak, and *via* single point calibration based again on either PTT or PET peak. In Tables 70 and 71, the bias and confidence limit values are highlighted in yellow when higher than 1 and lower than 2, and in red if higher than 2. For the HORRAT parameters, values between 2 and 4.are highlighted in yellow, and in red if higher than 4.

Table 66: Results of the collaborative trial.
(ISO 5725-5, multipoint calibration curve based on PTT peak)

	Sample A		Sample B		Sample C		Sample D		Sample E		Sample F	
Lab code	PTT %	SD %	PTT %	SD %	PTT %	SD %	PTT %	SD %	PTT %	SD %	PTT %	SD %
1	72.96	0.43	29.55	1.13	79.58	0.22	72.38	0.44	55.33	1.10	59.26	0.59
2	80.15	3.18	30.08	0.10	79.35	1.01	70.03	2.78	63.59	1.93	59.84	2.89
3	78.28	0.49	29.54	0.38	74.95	2.62	70.44	0.88	63.98	1.24	57.26	1.28
4	73.11	2.92	34.95	0.36	77.19	0.18	74.81	1.31	65.68	1.65	60.73	2.37
5	76.20	0.28	28.49	0.23	76.80	1.23	70.73	0.98	63.52	0.26	59.41	0.32
6	75.56	0.82	28.96	0.40	76.61	1.14	70.46	0.53	63.70	0.81	59.47	1.53
7	72.61	0.84	29.23	0.32	76.73	1.02	70.68	1.35	70.42	1.99	58.35	1.08
8	71.31	8.89	29.47	1.50	70.62	1.35	71.89	2.83	64.80	5.53	58.34	5.52
9	73.83	5.40	38.49	1.04	78.98	0.74	74.99	0.32	69.06	2.16	64.21	2.09
10	74.42	0.38	29.67	0.84	76.68	0.85	72.59	1.64	63.36	0.89	59.56	0.37
11	78.13	0.26	26.57	1.61	73.91	3.06	70.81	0.61	63.89	1.83	58.11	1.97
12	71.94	0.23	28.64	0.49	72.43	0.64	69.83	0.93	64.01	0.20	61.54	1.84
13	76.31	0.12	28.44	0.79	77.25	0.32	71.01	1.13	63.48	0.44	59.05	0.52
14	75.46	0.60	29.26	0.55	77.14	0.83	73.30	0.58	63.52	0.82	58.53	2.13
15	74.22	2.64	26.97	0.48	72.08	0.45	72.21	2.35	62.81	1.33	59.99	2.23

Table 67: Results of the collaborative trial.
(ISO 5725-5, multipoint calibration curve based on PET peak)

	Sample A		Sample B		Sample C		Sample D		Sample E		Sample F	
Lab code	PTT %	SD %	PTT %	SD %	PTT %	SD %	PTT %	SD %	PTT %	SD %	PTT %	SD %
1	75.23	2.36	25.49	2.56	77.34	0.90	69.89	1.00	54.22	0.77	57.28	0.30
2	70.63	0.96	27.18	1.70	73.61	0.46	69.11	1.52	61.54	3.02	55.30	2.49
3	73.37	1.22	28.91	1.41	74.31	1.04	68.68	0.22	65.06	0.62	57.27	0.46
4	75.54	2.10	22.96	2.36	73.80	0.20	67.60	0.65	59.01	1.41	55.89	2.60
5	72.82	0.54	27.28	0.35	75.19	0.10	69.32	0.73	61.19	0.64	56.23	0.10
6	72.91	0.80	30.45	2.08	75.40	0.61	69.73	0.09	62.19	0.37	57.29	0.26
7	71.92	2.25	30.37	0.88	76.88	1.40	71.12	0.61	61.65	1.33	54.76	1.96
8	77.05	2.82	27.72	3.90	76.94	0.81	71.20	0.28	64.45	1.58	60.21	2.63
9	62.03	6.65	52.88	1.02	73.78	0.80	63.28	2.44	62.44	0.66	53.65	3.61
10	72.00	0.54	28.49	2.49	76.12	0.56	67.37	0.58	60.38	0.30	54.91	0.42
11	74.03	0.17	38.43	2.21	71.17	0.41	68.78	0.47	60.03	0.58	57.13	0.44
12	76.44	0.39	31.32	2.41	75.61	1.13	70.51	0.23	62.15	0.64	55.44	2.09
13	75.31	0.20	29.50	0.51	75.66	0.24	69.35	0.21	60.91	0.49	55.29	0.72
14	73.66	0.27	28.14	0.33	75.86	0.22	69.09	0.61	61.77	0.16	58.39	1.97
15	72.93	2.01	35.16	1.05	76.60	0.98	68.40	2.03	61.33	0.86	55.12	0.28

Table 68: Results of the collaborative trial.
(ISO 5725-5, single point calibration based on PTT peak)

	Sample A		Sample B		Sample C		Sample D		Sample E		Sample F	
Lab code	PTT %	SD %	PTT %	SD %	PTT %	SD %	PTT %	SD %	PTT %	SD %	PTT %	SD %
1	71.96	0.43	29.65	1.13	77.26	0.24	70.88	0.43	63.42	1.06	57.13	0.57
2	77.04	3.58	30.92	0.11	79.11	1.01	76.49	3.04	64.21	1.95	61.65	2.98
3	73.93	0.56	28.86	0.37	76.56	2.68	76.80	0.96	61.56	1.19	55.59	1.24
4	74.12	4.21	29.46	0.33	76.91	0.18	79.97	1.83	65.95	1.66	60.61	2.37
5	74.99	0.27	29.02	0.23	77.26	1.23	70.61	0.97	63.13	0.26	58.76	0.32
6	72.63	0.79	28.79	0.39	76,41	1.14	70.50	0.53	64.17	0.81	58.40	1.50
7	71.66	0.82	29.12	0.32	76,87	1.02	69.62	1.33	71.09	2.01	58.58	1.09
8	70.36	8.78	28.46	1.45	67,66	1.30	70.70	2.79	64.54	5.51	59.28	5.61
9	71.25	5.21	39.21	1.06	75,83	0.71	74.53	0.32	67.83	2.13	64.17	2.09
10	70.34	0.42	29.64	0.84	75,68	0.84	76.11	1.57	61.07	0.86	58.66	0.36
11	76.05	0.25	33.89	1.88	77,67	3.22	71.69	0.62	64.29	1.84	58.79	1.99
12	74.53	0.23	28.23	0.48	73.10	0.64	68.87	0.92	65.50	0.21	62.85	1.89
13	74.43	0.11	28.55	0.79	76.49	0.31	70.08	1.11	63.09	0.44	58.40	0.52
14	71.75	0.68	28.63	0.53	76.36	0.82	71.86	0.57	63.81	0.82	57.29	2.09
15	76.50	2.72	27.10	0.48	73.98	0.47	70.61	2.31	61.72	1.28	58.02	2.15

Table 69: Results of the collaborative trial.
(ISO 5725-5, single point calibration based on PET peak)

	Sample A		Sample B		Sample C		Sample D		Sample E		Sample F	
Lab code	PTT %	SD %	PTT %	SD %	PTT %	SD %	PTT %	SD %	PTT %	SD %	PTT %	SD %
1	71.24	2.74	27.20	2.50	77.01	0.92	69.48	1.01	63.70	0.74	57.77	0.30
2	67.87	1.05	25.94	1.73	74.15	0.45	68.66	1.54	60.32	3.11	54.72	2.52
3	79.91	0.92	29.60	1.40	74.47	1.03	69.12	0.22	64.95	0.63	61.51	0.42
4	75.50	2.11	23.00	2.36	74.48	0.19	68.07	0.63	58.19	1.44	57.62	2.50
5	72.97	0.36	28.53	0.30	75.47	0.04	69.67	0.28	61.15	0.48	56.34	0.10
6	78.79	0.59	31.40	1.71	75.33	0.41	70.09	0.09	61.40	0.32	62.59	0.23
7	79.29	1.89	30.12	0.89	77.50	1.36	71.71	0.60	69.20	1.22	62.26	1.92
8	76.73	2.86	30.87	3.66	77.32	0.48	73.89	0.22	65.29	0.86	63.76	2.40
9	71.20	5.82	51.35	1.05	74.67	0.77	68.16	2.30	62.95	0.65	58.09	3.49
10	79.64	0.41	29.13	2.64	76.47	0.55	66.62	0.59	61.71	0.29	61.61	0.36
11	74.67	0.17	38.90	2.19	71.21	0.40	69.12	0.46	59.61	0.58	57.46	0.44
12	76.55	0.28	29.28	2.48	76.17	1.11	70.83	0.08	61.28	0.66	55.33	2.10
13	75.85	0.19	29.34	0.19	75.80	0.16	69.05	0.07	60.17	0.28	55.70	0.71
14	73.79	0.16	28.71	0.32	75.66	0.11	69.62	0.43	62.42	0.05	63.98	1.71
15	78.73	0.70	35.23	1.00	76.07	0.93	66.91	0.76	60.67	0.38	55.46	0.28

Table 70: Results of the collaborative trial.
(ISO 5725-5, multipoint calibration)

	ISO 5725-5	A	B	C	D	E	F
	N. of labs	15	15	15	15	15	15
	PTT (MS), %	74.09	28.5	76.04	70.32	62.91	
PTT peak	PTT (DSC), %	74.88	29.21	76.10	71.62	64.02	59.38
	Bias, %	0.79	0.71	0.06	1.30	1.11	
	Conf. limit, %	1.05	0.91	0.86	0.60	1.07	0.73
	r, %	3.10	2.09	2.85	3.64	4.17	5.31
	R, %	7.63	3.57	8.16	4.86	4.19	5.31
	HORRAT	1.78	1.87	1.86	1.18	1.13	1.50
	N. of labs with z-score > |2|	3	5	5	3	4	1
PET peak	PTT (DSC), %	73.60	29.69	75.34	69.14	61.43	56.18
	Bias, %	-0.49	1.19	-0.70	-1.18	-1.49	
	Conf. limit, %	1.18	2.14	0.52	0.61	0.78	0.66
	r, %	4.46	5.34	2.13	2.01	2.55	4.77
	R, %	6.84	11.94	4.44	4.10	4.85	5.22
	HORRAT	1.59	6.25	1.01	1.00	1.30	1.54
	N. of labs with z-score > |2|	2	8	1	1	3	2

Table 71: Results of the collaborative trial.
(ISO 5725-5, single point calibration)

	ISO 5725-5	A	B	C	D	E	F
	N. of labs	15	15	15	15	15	15
	PTT (MS), %	74.09	28.5	76.04	70.32	62.91	
PTT peak	PTT (DSC), %	73.44	29.26	76.24	72.46	64.07	59.06
	Bias, %	-0.65	0.76	0.20	2.14	1.16	
	Conf. limit, %	1.00	0.90	0.85	1.04	0.88	0.86
	r, %	3.19	2.09	2.83	3.86	4.14	5.28
	R, %	7.12	3.59	4.76	9.53	6.37	6.62
	HORRAT	1.66	1.88	1.09	2.32	1.71	1.87
	N. of labs with z-score > |2|	2	4	1	5	3	4
PET peak	PTT (DSC), %	75.66	30.09	75.61	69.27	61.97	58.95
	Bias, %	1.57	1.59	-0.43	-1.05	-0.94	
	Conf. limit, %	1.16	2.01	1.64	0.57	0.84	1.06
	r, %	3.49	5.29	1.97	1.63	2.02	4.57
	R, %	10.50	11.08	3.88	4.76	6.93	10.71
	HORRAT	2.45	5.80	0.88	1.16	1.86	3.02
	N. of labs with z-score > |2|	6	8	1	3	4	11

Tables 70-71 contain the bias, the confidence limit at 95 % probability, the repeatability limit r, the reproducibility limit R, the HORRAT parameter and the number of laboratories showing z-score higher than 2.

Biases equal or lower than 1 % indicated that the DSC method was accurate in terms of trueness. In fact, when the composition of a binary mixture is determined both via one of the validated chemical dissolution method and the manual separation (considered a reference method), the bias, which is the difference between the two results, is usually below 1 %. At the same time, confidence limits, at 95 % probability, lower than 1 % were considered acceptable, because this is the confidence limit shown by the majority of the validated chemical dissolution methods described in the EU regulation 1007/2011, the other methods in the regulation show a confidence limit not greater than 2 %.

The Horwitz ratio, called HORRAT parameter, is a normalised performance parameter that indicates the acceptability of a method of analysis with regards to the reproducibility, which is the among-laboratory precision. The HORRAT value is equal to the ratio of the observed coefficient of variation among laboratories, calculated from the data of the collaborative trial, to the corresponding predicted coefficient of variation calculated with the Horwitz equation [14]:

$$PCV_R(\%) = 2 * C^{-0.15} \qquad 7.4.1$$

where C is the concentration found, expressed as a mass fraction. The importance of the Horwitz ratio is that it is almost independent from analyte, matrix and method. It is for this reason often used as criteria of acceptability for analytical methods. The precision of the method is better than expected if the ratio is lower than 1 and poorer if it greater than 1. The empirical interpretation of the HORRAT parameter considers that the precision of a method, in terms of reproducibility, is acceptable if this parameter is in the range 0.5 – 2. At the same time, values lower than 0.5 may be due to unreported averaging or excellent training and experience, while values higher than 2 may indicate inhomogeneity of test samples, need for further method optimisation or training, operating below the limit of determination or an unsatisfactory method.

According to the statistic evaluation with ISO 5725-5, the best results in terms of bias, confidence limit and HORRAT parameter were obtained when the quantification was done with multipoint calibration curves based on the integration of PTT melting peak. In fact, in this case, the biases calculated for the five samples **A-E** were at most 1.30 %, the confidence limit at most 1.07 % and the HORRAT values always between 0.5 and 2. When multipoint calibration curves were based on PET peak, the HORRAT value for sample **B** was as high as 6.25, the highest confidence limit and bias were equal to 2.14 % and -1.49 %, respectively. The comparison of results obtained using multipoint calibration curves and single point calibrations showed that the quantification carried out on the basis of a single point is less reliable, in particular when the peak integrated is the one of PET. In this case, for three out of six samples the calculated Horwitz ratio was much greater than 2 and the four out of six confidence limits were higher than 1 %.

For comparison purposes, the data obtained with multipoint calibration curves based on PTT peak were also treated according to Part 2 of ISO 5725. This Part foresees a specific procedure for the identification of outliers. The consistency statistic, Mandel h, and the within-laboratory consistency statistic, Mandel k, have to be calculated for each laboratory. Plots of h and k have to be prepared and evaluated. Test results need to be initially analysed using the Cochran's test in order to identify exceeding intra-laboratory standard deviations. Then, Grubbs' test has to be successively applied for the outlier identification of individual test results (if $n > 2$) and laboratory mean values. The item tested (respectively standard deviation or mean) is:

- accepted as correct if the test statistic is less than or equal to its 5 % critical value;
- called "straggler" if the test statistic is comprised between 5 % and 1 % of the critical value;
- called "statistical outlier" if the test statistic is greater than its 1 % critical value.

Straggler data have to be marked by a single asterisk and statistical outlier with a double asterisk. The reasons for straggler and statistical outlier need to be investigated and, if possible, wrong data replaced by the correct values. In case stragglers and/or statistical outliers cannot be explained or rejected as belonging to an outlying laboratory, the stragglers have to be retained as correct items and the statistical outliers have to be discarded (unless the statistician has good reasons to retain them).

Laboratories' performances in terms of mean values and variances were assessed through the Mandel's h and k statistics. The resulting plots are reported in Annex VII. The plot of Mandel's k showed that Laboratory 08 had some problems regarding the repeatability of data. In fact, the laboratory was considered a Mandel's k outlier (k**) for the variance for three out of six samples, and a straggler (k*) for other two samples. Considering Mandel's h plot, Laboratory 09 was considered an outlier (h**) in two cases out of six and a straggler (h*) in one case. The approach recommended in ISO 5725-2 is to keep all data except the ones considered statistical outlier for either Cochran's or Grubbs' tests, so no exclusion was decided on the basis of Mandel's plots. In Table 72 are reported the outliers and stragglers identified according to Mandel's h and k statistics, and Cochran's and Grubbs' tests.

Table 72: Statistical outliers and stragglers according to Mandel's h and k, Cochran and Grubbs.

Lab	A	B	C	D	E	F
1					h**,G*	
2	h*			k*		
3			k*			
4		G**		h*		
5						
6						
7					h*	
8	k**,C**	k*	h*	k*	k**,C**	k**,C**
9	k*,C**	h**,G**		h*		h**,G*
10						
11		k*	k**,C*			
12						
13						
14						
15						

Finally, again for comparison purposes, the data obtained with multipoint calibration curves based on PTT peak were also treated according to Part 2 of ISO 5725, after the elimination of all data from Laboratories 08 and 09, which were rejected as outliers for the variance and the mean, respectively, on the basis of Mandel's h and k statistics.

As reported in Table 73, a good agreement was observed among results obtained with ISO 5725-5, Part-2 and with Part-2 excluding the data from Laboratories 08 and 09. This implies that all the three approaches were substantially equivalent. The values are highlighted in yellow when higher than 1 in the case of the bias and confidence limit, and 2 for the HORRAT parameter.

Table 73: Results of the collaborative trial according to various approaches. (multipoint calibration based on PTT peak)

		A	B	C	D	E	F
ISO 5725-5		0.79	0.71	0.06	1.30	1.11	
ISO 5725-2	**bias, %**	1.24	0.34	-0.02	1.42	1.12	
ISO 5725-2 without L08, L09		1.24	0.28	0.17	1.16	0.73	
ISO 5725-5		1.78	1.87	1.86	1.18	1.13	1.50
ISO 5725-2	**HORRAT**	1.78	1.79	1.83	1.37	2.63	1.70
ISO 5725-2 without L08, L09		1.75	1.76	1.60	1.23	2.44	1.38
ISO 5725-5		1.05	0.91	0.86	0.60	1.07	0.73
ISO 5725-2	**confidence limit, %**	0.87	0.39	0.86	0.60	1.08	0.68
ISO 5725-2 without L08, L09		0.87	0.41	0.81	0.59	1.05	0.57
ISO 5725-5		3.10	2.09	2.85	3.64	4.17	5.31
ISO 5725-2	**r, %**	4.08	2.26	3.62	4.09	3.73	4.75
ISO 5725-2 without L08, L09		4.08	2.03	3.71	3.81	3.49	4.66
ISO 5725-5		7.63	3.57	8.16	4.86	4.19	5.31
ISO 5725-2	**R, %**	7.61	3.42	8.03	5.62	9.82	6.09
ISO 5725-2 without L08, L09		7.61	3.39	7.05	5.12	9.22	4.91

In order to take into consideration all data, it was decided to present the final results of the collaborative study, in terms of method precision, according to the approach described in Part-5. In Table 74 are reported the final results of the collaborative trial using multipoint calibration curves based on PTT melting peak. In Annex VII the graphic illustrations of all results are reported.

As can be seen in Figure 27, no apparent relationship between the bias values and the PTT content was observed: the two lowest biases correspond to either the samples with the highest or the lowest PTT content.

Table 74: Results of the collaborative trial.
(ISO 5725-5, multipoint calibration based on PTT peak)

ISO 5725-5	A	B	C	D	E	F
Labs	15	15	15	15	15	15
z-score > |2|	3	5	5	3	4	1
PTT, %	74.88	29.21	76.10	71.62	64.02	59.38
bias, %	0.79	0.71	0.06	1.30	1.11	
conf. limit, %	1.05	0.91	0.86	0.60	1.07	0.73
SD r, %	1.12	0.75	1.03	1.32	1.51	1.92
CV r, %	1.49	2.58	1.35	1.84	2.35	3.23
r, %	3.10	2.09	2.85	3.64	4.17	5.31
SD R, %	2.75	1.29	2.95	1.76	1.51	1.92
CV R, %	3.68	4.41	3.87	2.45	2.36	3.23
R, %	7.63	3.57	8.16	4.86	4.19	5.31
HORRAT	1.78	1.87	1.86	1.18	1.13	1.50

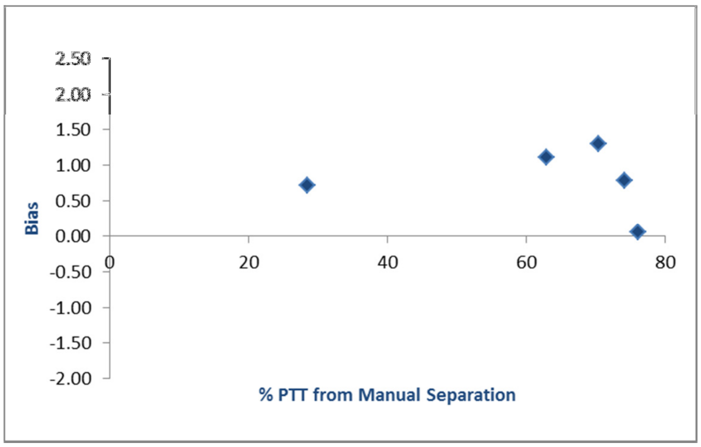


Figure 27: Relationship between bias and PTT content (%).

In conclusion, the DSC method for the quantification of PTT in mixtures with PET was considered acceptable, fit-for-purpose and fully validated as it gave good results in terms of bias (below 1.3 % for all samples), confidence limit (always below 1.1 %) and HORRAT (always below 2). Repeatability and reproducibility limits, however, pointed out that there is room for improvement. For this reason the performances of participating laboratories were evaluated in terms of z-scores according to ISO 13528 [17] and the International Harmonised Protocol [12].

The z-scores compared the participants' deviation from the assigned value with the standard deviation obtained from the precision experiment. The usual interpretation of z-scores considers values above 3.0 or below -3.0 as unsatisfactory, and above 2.0 or below -2.0 as questionable. The z-scores per laboratory and sample are shown in Table 75 and 76, and the graphical representation is reported in Annex VII. The values are highlighted in red when higher than 3, and in yellow if higher than 2 but lower than 3.

Z-score values showed that Laboratories 5, 6, 10 and 13 were the ones which performed best (all |z| < 1). On the contrary, Laboratories 02, 04 and 09 were among the least performance, with Laboratory 9 being the one presenting the worst performance.

Table 75: z-scores per laboratory and sample.

Lab	A	B	C	D	E	F
1	-0.731	1.530	2.236	1.389	-5.618	-0.314
2	3.911	2.300	2.091	-0.198	0.507	0.135
3	2.701	1.506	-0.686	0.083	0.796	-1.862
4	-0.634	9.368	0.726	3.030	2.056	0.823
5	1.363	-0.01	0.477	0.279	0.455	-0.198
6	0.946	0.673	0.36	0.092	0.588	-0.149
7	-0.955	1.060	0.435	0.245	5.564	-1.023
8	-1.795	1.409	-3.418	1.061	1.401	-1.028
9	-0.17	14.504	1.853	3.151	4.556	3.526
10	0.215	1.704	0.404	1.531	0.336	-0.082
11	2.608	-2.803	-1.346	0.328	0.726	-1.209
12	-1.389	0.198	-2.278	-0.333	0.813	1.456
13	1.430	-0.092	0.766	0.465	0.425	-0.477
14	0.884	1.099	0.692	2.012	0.452	-0.88
15	0.082	-2.227	-2.499	1.272	-0.074	0.254

Table 76: z-scores per laboratory.

Lab	z>I3I	2<IzI>3	z<I2I
1	1	1	4
2	1	2	3
3	0	1	4
4	2	1	3
5	0	0	6
6	0	0	6
7	1	0	5
8	1	0	5
9	4	0	2
10	0	0	6
11	0	2	4
12	0	1	5
13	0	0	6
14	0	1	5
15	0	2	4

8. Conclusions

The experimental work conducted at the JRC confirmed that test methods are available for the identification and quantification of the PTT fibre when in mixtures with other fibres. The conclusions of this work were presented, discussed and agreed upon with the members of the ENNETL network on 4th October 2013.

Regarding the identification, pure samples of PTT and other types of polyesters (PET and PBT) can be distinguished with FT-IR, whereas the melting point determined by DSC can be used to differentiate PTT from PET, but not from PBT. However, based on their crystallization peaks all the three polyesters can be differentiated. On the contrary, optical microscopy is not an adequate method to identify PTT, as it cannot be differentiated neither via longitudinal nor via cross-section view from the other polyesters. Solubility properties can be used only to confirm the polyester nature of the fibre.

When measured at 25 % elongation, PTT showed quite good elastic recovery (65.7 - 78.1 %, corresponding to a permanent deformation of 5.4 - 8.8 %), but the fibre could not be considered elastic essentially because of its moderated elongation at break (usually in the range 33 – 64 % elongation).

Concerning quantification, the pre-treatment described in the EU Regulation 1007/2011 is applicable to the PTT fibre. The *agreed allowance* (humidity regain) of PTT and its correction factor for mass loss during pre-treatment were experimentally evaluated and the values, proposed by the JRC and accepted by the network of national experts from Member States, are 1.50 % and 0 %, respectively.

The solubility properties of the PTT fibre were evaluated with all methods described in the EU Regulation 1007/2011 (with the exception of method 12, concerning the organic nitrogen content). The chemical dissolution methods 1-11, 13, 14 and 16 can be applied to mixtures containing PTT. The d correction factors were established by the JRC on the basis of 20 replicates. PTT was completely insoluble in methods 2, 3, 7 and 11 (d = 1.00) and it could be considered insoluble in methods 1, 4, 5, 9 and 10 (d = 1.01), in method 13 (d = 1.02) and in methods 6, 8 and 16 (d = 1.03). Method 15 cannot be used to quantify blends containing PTT as it is partially soluble in these conditions ($d = 1.05$). Finally, PTT is completely soluble in method 14. In addition, as the elastane d correction factors were absent in the EU Regulation 1007/2011, the ones that were needed in this work were determined. Elastane proved to be soluble in methods 3, 7, 8 and 14 and insoluble in method 4, with a d correction factor of 1.02.

When feasible, manual separation was performed to quantify binary and ternary mixtures. Reference values were thus obtained and used to evaluate the trueness of gravimetric and DSC methods. The quantification results, obtained by the JRC and the petitioner, were in excellent agreement, confirming that the manual separation is a very accurate, repeatable and reproducible method.

Binary mixtures of PTT and PET could not be quantified by any of the chemical methods due to their chemical similarity. The quantification of binary mixtures PTT/cotton, PTT/elastane, PTT/polyamide and PTT/wool were performed using the appropriate methods. In two cases, samples **306** (PTT/polyamide) and **322** (PTT/wool), the comparison between quantification done *via* manual separation and methods 4 and 2, respectively, could be made and a very good agreement was shown. In the case of the ternary mixtures, the accuracy of the chemical methods used for quantification (methods 4, 7 and 8) could not be measured, because manual separation was not feasible. In addition, one ternary mixture could not even be quantified through chemical methods, as PET and PTT cannot be distinguished by them.

DSC was used to quantify PTT/ PET blends. Initial results revealed that various pure PTT samples, produced in different production plants and periods of time, showed different fusion enthalpies, both on the first and on the second heating cycle. This is most probably the reason why the quantification carried out with multipoint calibration (an independent calibration, prepared with fibres not directly separated from the samples under investigation) based on PTT melting peak provided inaccurate results, as confirmed by bias values up to 3.5 %.

Three approaches were used to try to solve this problem. The first attempt was done by varying the cooling rate between the first and the second heating cycle, in order to either give time to the melted sample to form crystals or to let it solidify in an amorphous phase. The second attempt consisted in applying a heat-treatment to the samples before the DSC analysis (annealing), in order to erase the thermal history of the samples and homogenise as much as possible the crystallisation degree of samples. The third and last attempt to develop a DSC quantification method based on independent calibration curves trying to obtain PTT samples in an amorphous phase by melting them, either in a muffle or in a ventilated oven for a given time, and then quenching them with liquid nitrogen to achieve a very fast cooling rate. The influence of the temperature and time of the heat treatment, as well as the time of the quenching step with liquid nitrogen were studied.

The results obtained with these approaches clearly showed that it is impossible to develop an accurate DSC quantitative method using calibration curves prepared with any kind of independent PTT and PET yarns. The use of calibration curves built up with PTT and PET yarns manually separated from each fabric sample under evaluation proved to be the only possibility to obtain satisfactory results in terms of accuracy, including both trueness and precision. In fact, in this way both the pure fibres used for the calibration points and the corresponding blend to be quantified have the same thermal history.

The DSC method was developed and in-house optimised, taking into consideration the sample quantity, the temperature programme, the type of integration and calibration curve to be used. The influence on quantitative results of five types of integration was studied, as well as the influence of using multipoint or single point calibrations based on PTT or PET melting peaks. Analyses were carried out on four PTT/PET blends in four replicates and in three different days. The repeatability and the within laboratory reproducibility were evaluated, both in the JRC and DuPont's laboratories. The outcome of these tests highlighted the strong influence that the integration approach used can have on final results: the best results in terms of trueness (bias) were obtained using linear integration, both in the case of multipoint and single point calibrations. In these cases the bias was usually lower than 2 %. The DSC method was further optimised, with the main conditions being the use of a range of sample between 5 and 8 mg, temperature programme (150 °C – 5 °C/min – 300 °C), nitrogen atmosphere (50 ml/min of flow), piercing the pans lids to allow samples to be in contact with the inert atmosphere and an integration of the PTT melting peak on the first (and unique) heating cycle. The analyses were performed on four PTT/PET mixtures in six different days, applying five types of integration and four calibrations for each sample (multi and single point based on PTT and PET melting peak). These results proved the excellence of method, with biases values always lower than 2 % and in many cases even lower than 1 %. On the basis of these excellent results, the linear fixed range integration was included in the SOP of the method that was then submitted to the validation exercise.

The collaborative study was organised following the rules of ISO 5725-2:1994. It was planned as a balanced uniform–level experiment. Six PTT/PET binary mixtures with different concentrations of PTT were quantified and fifteen laboratories took part in the exercise, twelve from Member States, one from United States and two from the JRC. The accuracy of the quantification results using multipoint and single point calibration curves based on both PTT and PET peaks was evaluated and compared. The homogeneity of each sample was evaluated and confirmed according to the test for 'sufficient homogeneity' included in the IUPAC harmonised protocol for proficiency testing.

The statistical analysis of the test results received from all laboratories was conducted using the robust method described in ISO 5725-5, which does not exclude any outlier but calculates parameters so that they are not influenced much by data of low quality. PTT content, confidence limit, bias, limits of repeatability and reproducibility and HORRAT parameter were calculated for each sample considering the four types of calibration curves (multipoint and single point calibration curves based on PTT and PET enthalpies of fusion). The obtained results were comparable to those calculated using the classical statistical approach described in ISO 5725-2.

The best quantification results, considering bias, confidence limit and HORRAT parameter, were obtained when the quantification was done with multipoint calibration curves based on the integration of PTT melting peak. In this case, the biases varied between 0.06 and 1.30 %, the confidence limit between 0.60 and 1.07 % and HORRAT parameter values were in the range 0.5 - 2. Consequently, this approach will be the only one described in the SOP of the method. The outcome of the validation study in terms of trueness, within and between laboratories precision for each level is summarised in the following table.

	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
PTT, %	29.21	59.38	64.02	71.62	74.88	76.10
bias, %	0.71	nd	1.11	1.30	0.79	0.06
r, %	2.09	5.31	4.17	3.64	3.10	2.85
R, %	3.57	5.31	4.19	4.86	7.63	8.16

These results proved that the method is accurate in terms of both trueness and precision. No relation was found between the bias and the corresponding PTT content. Experts of the ENNETL network agreed that the optimised DSC method for the quantification of PTT in mixtures with PET can be considered fully validated as for each level more than eight valid results were obtained. However, the evaluation of results pointed out that it would be more appropriate to use a linear instead of linear fixed range integration. Experts agreed on this point, consequently the SOP will be changed accordingly.

As shown by the analysis of the z-scores evaluated for each laboratory, the values of repeatability and reproducibility limits could be improved enhancing the performance of laboratories. This is understandable considering that several participants were not much experienced in DSC analysis, in general, and in the method under validation, in particular.

The ENNETL network confirmed the PTT's definition proposed by the applicant: *“fibre formed of linear macromolecules comprising at least 85 % (by mass) in the chain of an ester of 1,3-propane diol and terephtalic acid”*. The experts agreed that the name proposed by the petitioner (*triexta*) cannot be accepted as it does not reflect the chemical nature of the fibre. During the 13th ENNETL meeting the name proposed by the applicant (*triexta*) was rejected by the experts. Three alternative names were suggested: polytrimethylene terephthalate, polytrimethylester and polytriester, but no consensus was reached.

9. References

[1] Directive 2008/121/EC of the European Parliament and of the Council of 14 January 2009 on textile names (recast) *(Official Journal L019 of 23.1.2009 p. 0029-0048)*.

[2] EU Regulation 1007/2011 of the European Parliament and of the Council of 27 September 2011 on textile fibre names and related labelling and marking of the fibre composition of textile products and repealing Council Directive 73/44/EEC and Directives 96/73/EC and 2008/121/EC of the European Parliament and of the Council *(Official Journal L272 of 18.10.2011 p. 0001-0064)*.

[3] Wu, T., Li, Y., Wu, Q., Song, L., Wu, G., Thermal analysis of the melting process of poly-(trimethylene terephthalate) using FTIR micro-spectroscopy, *European Polymer Science*, 2005, **41**, 2216 – 2223.

[4] Donelli, I., Freddi, G., Nierstrasz, V., Taddei, P., Surface structures and properties of poly-(ethylene terephthalate) hydrolysed by alkali and cutinase, *Polymer Degradation and Stability*, 2010, **95**, 1542 – 1550.

[5] Test methods for bare elastane yarns, BISFA, 1998.

[6] EN 15930 (2010). Fibres. Elasticity of fibres. Test methods.

[7] ISO 1973 (1995). Textile fibres - Determination of linear density - Gravimetric method and vibroscope method. International Organisation for Standardisation, Geneva, Switzerland.

[8] ISO 6348 (1980). Textiles. Determination of mass. Vocabulary. International Organisation for Standardisation, Geneva, Switzerland.

[9] ISO 5725-2 (1994). Accuracy (trueness and precision) of measurement methods and results. Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method. International Organization for Standardisation, Geneva, Switzerland.

[10] ISO 5725-1 (1994). Accuracy (trueness and precision) of measurement methods and results. Part 1: General principles and definitions. International Organization for Standardisation, Geneva, Switzerland.

[11] Miller, J. N., Miller, J. C., Statistics and Chemometrics for Analytical Chemistry. Pearson Education Limited, .5th Ed, 2005.

[12] Thompson M., Ellison S. L. R., Wood R., The international harmonized protocol for the proficiency testing of analytica chemistry laboratories (IUPAC Technical Report), *Pure Appl. Chem.*, 2006, **78 (1)**, 145.

[13] Shapiro S. S., Wilk M. B., An analysis of variance test for normality (complete samples), *Biometrika*, 1965, **52 (3-4)**, 591.

[14] Horwitz W., Albert R., The Horwitz Ratio (HorRat): a useful index of method performance with respect to precision, *J. of AOAC International*, 2006, **89 (4)**, 1095.

[15] ISO 5725-5 (1998). Accuracy (trueness and precision) of measurement methods and results. Part 5: Alternative methods for the determination of precision of a standard measurement method. International Organization for Standardisation, Geneva, Switzerland.

[16] ProLab Software, QuoData, Drezden – www.quodata.de

[17] ISO 13528 (2005). Statistical methods for use in proficiency testing by inter-laboratory comparisons. International Organization for Standardisation, Geneva, Switzerland.

Annex I

Analytical methods proposed by the applicant

4. Identification of PTT Polymers, Fibers, and Fabrics

Another requirement for the designation of a new generic classification is that the new material must be identifiable, qualitatively and quantitatively, on its own, as well as in binary and ternary systems. Again, PTT meets this requirement with ease. A number of analytical techniques requiring varying levels of technology are available to identify PTT and distinguish it from other materials. In order to exhibit how this process can be performed, several examples will be presented. The samples shown here are given as examples and not in anyway intended to be equally applicable to other samples or testing environments. Information shared here is unique to the samples and test methods employed.

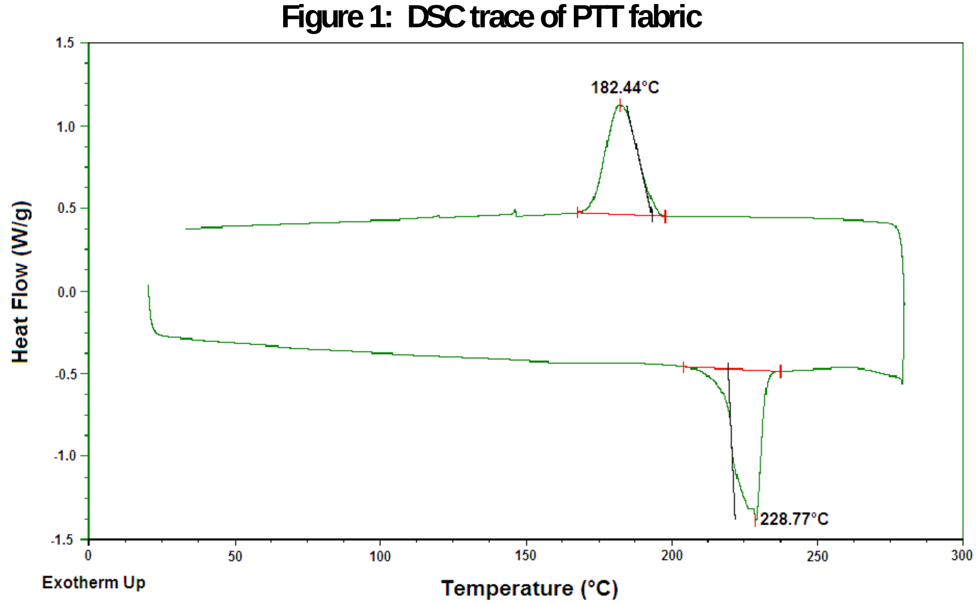
4.1 PTT in a Unary Sample

To begin with, the analytical results for a pure PTT fabric sample are presented.

4.1.1 Differential Scanning Calorimetry (DSC)

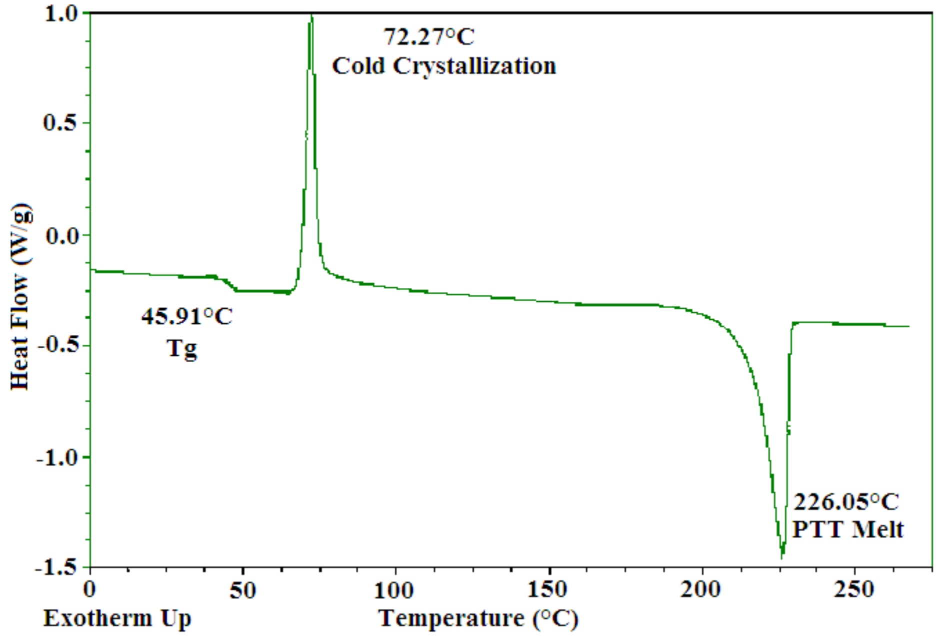
DSC works by measuring the heat absorbed or released by the sample as the temperature is slowly increased to a maximum temperature and then allowed to cool. By carefully reviewing the endotherms and exotherms in the DSC trace for the sample, conclusions can be made about the composition and transitions of the materials present. For the DSC experiment, a small sample of this fabric will be cut out so that all fabric components are included. A DSC trace of a PTT fabric sample is shown in Figure 21.

Figure 1: DSC trace of PTT fabric



As indicated on the DSC trace, PTT fiber has a melting point of 228.8°C. It shows a crystallization peak from melt at 182°C. PTT fibers, since they are already crystallized, do not exhibit the distinct cold crystallization seen in amorphous PTT samples. A distinct glass transition and cold crystallization can be seen for a PTT fiber sample, however, if the DSC sample is quenched with liquid nitrogen immediately following the heating cycle. This quenched sample becomes amorphous because the rapid cooling inhibits the formation of crystals. The reheat DSC trace for a PTT sample treated in this way is shown in Figure 22. The glass transition temperature, T_g, is very distinct for PTT and can be used as a reliable measurement tool in its identification and characterization.

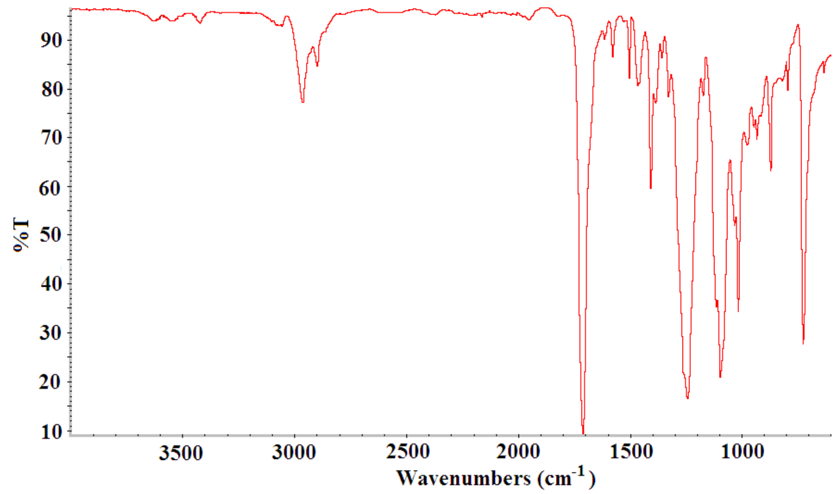
Figure 2: DSC trace for the reheat of an amorphous PTT fiber after a first heat and quench



4.1.2 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy is another powerful analytical tool that can be used to distinguish between PTT and other fibers, including PET. FTIR is a type of spectroscopic analysis in which infrared light is directed at a sample and either the reflected or transmitted light from the sample is detected. For fabrics, attenuated total reflectance IR (ATR-IR) is an effective technique. Based on the different bond types, orientations, and functional groups present, particular light bands are absorbed by the sample. This process generates an absorbance spectrum that can be used as a fingerprint for identification of the sample. The full FTIR spectrum for PTT is shown in Figure 23.

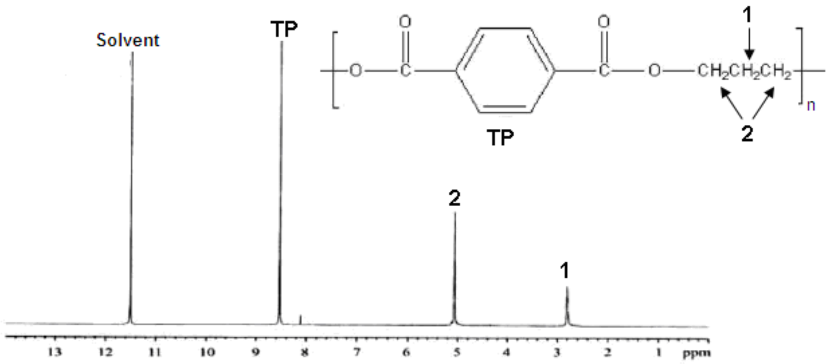
Figure 3: Full FTIR spectrum for PTT



4.1.3 Nuclear Magnetic Resonance (NMR)

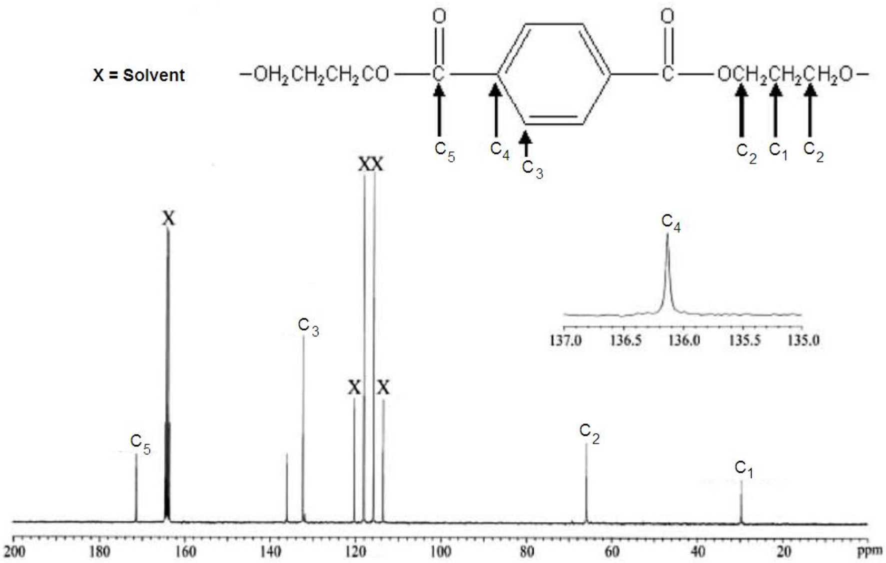
Proton and carbon NMR spectroscopies are two additional methods by which PTT fibers may be identified. The ^{1}H-NMR spectrum for PTT is given in Figure 24. Deuterated trifluoroacetic acid was used as the solvent to take this spectrum. The resonance for this solvent was set to 11.50 ppm. The resulting chemical shifts for PTT were 8.53 ppm for the protons on benzene, 5.06 ppm for the protons α to the carbonyl, and 2.80 ppm for the protons β to the carbonyl.[1]

Figure 4: ^{1}H-NMR for PTT[1]



Just as proton NMR can be used to identify PTT, carbon-13 NMR can also be employed for this purpose. Figure 25 shows the carbon-13 spectra for PTT. The shift assignments for PTT are 29.70 ppm for the middle carbon and 65.93 ppm for the two end carbons of the propanediol portion of the polymer, 132.37 ppm for the aromatic alkyl carbons of the benzene ring, 136.13 ppm for the aromatic quaternary carbons of the benzene ring, and 171.51 ppm for the carbonyl carbon atom.[1]

Figure 5: ^{13}C-NMR of PTT[1]



4.1.4 Intrinsic Viscosity (IV)

Intrinsic viscosity is the ratio of a solution's specific viscosity to the concentration of the solute extrapolated to zero concentration, as shown in Equation 1.

Equation 1: Definition of intrinsic viscosity

$$[\eta] = \lim_{c \to 0} \eta_{red}$$

Where $[\eta]$ = intrinsic viscosity
η_{red} = specific viscosity
c = concentration in g

Inherent viscosity, another measurement common for polymer identification, is defined as the ratio of the natural logarithm of the relative viscosity, η_{rel}, to the mass concentration of the polymer, c, as shown in Equation 2.

Equation 2: Definition of inherent viscosity

$$[\eta]_{inh} = \frac{\ln \eta_{rel}}{c}$$

Viscosity behavior of macromolecular substances in solution is one of the most frequently used approaches for polymer and fiber characterization. IV is measured by dissolving a polymer or fiber in a solvent and determining the flow time of a set volume of the solution through a given length of a capillary tube. Since different polymers tend to have different IVs, this relatively simple method can be an effective tool in the identification and differentiation of polymers. Table 9 shows the intrinsic viscosity of PTT and PET polymer pellets as measured by a method similar to ASTM D2857.95. A 0.92 dL/g IV PTT has about the same molecular weight of a 0.64 dL/g IV PET. Fiber grade PTT polymer pellets typically have an IV in the range 0.90-1.02 dL/g, very different than that of PET, which is 0.64 dL/g for textile fiber applications. While IV does depend on manufacturing characteristics, which can introduce some variability in IV, this method is widely used and can serve well in supporting other analytical methods used to identify a polymer, fiber, or fabric. This is an important distinction that will impact PTT polymer processors.

Table 1: Typical intrinsic viscosities of PTT and PET pellets used for making textile fibers

Polymer	IV (dL/g)
PTT	1.02
PET	0.64

4.1.5 Density

Measuring density is another simple, yet effective, method of identifying polymeric materials. This method can be very helpful in the identification of PTT. To measure density, a sample of the polymer is floated in different solvents of known densities. A density gradient column is usually used. Based on where the sample comes to rest in the solvents, a density can be established for the material. There are a number of other techniques available for measuring densities of polymers and fibers as well. Table 10 shows the density of PTT and a number of polymers commonly used to make textile fibers.

Table 2: Densities of various polymers[2]

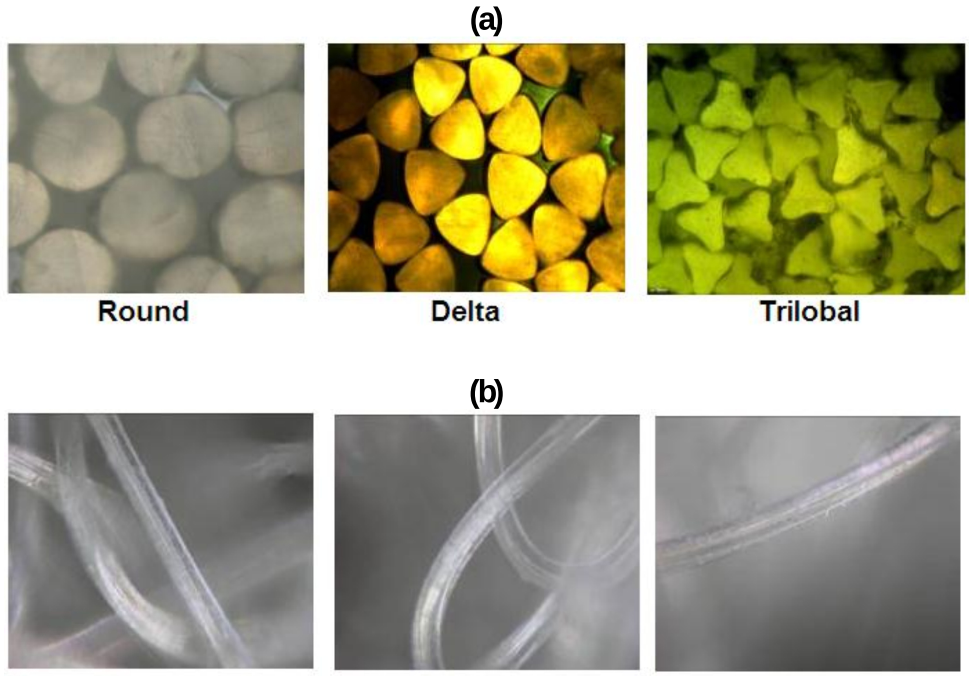
Polymer	Density (g/mL)
PTT*	1.33
PET	1.38-1.41
PBT	1.31
Nylon 6	1.12-1.15
Nylon 6,6	1.13-1.16

*****The density for PTT is not from reference 2 but was established experimentally by the Applicant.

4.1.6 Microscopic Inspection of Fibers

In many cases, microscopic observation of textile fibers can be used to aid in the identification of fibers. PTT fibers can be given a variety of cross section shapes. Several examples of fiber cross sections that are possible with PTT, as well as images of PTT filaments, are shown in Figure 26. While this figure does not contain all of the potential cross sections that PTT fibers can have, it does show a representative selection of what cross sections can be given to PTT. This information may be helpful in identifying a fiber, especially when more advanced techniques are not available.

Figure 6: Images of example (a) PTT fiber cross sections and (b) PTT filaments



4.1.7 Mechanical Property Analysis

As it was shown above, different fibers exhibit different mechanical properties. Therefore, stress-strain analysis can provide helpful information for identifying unknown fibers. The modulus, stress-strain curve shape, and elongation and tenacity at break all provide helpful information for fiber identification. A typical stress-strain curve for a PTT fiber is shown in Figure 27. Comparing the curve for an unknown fiber with that shown in Figure 26 could aid in its identification.

Figure 7: Stress-strain curve for PTT fiber

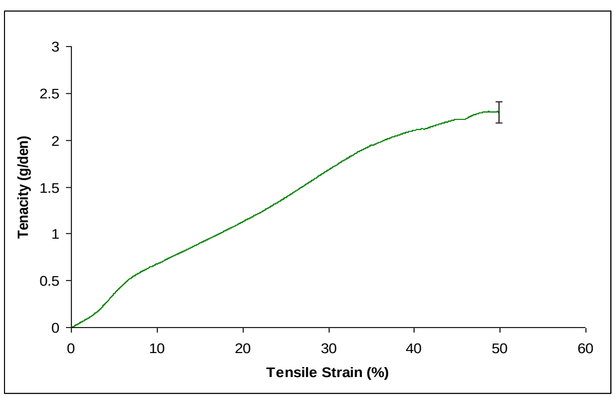
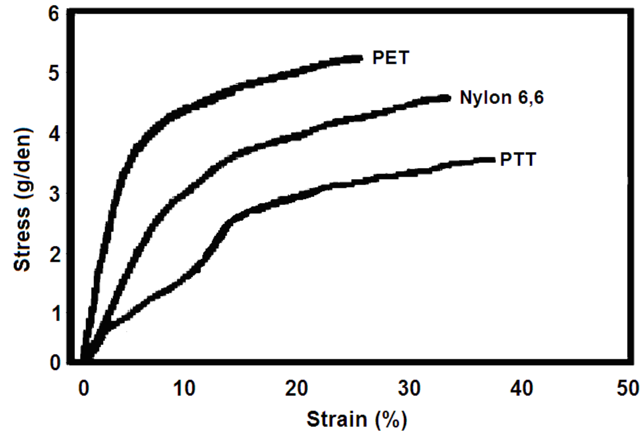


Figure 28 shows typical stress strain for PTT along with a number of other common textile fibers for reference and to show how distinct stress-strain curves are for different materials.

Figure 8: Stress-strain curves for various textile fibers Error! Bookmark not defined.



Fiber elastic modulus is one specific component of the mechanical properties of fibers that can be helpful in their identification. Observation of the elastic deformation region of the stress-strain curves in Figure 28 shows that these materials all have unique moduli. As listed above in Table 6, PTT has a modulus of 2.58 gN/m^2 while PET, in comparison, has a modulus of 9.15 gN/m^2. While it is a relatively simple method, determination of the mechanical properties of fibers can be very helpful in their identification.

4.1.8 Summary

By looking at the results of a variety of analytical tests, the pure PTT sample can readily be identified as such. Table 11 summarizes the analytical methods listed here, as well as a number of other tests that can be used to identify PTT and differentiate it from other materials. This same analytical approach can be taken to identify PTT in binary, ternary, and higher order multi-component systems as well. The following sections present examples of how such analyses would be carried out, along with information regarding the quantification of PTT present in the system.

Table 3: Summary of analytical tests to identify PTT fibers

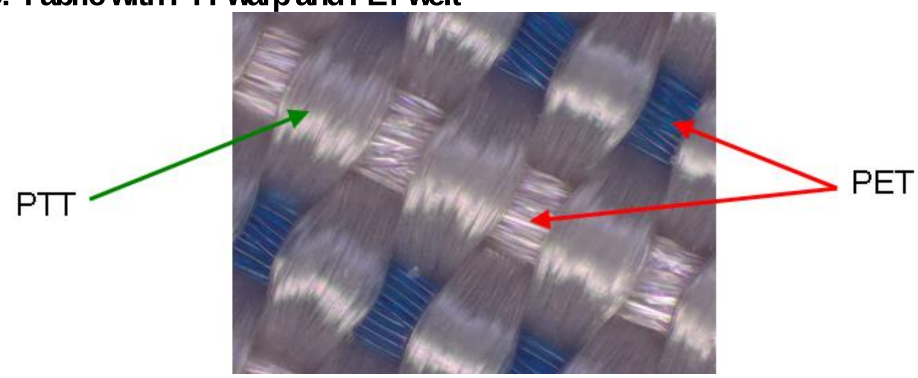
	Analytical Method	Comments
1	Visual/Microscopic Inspection	Compare fiber appearance and cross-section with reference samples
2	Density Measurement	Density for PTT is 1.33g/mL
3	DSC Thermal Measurement	Tg and Tm can be used to identify PTT
4	Mechanical Property Analysis	Observe modulus, stress-strain curve shape, elasticity, and tenacity at break
5	Intrinsic Viscosity	IV for a PTT fiber is usually higher than that for PET fiber, e.g., PTT fiber IV is >0.85 dL/g.
6	FTIR	Compare with the unique IR fingerprint of PTT
7	Chemical Analysis	Identify presence of dipropylene glycol (DPG) via GC method. PET will contain diethylene glycol (DEG).
8	ICP Elemental Analysis	PET may contain Sb as a catalyst residue. The catalyst residue is different for PTT.
9	Chemical Glycolysis and GC	Identify whether or not 1,3-propanediol is present by GC.

		Quantification is possible.
10	^{1}H-NMR	Compare with the chemical shifts for PTT and quantify amount.
11	^{13}C-NMR	Compare with the chemical shifts for PTT and quantify amount.

4.2 Examples of Fabric Analysis and Characterization: PTT in a Binary Fabric Sample with PET

Pictured in Figure 29 is a woven fabric material containing a PTT warp and two PET fiber types, one cationic dyed (blue) and another disperse dyed (silver). The following sub-sections give examples of the analytical techniques that could be employed for qualitative and quantitative assessment of the sample.

Figure 9: Fabric with PTT warp and PET weft

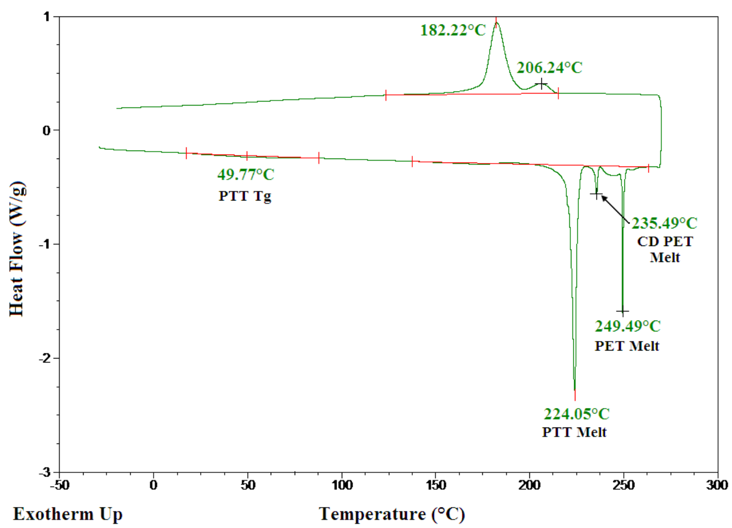


4.2.1 DSC

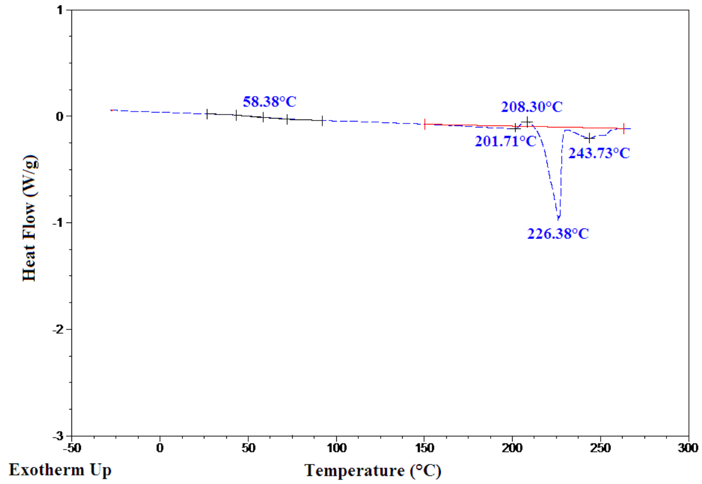
A heat-cool-reheat cycle will be used so that the initial response of the materials can be observed in addition to the behavior of the materials after their heat history has been erased by melting. Figures 30(a) and 30(b) show the DSC traces that were obtained for this fabric.

Figure 10: (a) First heat and (b) second heat DSC traces for fabric with PTT and PET

(a)



(b)



It can immediately be determined from Figure 30(a) that this sample contains more than one component by the simple observation that there are two well-defined endotherms as well as two distinct recrystallization exotherms. There is another small well-defined endotherm in between the first two corresponding to another component, possibly a PET copolyester. These features give a strong indication that this sample is not a unary system.

Further analysis of the DSC trace will allow for identification of these components. The first melt endotherm at 224°C, though a few degrees lower, is near the melting point for PTT seen above in Figure 21. Additionally, further evidence is provided by the recrystallization exotherm at 182°C, which is where the exotherm was seen for PTT in Figure 21. These two features together indicate that PTT is likely present in this sample.

In the second heat, a distinct glass transition (T_g) is present, centered around 58.38°C. This T_g is characteristic of PTT, although at a slightly higher temperature compared to neat PTT. This may be the result of a small degree of transesterification between PTT and PET, which could have occurred while the sample was held at 270°C during DSC analysis. Once this heat history was erased by the first heating cycle and the polymer was allowed to recrystallize, the T_g was present in the second heat. T_g can be a very useful feature in identifying PTT since it is quite distinct from that of other materials, most notably PET.

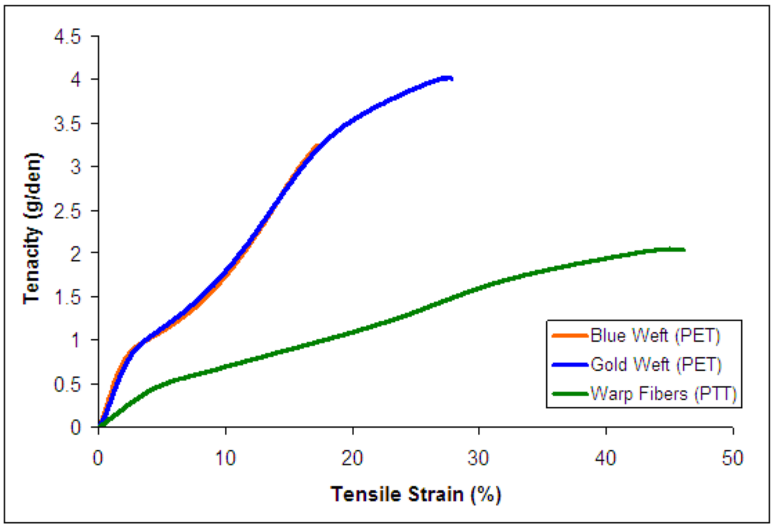
The presence of the melting endotherm at 249°C and the recrystallization exotherm at 206°C give strong evidence that a second component is present in this sample. As listed in Table 2 above, the melting temperature of neat PET is 260°C. Since fiber melting point is dependent upon the degree of crystallinity, which is a function of draw ratio when the fiber is spun, and it is likely that the fiber is not necessarily made from neat PET polymer, it is possible for PET to have a slightly lower melting temperature than 260°C. It is very common in the PET industry to use comonomers such as isophthalic acid in the PET manufacturing process, which will lower the melting point of PET fibers.

The DSC results for this sample provide evidence that this sample likely contains both PTT and PET. As with any analytical method, however, additional techniques are needed to verify these findings. Nonetheless, DSC is an effective tool for narrowing the possibilities and selecting candidates for the materials that are in this sample. To further verify these results and to better identify the components of the system, the individual fibers that make up the fabric can be mechanically separated and analyzed with DSC as well.

4.2.2 Mechanical Property Measurement

As previously shown, fibers made from different materials exhibit different mechanical properties. Therefore, testing the mechanical properties of the fibers in a binary system can be an effective analytical method for identifying the fiber. Figure 31 shows the stress-strain plots for the warp and weft fibers of the fabric sample presently under investigation.

Figure 11: Stress-strain plots for warp and weft fibers in binary PET/PTT sample



The warp and weft fibers from the binary sample under investigation here are definitively made of two different materials. These fibers can readily be identified by comparison with the stress-strain curves of known fibers, shown in Figure 28 above. Both the blue and the gold weft fibers match the curve typical for PET. Similarly, the stress-strain curve for the warp fibers matches the distinct curve of PTT. By simply stretching fibers made of PTT and PET, these materials may be differentiated even identified.

4.2.3 FTIR

Because FTIR gives a unique fingerprint for different materials, it is helpful not only for identification of a pure material, but also for distinction between two different materials. By identifying the unique regions of the spectra for these materials, an effective method for their differentiation can be made. For the identification of a binary sample, such as that under investigation here, the fabric can first be dissected, separating the warp and weft fibers from one another. Analysis of these components will result in the spectra shown in Figure 32 and Figures 33(a) and 33(b).

Figure 12: FTIR spectra for PTT (red) and PET (black)

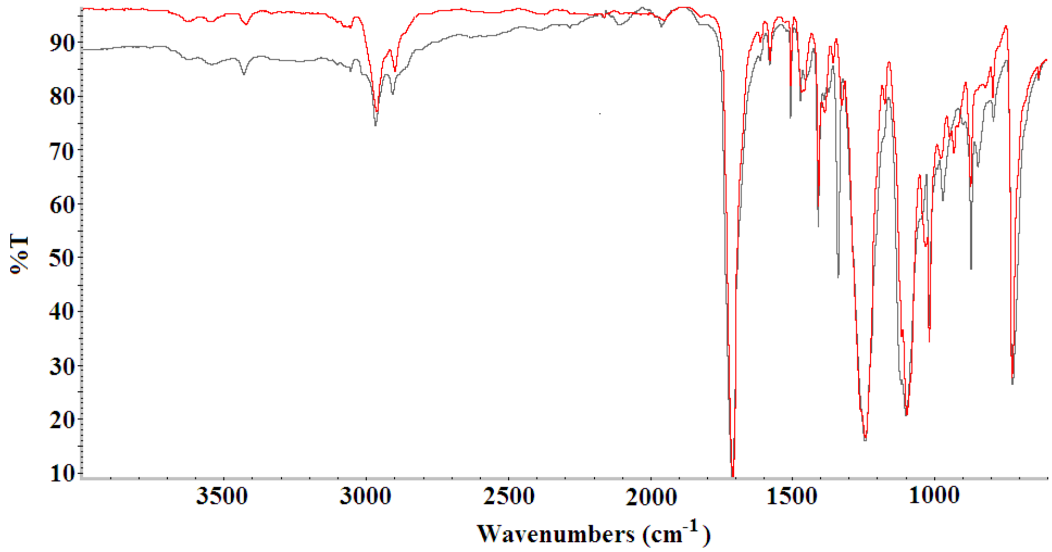
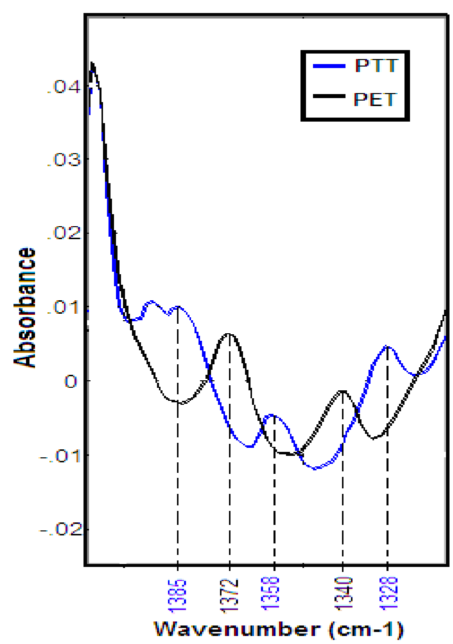
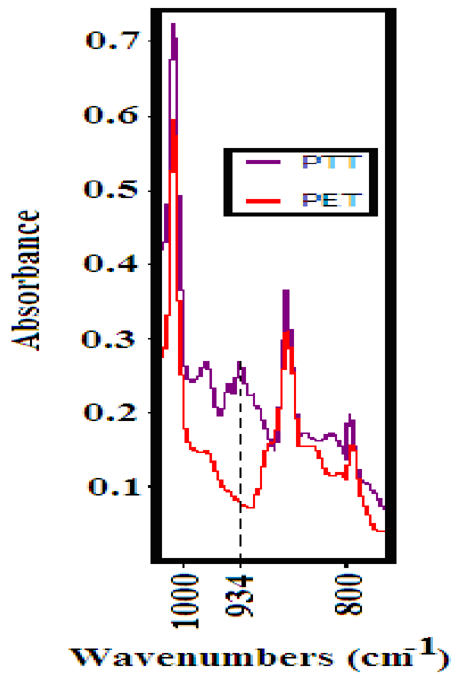


Figure 13: FTIR spectra for PTT and PET from (a) 1300 cm-1 to 1400 cm-1 and (b) 800 cm-1 to 1000 cm-1

(a)



(b)



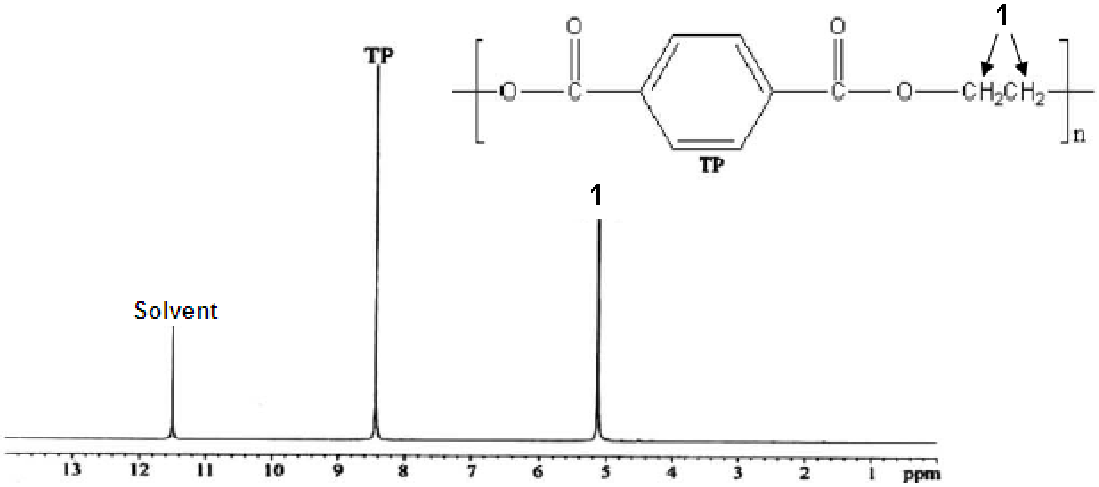
While in general the PTT and PET spectra appear to be quite similar, there are several key regions by which the two materials can readily be distinguished from one another. There are three peaks in the 1300 cm^{-1} to 1400 cm^{-1} region (Figure 33 (a)) that are characteristic of PTT and two that are characteristic of PET. The peaks corresponding to PTT occur at 1328 cm^{-1}, 1358 cm^{-1}, and 1385 cm^{-1}.[3] The peaks indicative of PET are at 1340 cm^{-1} and 1372 cm^{-1}.[4]

Another important region that may be used to identify PTT and distinguish it from PET falls between 800 cm^{-1} and 1000 cm^{-1} (Figure 33(b)). In this region, a distinct peak is present for PTT at 934 cm^{-1}.[3] This peak can be used as another important means of differentiating PTT from PET, which is devoid of peaks in this area.

4.2.4 NMR

^{1}H-NMR and ^{13}C-NMR can be very effective tools in identifying textile components, especially if they are macroscopically inseparable. If a deuterated solvent that will dissolve all components of a binary system is available, a spectrum of the resulting solution will allow for determination of what components are present. Figure 34 shows the spectrum for pure PET. Comparison of the resulting spectrum taken for the binary PTT/PET sample with the spectra for PTT (Figure 24) and pure PET (Figure 34) will allow for verification that these, in fact, are the two materials present.

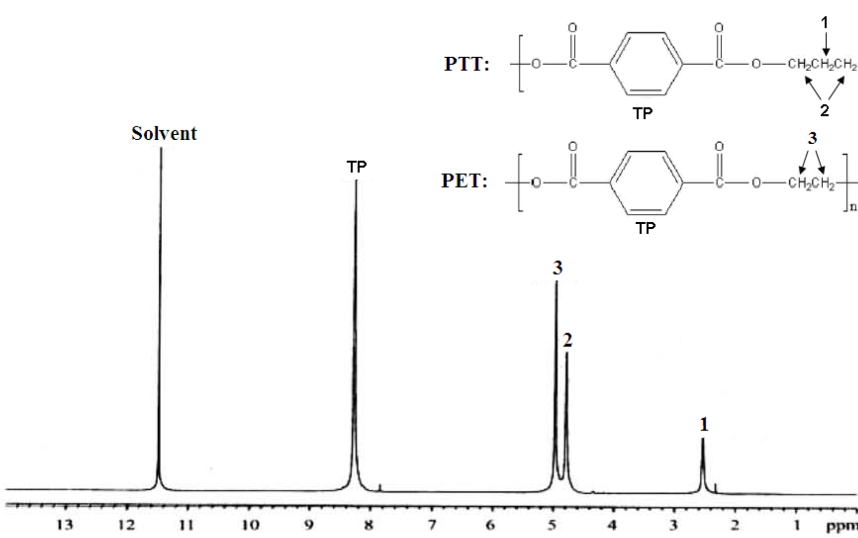
Figure 14: ^{1}H-NMR spectrum for PET[1]



As with PTT above, deuterated trifluoroacetic acid was used as the solvent to take the NMR spectrum of PET. The spectrum for PET shows the chemical shifts of the protons to be 8.44 ppm for those on benzene and 5.12 ppm for the ethylene protons.[1] Comparison of the pure PTT and pure PET spectra shows that, in addition to the small shift difference for the benzene protons and those α to the carbonyl groups, PTT can also be definitively differentiated from PET by the presence of the peak at 2.80 ppm corresponding to the protons β to the carbonyl. The presence of the well separated, easily distinguishable peak at 2.80 ppm in PTT is an important tool that may be used to differentiate these polymers.

A number of binary fabric samples were analyzed by dissolving all components of the fabric sample in deuterated solvent without separation to show the usefulness of ^{1}H-NMR for qualitative analysis. Figure 35 shows the NMR spectrum for a PTT and PET binary system. Since the relevant peaks for PTT and PET do not overlap, it is possible to quantify the relative amounts of these two materials via peak integration to determine the relative area under the curves.

Figure 15: Example ^{1}H-NMR spectrum for a binary sample containing PTT and PET[1]



The fabrics tested with this method and their results are shown in Table 12. The claimed sample composition and the measured sample composition are shown for comparison.

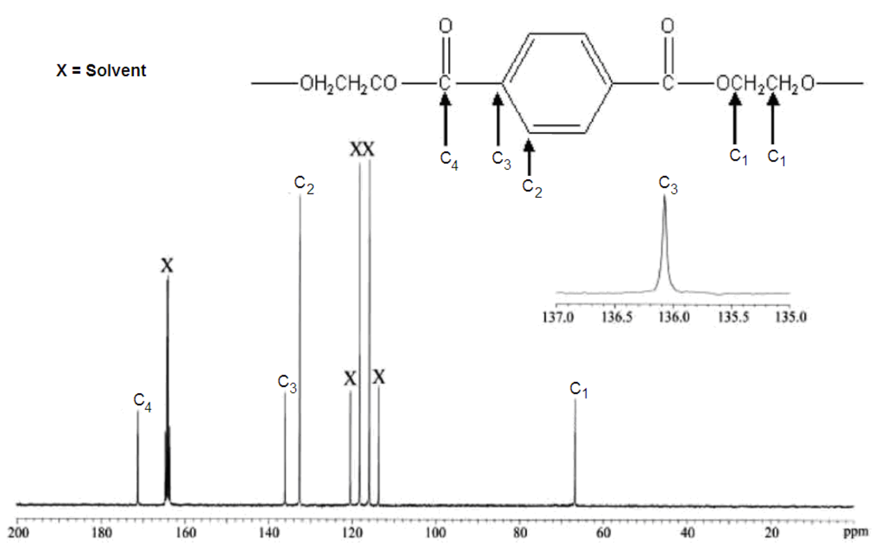
Table 4: ^{1}H-NMR quantification of binary fabric samples containing PTT and another fiber material

Fabric #	Sample Label	Claimed			NMR Quantification		
		PTT (%)	PET (%)	Other (%)	PTT (%)	PET (%)	Other (%)
1	25% PTT staple, 75% PET staple	25	75	0	24.6	75.4	0.0
2	96% PTT filament, 4% spandex	96	0	4	94.4	0.0	5.6
3	94% PTT filament, 6% viscose	94	0	6	93.6	0.0	6.4
4	46% PTT filament, 54% nylon	46	0	54	43.1	0.0	56.9
5	27% PTT, 73% cotton	27	0	73	28.8	0.0	71.2
6	100% PTT filament	100	0	0	100.0	0.0	0.0

It is evident from the various examples in Table 12 that PTT can be easily quantified in systems containing PET and other materials as well. All of these samples were experimentally determined to have essentially the same compositions as the label claimed. This experiment shows that ^{1}H-NMR analysis is incredibly effective method for the quantification and identification of PTT in binary fabrics.

For additional verification of the identities of the components in this binary sample, ^{13}C-NMR can be performed as well. Again, deuterated trifluoroacetic acid can be used as the NMR solvent. Figure 36 shows the carbon-13 spectra for PET. The shift assignments for PET are 66.70 ppm for the ethylene glycol carbons, 132.59 ppm for the aromatic alkyl carbons of the benzene ring, 136.08 ppm for the aromatic quaternary carbon atoms of the benzene ring, and 171.34 ppm for the carbonyl carbon atoms.[1] As with the proton NMR, the carbon NMR spectra for these two polymers can be explicitly differentiated by the peak that corresponds to the additional proton present in the glycol unit of PTT. For the carbon spectra, this peak occurs at 29.70 ppm in PTT. Since the spectrum for PET is clear in this region, it is easy to differentiate these two materials by this method as well.

Figure 16: ^{13}C NMR for PET[1]



4.2.5 Intrinsic Viscosity (IV)

IV is a relatively simple test method that can provide helpful data in the identification of unknown polymeric or fiber materials. If the sample can be separated into its individual warp and weft components, this method may be utilized. The method for IV analysis is briefly outlined above in Section 4.1.4, along with Table 9 which contains IV data for PTT and PET. For the binary PTT/PET sample in question here, the individual fibers may readily be separated from one another, and therefore may be analyzed by IV. Since PTT and PET have very distinct IV values, this method is helpful in differentiating between these materials. It would be anticipated that analysis of the fibers from the binary sample would yield IVs that correspond to pellet IVs of 1.02 dL/g and 0.64 dL/g or similar, verifying the presence of PTT and PET in the system. For reference, IVs for fibers of PTT and PET are generally lower than IVs of the pellet forms of these polymers.

4.2.6 Density

Similar to IV analysis, density measurement is another relatively simple, yet effective, analytical method that can be helpful in the identification of textile materials when used in conjunction with other techniques. The method by which this test can be performed is outlined above in section 4.1.5. Table 10 above shows the densities of a variety of common textile polymeric materials. Again, this method requires that the individual components be separable from one another. Upon analysis, it is anticipated that the measured densities would be approximately 1.33 g/mL and around 1.38-1.41 g/mL for the sample used in this example, corresponding to PTT and PET respectively.

4.2.7 Chemical Analysis

There are various chemical analysis methods that can be employed to test textile materials. Glycolysis of PET and PTT materials followed by gas chromatographic analysis will allow for identification and quantification of diethylene glycol (DEG) and dipropylene glycol (DPG), respectively.

Additionally, inductively coupled plasma metal analysis can be used to differentiate between PET and PTT. Since antimony, the catalyst typically used to make PET, remains as a residue in the finished product, it will appear in ICP analysis of this fiber.

4.2.8 Quantitative Analysis

It is required by the Commission that fibers for a given generic not only be assessed qualitatively but also quantitatively. For a pure sample, as examined above in Section 4.1, quantification is not difficult because the material is made of only one material. Binary, ternary, and higher order samples, on the other hand, require quantification of the relative amounts of the materials present.

Gravimetric measurement methods coupled with elemental analysis can also be used for the quantitative assessment of fibers and fabrics.

4.2.8.1 Mechanical Separation

In situations where the individual fibers of a textile can be mechanically separated, such as the fabric sample currently under investigation, it is relatively simple to determine the percent composition of the sample. A method by which this analysis can be performed is presented in AATCC Method 20A-2008, Section 10.[5] Prior to analysis, the samples must be prepared using the procedure outlined in this method. Once the sample is ready for analysis, it is mechanically separated into its individual components which are then massed. Using Equation 3, the relative percent composition of each fiber can be determined.

Equation 3: Percent composition of mechanically separated materials

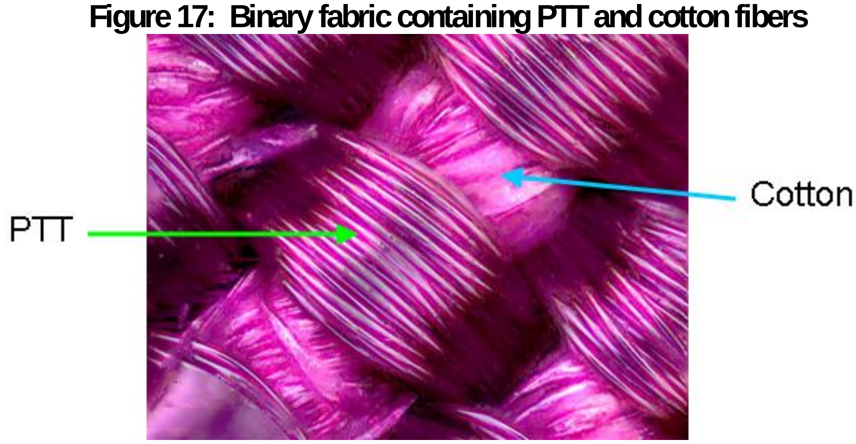
$$X_i = (W_i/E) * 100$$

Where X_i = percent content of fiber i
W_i = the oven-dry weight of fiber i, after separation
E = the weight of clean, oven-dry specimen taken for analysis

4.3 Example of Fabric Analysis and Identification: PTT with Cotton in a Binary Sample

As an example of identification of PTT in a sample containing a natural fiber, the fabric shown in Figure 37 will be considered here. This material is comprised of a PTT warp and cotton weft.

Figure 17: Binary fabric containing PTT and cotton fibers

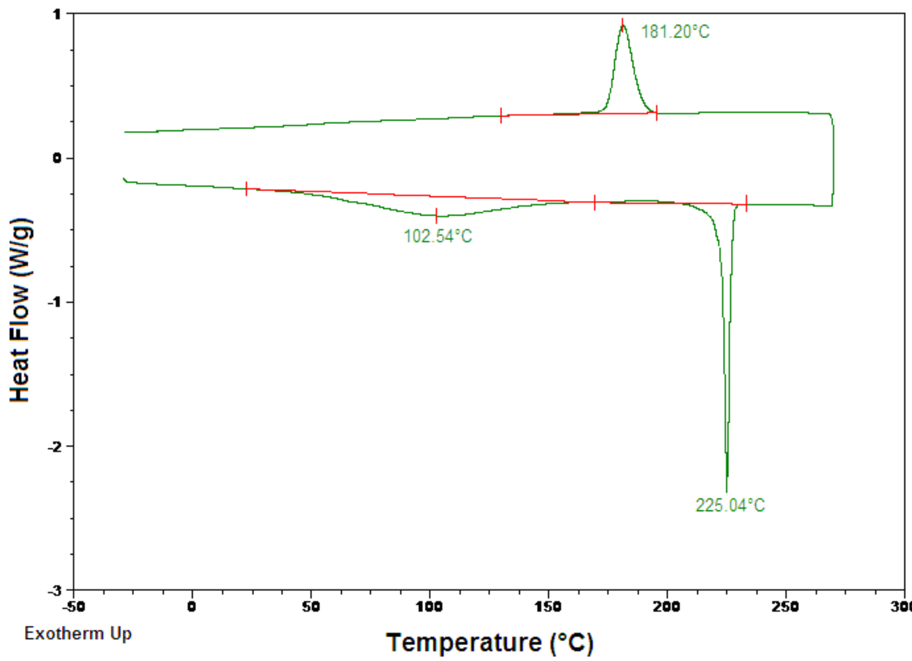


4.3.1 DSC

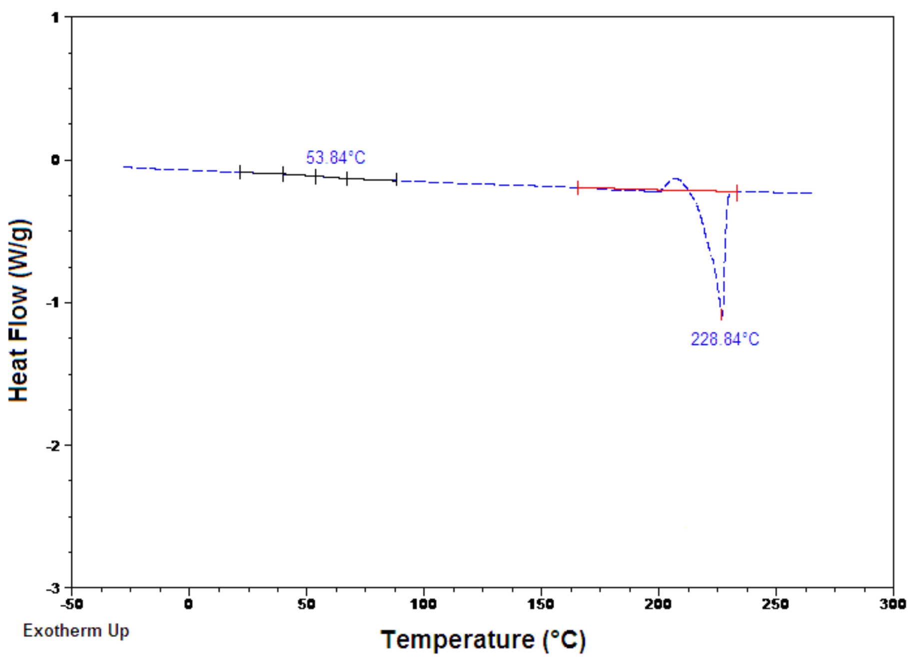
DSC has previously been shown to be an effective tool for fabric analysis. This holds true for the analysis of fabrics containing both synthetic and natural fibers as well. Figure 38 shows the first and second heat DSC trace for the binary fabric studied here.

Figure 18: DSC (a) first heat and (b) second heat for binary sample containing PTT and cotton

(a)



(b)



The sharp endotherm shows the likely presence of PTT in this binary fabric. The melt endotherm located at 225°C is right in the melting range for PTT fiber. On the second melt, after the heat history was erased by the first heat, the melt endotherm is observed at 229°C, which is very close to the expected melting point for pure PTT. Similarly, the single recrystallization peak present at

181°C is also characteristic of PTT. Additionally, the glass transition seen in the reheat cycle at 54°C corresponds to that expected for PTT. These observations can be verified by comparison with the DSC trace for PTT given in Figure 21 above.

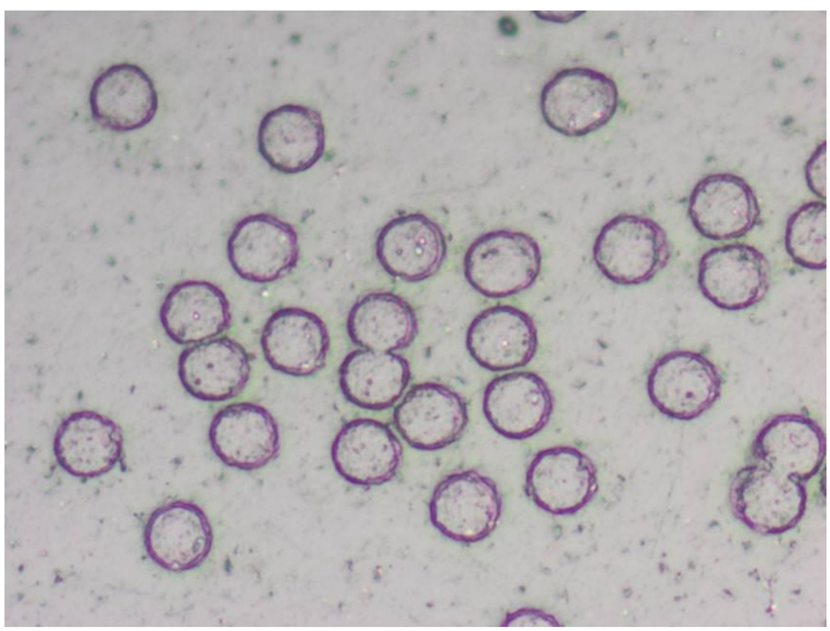
While no melt endotherms or recrystallization exotherms are present for cotton in the DSC trace shown in Figure 38, there is indication that a natural fiber is present. PTT is known to retain little water in comparison to that which is held by some natural fibers, including cotton. When a material containing free water is analyzed by DSC, a broad endotherm, such as that centered around 102°C in the trace above, is present. This broad endotherm corresponds to the evaporation of water from that sample. Therefore, while it cannot be concluded from this test alone that the second component is cotton, it can be determined that there is a second component in this system other than PTT and that it is likely that this other component is a natural fiber. In order to further identify this component, as well as verify the presence of PTT, additional analyses are necessary.

4.3.2 Cross-Section Analysis

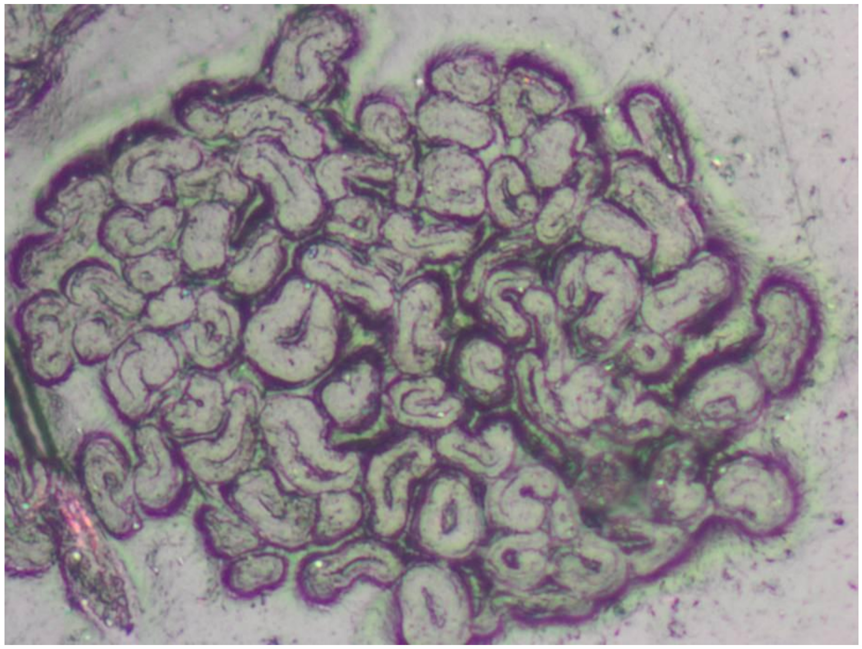
Since cross-sections of synthetic fibers tend to be unique in their appearance, microscopic cross-section analysis may be a helpful tool in identifying the second component of the sample in question. Figures 39(a) and 39(b) show the cross-sections of the warp and weft fibers for this sample.

Figure 19: Cross-section photomicrographs of the (a) warp and (b) weft fibers of the PTT and cotton binary sample

(a)



(b)



Based on the round shape of the cross-section for the warp fibers and the fact that DSC analysis indicated that PTT is present in the sample, it can be concluded that the warp component of this fabric is PTT.

The cross-section of the weft fibers from this sample shows the kidney shape that is characteristic of cotton, as depicted in Appendix I to AATCC TM 20-2007.[5] While simple visual observation is not enough to definitively say the weft material is cotton, the characteristic cross-section of cotton does provide strong evidence to this point.

4.3.3 Further Analysis

For further verification of the identity of the materials present in this sample fabric, the analytical tests presented above in Table 11 would prove effective for analysis of PTT as well as quantification of these fabric components. Complete identification of the cotton component of this sample could be carried out using the methods prescribed by the AATCC in TM 20-2007.[5]

4.4 PTT in Ternary and Higher Order Systems

When textiles are made from continuous filaments, it is generally easy to mechanically separate the material back into its individual components. Therefore, regardless of the order of the system, be it ternary or higher, analysis simply requires the initial separation of the sample into its original components, which can then readily be analyzed and identified on their own. In situations where this is the case, analysis of ternary and higher order systems is performed in the same manner as outlined above for unary and binary systems. When samples cannot be mechanically separated into their individual components, there are still a number of analytical tests that may be performed to identify the fiber samples. DSC, NMR, and glycolysis are just a few examples of the tests that can be performed on inseparable samples.

Methods to Quantify Triexta*/PET Polyester Mixtures

**Triexta is the proposed name for fibers made from poly(trimethylene terephthalate) or PTT*

Quantification of triexta/PET polyester intimate blends requires different analytical techniques than blends of other fibers because they exhibit similar solubility properties. Nonetheless, there are a number of techniques by which these blends can be quantified.

Two such methods are described here in detail, including the calculations, accuracy, precision, robustness, and repeatability of these techniques.

Sample Preparation:
In most instances, qualitative analysis of fibers can be carried out without pretreatment.
If heavy surface finishes such as waxes or oils are present on the fiber and will inhibit accurate analysis of the sample, the pretreatment methods described in Sections 1.2-1.7 of Annex II to Directive 96/73/EC can be performed.

1. Quantification by Differential Scanning Calorimetry

Principle:
Conventional differential scanning calorimeters (DSC) measure the relative heat flow of a sample as it is heated over a temperature range versus a standard material. Various thermal features of polymeric materials can be observed and measured by this method, including the heat of fusion. For a given material, the total heat of fusion per gram of sample present is consistent when all samples have similar degrees of crystallinity. Since this value is determined on a per gram basis, it is possible to generate a calibration curve for a material and back calculate from the total heat of fusion to determine the mass of the melting material. Triexta and PET polyester have distinct melt endotherms. Therefore, it is possible to use this method to quantify triexta/PET polyester mixtures.

Method:
Weigh a sample of fiber and place it in the vessel designed for the DSC. Typically this vessel is a small metal pan.
The conditions given in should be used for DSC analysis of triexta fibers and mixtures of triexta and PET polyester.

Table 5: DSC conditions for analysis of triexta fibers and blends thereof

Parameter	**Setting**
Temperature Range	20 °C – 280 °C
Heating Rate	10 °C/min
Purge Gas	Nitrogen
Heating Profile	Single heat ramp

A heat/quench/reheat heating profile can also be beneficial in analysis of triexta/PET polyester mixtures, as these materials exhibit different glass transition and cold crystallization features. The melt endotherms from the first heat cycle for triexta and PET polyester are typically used for quantitative measurements.

Analysis of Results:

Identify the various thermal features in the resulting DSC trace and the temperature at which they occur.
A DSC trace for amorphous triexta fiber is shown here in Figure 20. The ranges in which thermal features for triexta are typically observed are summarized in Table 6.

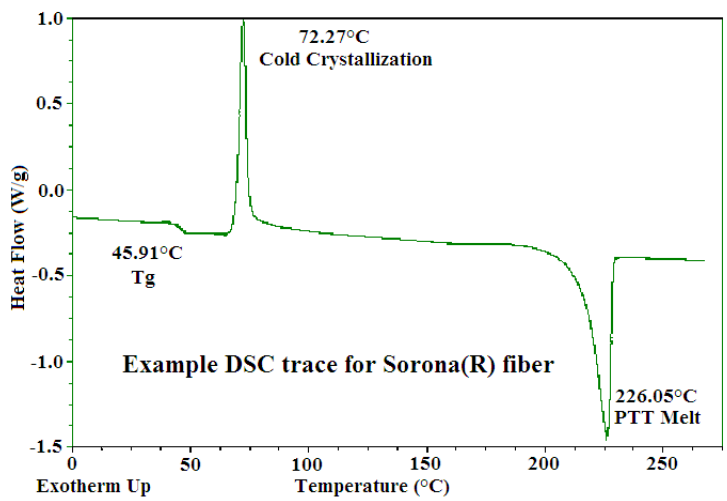


Figure 20: Typical DSC trace for amorphous PTT

Table 6: Typical thermal features of triexta

Feature	**Temperature**
Glass Transition Temperature	45 °C – 55 °C
Cold Crystallization	~70 °C
Melting Point	226 °C – 233 °C

In general, a sample containing triexta and PET polyester fibers will have a first heat DSC trace similar to that shown in Figure 21. The benefit of the first heat ramp is that there is good resolution between the melt endotherms for triexta and PET polyester. This allows for accurate quantification of the sample's composition.

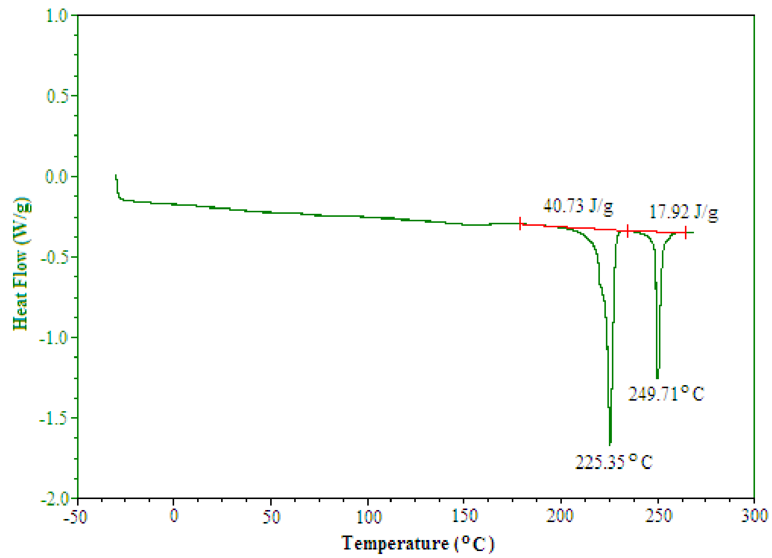


Figure 21: Example DSC trace for heat/cool cycle of triexta/PET polyester fabric.
[Triexta/PET % ratio is 66/34]

The DSC trace for the second heat of a sample, following quenching, is shown here in Figure 22, along with the first heat trace for reference. In the second heating cycle, after the sample's heat history has been erased, the distinct glass transition and cold crystallization peaks for triexta and PET polyester can be observed in the sample. These features are beneficial in verifying the presence of triexta and PET polyester materials in the sample.

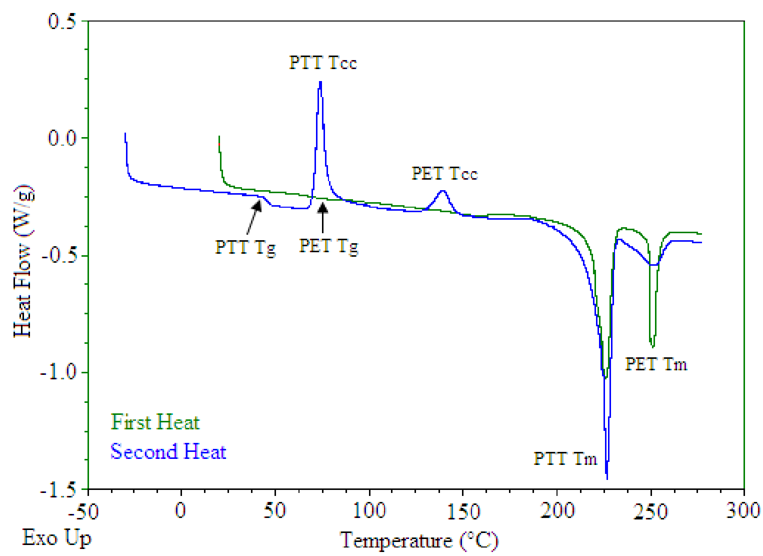


Figure 22: First and second heat DSC traces for an intimate blend of triexta and PET polyester fibers

Table 7: Important thermal features in the DSC trace for an intimate blend of triexta and PET polyester fibers

Feature	Heat Cycle	Temperature	Fiber Type
Glass Transition (T_g)	First	70.9°C	PET Polyester
	Second	46.3°C	Triexta
Cold Crystallization (T_{cc})	Second	73.9°C	Triexta
	Second	139.8°C	PET Polyester
Melt (T_m)	First / Second	226.7°C / 225.8°C	Triexta
	First / Second	250.9°C / 251.4°C	PET Polyester

Quantification:

The quantification method presented here is similar to the DSC quantification method presented in the JRC report "*Fibre Labeling Elastomultiester—DuPont*" for the quantification of elastomultiester in fabrics.[1] The "3GT" component that allowed for differentiation between elastomultiester and PET polyester is the same material as triexta. Therefore, the method can easily be modified to quantify mixtures of triexta and PET polyester.

As shown in the above example, triexta and PET polyester have distinct melt endotherms, which allows for determination of their individual total heats of fusion. These total heats of fusion can be used to calculate the quantity of triexta and PET polyester present in a given sample.

To quantify triexta and PET polyester in a blended fabric, a calibration curve must first be developed for the system. This curve can easily be established by creating mixtures of triexta and PET polyester fiber in varying ratios and performing DSC analysis on these mixtures. The resulting heat of fusion values from the first heating cycle for the triexta melt endotherms can be plotted as a function of the percent present in the sample. The result of this analysis is a linear regression showing an increase in the heat of fusion for the triexta melt peak as the percentage of triexta present in the sample increases from 0% to 100%. This relationship is shown below as Equation 1.

$$y = mx + b \qquad \textit{Eq. 1}$$

where, y = heat of fusion for triexta melt endotherm
x = percent triexta in sample, m = angular coefficient, b = y-intercept

The angular coefficient of the linear regression can then be used to determine the composition of unknown samples containing triexta using the following approach. Since a sample with 0% triexta will have a melt endotherm heat of fusion of 0 J/g, *b*

[1] *Picciinini, P., Senaldi, C. "Fibre Labeling Elastomultiester—DuPont, Final Report." Administrative Arrangement N. 2003—21200, European Commission, Joint Research Centre, Institute for Health and Consumer Protection, p. 30.*

$= 0$. The heat of fusion, y, for the unknown sample is determined experimentally. Then, rearranging Equation 1 allows for determination of the percentage of triexta in an unknown sample with Equation 2,

$$x = y/m \qquad \textit{Eq. 2}$$

Accuracy and Precision:

In order for any quantification method to be valid, it is necessary to establish an understanding of the methods accuracy and precision. The accuracy of the method can be established by analyzing samples of known composition with the new method and comparing the analytical results with the theoretical values. Precision can be determined by assessing the variability in multiple measurements of a given sample.

In order to evaluate the accuracy and precision of this method, six samples containing various weight ratios of triexta and PET polyester were quantified with it. The results of this analysis are shown below in Figure 23. This figure was created by plotting the measured heat of fusion for the melt endotherm at 226°C for each sample versus the known triexta composition of the fabric sample being evaluated. Based on this analysis, it is easily observed that there is a strong linear relationship between the heat of fusion of triexta and the weight percent of this material in the fabric. With the calibration curve established, it is then possible to use the equation of the linear regression for the control samples in order to quantify unknown samples. The equation of the line established in this experiment has an R^2 value of 0.9935, showing a strong linear relationship for the data. It can therefore be concluded that the DSC method is very effective for the quantification of triexta in fabrics that contain intimate blends of triexta and PET polyester.

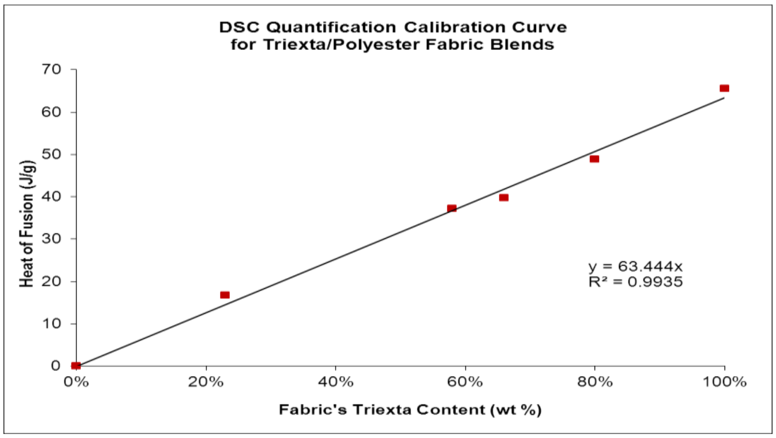


Figure 23: Calibration curve for quantification of triexta/PET polyester fabric blends by DSC

With the accuracy of this method verified, the precision of the method was determined by analyzing a sample containing 80% triexta and 20% PET polyester. Three specimens from the sample were analyzed and quantified in this experiment.

The data for these repeat trials are shown in Table 8. Additional statistical information for these data is shown below in Figure 24.

Table 8: Data for replicate DSC measurements on control sample

Description	First Melt Heat
80/20 PTT/PET	48.94
80/20 PTT/PET	48.43
80/20 PTT/PET	49.26
Average	**48.87666667**
95% Con.	**1.04**

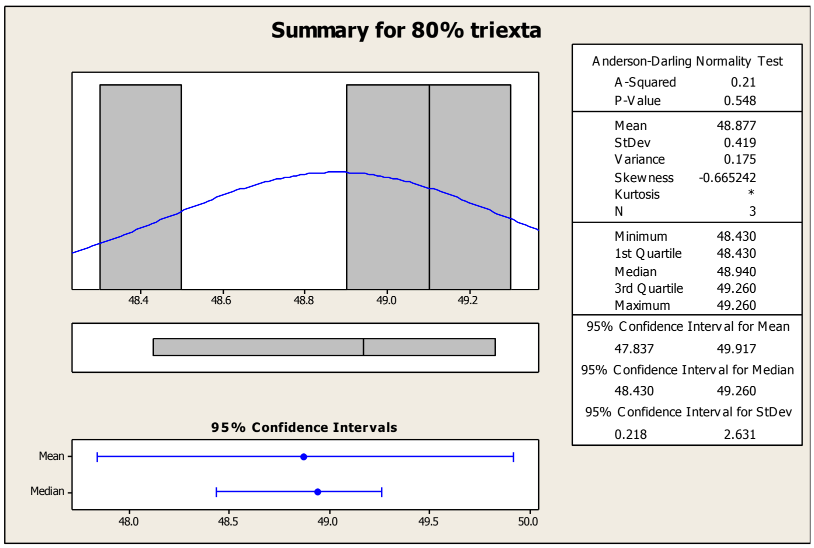


Figure 24: Precision of DSC quantification for 80/20 triexta/PET polyester fabric

The 95% confidence interval for the mean is an important value, often used to convey the precision of an experiment. In this case, the 95% confidence interval was 1.04%. This result is consistent with what has been observed for other samples examined by this same method.

With the high degree of accuracy and precision demonstrated for the DSC quantification method, it can be concluded that this is an effective approach for the quantification of fabrics with unknown compositions of triexta and PET polyester.

2. <u>Quantification by Nuclear Magnetic Resonance</u>

Principle:

Nuclear magnetic resonance (NMR) spectroscopy yields a detailed spectrum showing the relative chemical peak shifts for the nuclei being analyzed. In this method, the sample is exposed to a strong magnetic field, which then induces the emission of electromagnetic energy by the atoms being analyzed. These atoms are commonly protons. The chemical shifts are different for each molecule, depending on the surrounding electronic environment, allowing for identification of different materials. Since each peak in the proton NMR (H-NMR) spectrum corresponds to a unique type of proton, the relative amounts of different types of protons present in the sample can be quantified, allowing for quantification of the different materials present in the sample initially.

Method:
Dissolve the sample in an appropriate solvent system for the material present. Sample concentration should be 5-25 mg/mL. Triexta and PET polyester are both soluble in a 5:1 mixture of *d*-chloroform and *d*-trifluoroacetic acid, allowing them to be analyzed, identified, and quantified, even in inseparable blends. Other solvent systems can be used as well.
Using the standard procedure for the particular NMR instrument being used, introduce the sample tube into the magnetic field. Following the necessary shimming and alignment of the magnetic field, then perform a minimum of 16 scans of the sample.

Analysis:
With the appropriate computer interface, assign the solvent resonance the appropriate chemical shift to calibrate the spectrum. For the *d*-chloroform and *d*-trifluoroacetic acid mixture recommended for triexta and PET polyester, the peak associated with *d*-chloroform should be assigned to 7.62 ppm in the proton NMR. Identify and integrate the various peaks present in the spectrum for the sample.
The proton NMR spectra are shown here in Figure 25 with annotations indicating the peaks that correspond to triexta fiber and which proton type gives rise to the peak.

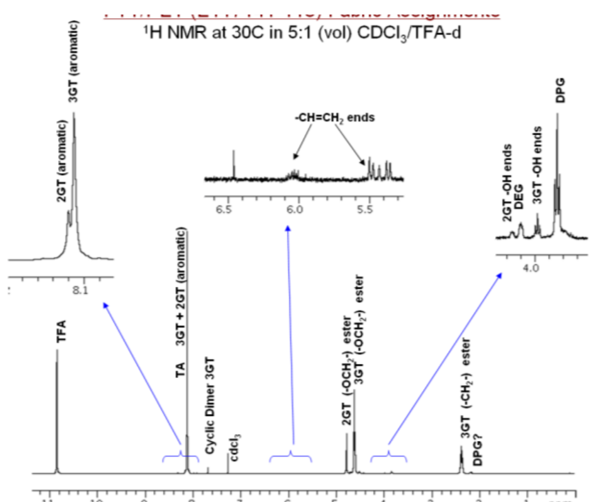


Figure 25: Proton NMR spectrum for triexta/PET polyester blended fabric with peak assignments

An example proton spectrum is shown below in Figure 26 as well with the area values for the various peaks. Note that the area under peak values obtained for each peak have arbitrary units because they are reported with values relative to the total peak area for the spectrum. In this case, the area under the peaks corresponding to the four protons on the benzene ring for both triexta and PET polyester were assigned the value of 400 units, 100 units for each proton.

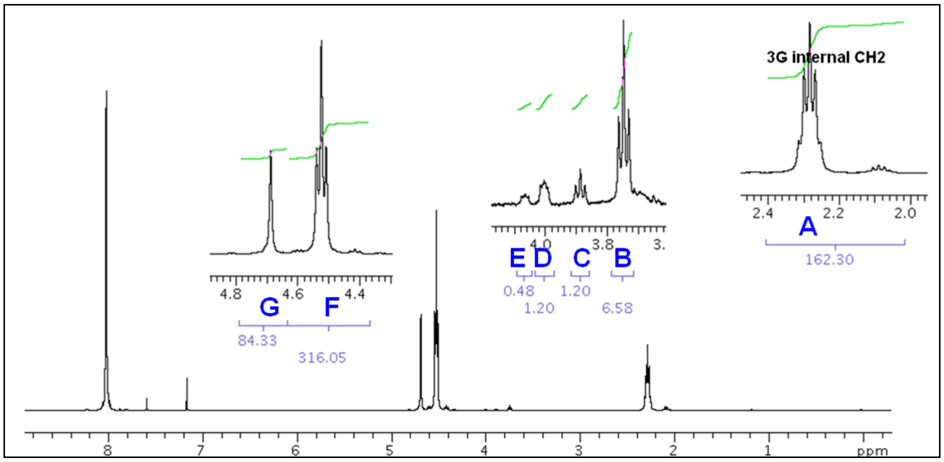


Figure 26: Integrated proton NMR spectrum for triexta/PET polyester fabric blend

Peaks G and F correspond to the four protons in the alpha position relative to the ester linkages in PET polyester and triexta, respectively. Peak A is due to the two protons on the central methyl carbon of the glycol portion of triexta and the central carbon in dipropylene glycol (DPG) and the chain end groups. Peaks E and D correspond to the protons alpha to the hydroxyl end-groups and to the ether link in diethylene glycol (DEG). Peaks C and B correspond to the protons alpha to the hydroxyl end groups and to the ether link in DPG.

Quantification of the components in the sample is carried out by the following method:

1. Divide the area of peak A by 2, since two protons contribute to each of the peaks in this region. The resulting value is equivalent to the number of mol equivalents of triexta present in the sample.

$$\text{mols of triexta} = (162.30 \text{ units})/(2 \text{ units/mol}) = 81.15 \text{ mol}$$

2. Sum peaks B, C, D, E, F, and G and divide by 4 since 4 protons contribute to each of these peaks. The result of this step is the total relative number of mol equivalents of triexta and PET polyester present in the sample.

$$\text{total mols in sample} = (6.58 \text{ units} + 1.20 \text{ units} + 1.20 \text{ units} + 0.48 \text{ units} + 316.05 \text{ units} + 84.33 \text{ units})/(4 \text{ units/mol}) = 102.46 \text{ mol}$$

3. Since the sample is composed of only triexta and PET polyester, subtract the number of mol equivalents of triexta (step 1) from the total relative number of mols (step 2).

$$\text{mols of PET polyester} = 102.46 \text{ mol} - 81.15 \text{ mol} = 21.31 \text{ mol}$$

4. With the number of mol equivalents of each material present, use the molecular weight of each material to calculate the mass of each in the sample.

mass of triexta = (81.15 mol)*(206.17 g/mol) = 16730.70 g triexta

mass of PET polyester = (21.31 mol)*(192.14 g/mol) = 4094.50 g PET polyester

5. Determine the weight percent of each material in the sample.

% wt triexta = ((16730.70 g)/(16730.70 g + 4094.50 g))*100 = 80.34%

% wt PET polyester = ((4094.50 g)/(16730.70 g + 4094.50 g))*100 = 19.66%

The manufacturer's claimed composition for the sample quantified in the example above is 80% triexta, 20% PET polyester. Comparing the experimental results with the theoretical values, it is observed that quantification by NMR is very accurate.

Below in Table 4 the resulting data is presented for the quantification of a variety of mixtures of triexta with other types of fiber, including PET polyester, using proton NMR. In this analysis, the sample composition as evaluated by proton NMR was compared with the manufacturer's claimed sample composition. Again, it is observed that NMR is a very accurate method for quantification of intimate fiber blends. The error for the experiments in Table 4 is shown in Table 5.

Table 9: Proton NMR quantification results for various fabrics containing triexta

#	Sample Label	Claimed			NMR Quantification		
		Triexta (%)	**PET Polyester (%)**	**Other (%)**	**Triexta (%)**	**PET Polyester (%)**	**Other (%)**
1	25% triexta staple, 75% PET polyester staple	25	75	0	24.6	75.4	0.0
2	96% triexta filament, 4% spandex	96	0	4	94.4	0.0	5.6
3	94% triexta filament, 6% viscose	94	0	6	93.6	0.0	6.4
4	46% triexta filament, 54% nylon	46	0	54	43.1	0.0	56.9
5	27% triexta, 73% cotton	27	0	73	28.8	0.0	71.2
6	100% triexta filament	100	0	0	100.0	0.0	0.0

Table 10: Error in Proton NMR Quantification Experiments

#	Sample Description	Error (%)		
		triexta	PET polyester	other
1	25% triexta staple, 75% PET polyester staple	-0.4	0.4	-------------
2	96% triexta filament, 4% spandex	-1.6	-------------	1.6
3	94% triexta filament, 6% viscose	-0.4	-------------	0.4
4	46% triexta filament, 54% nylon	-2.9	-------------	2.9
5	27% triexta, 73% cotton	1.8	-------------	-1.8
6	100% triexta filament	0	-------------	-------------

To evaluate the precision of the method for quantification of triexta/PET polyester blends by proton NMR, three independent samples of the 80/20 triexta/PET polyester sample analyzed in the example above were quantified by this method. The samples for this study were taken from three different parts of the fabric and prepared separately. The instrument and analyst were the same for all three measurements. The method was also the same for all three samples, following the procedure outlined above. The results for this analysis are presented in Table 11. The statistical information for the calculated percent triexta is shown in Figure 27. Since the percentage results for PET polyester and triexta are dependent on one another, the precision is the same for each component. As seen in Figure 27, the 95% confidence interval for the percent composition measurements is 0.86%. Therefore, NMR is both an accurate and precise method for the quantification of triexta/PET polyester fabric mixtures.

Table 11: Precision of Proton NMR quantification method

Polymer Type	Molecular Weight (g/mol)	Trial 1 (wt%)	Trial 2 (wt%)	Trial 3 (wt%)
Triexta	206.17	80.6	80.3	80.6
PET Polyester	192.14	19.4	19.7	19.4

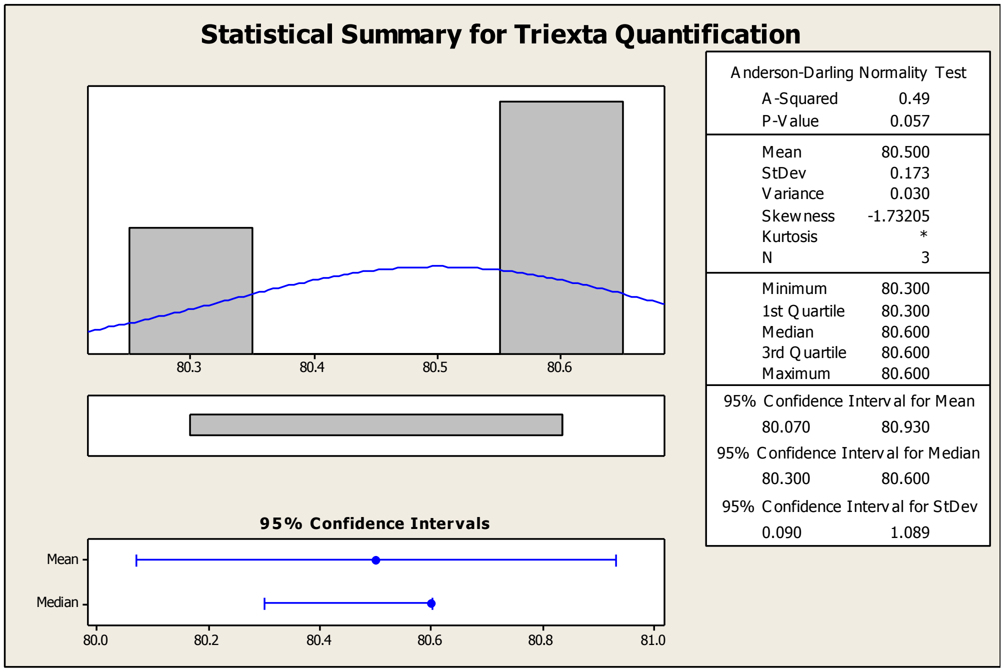


Figure 27: Statistical information for determination of precision of proton NMR method

Annex II

Microscopic analysis

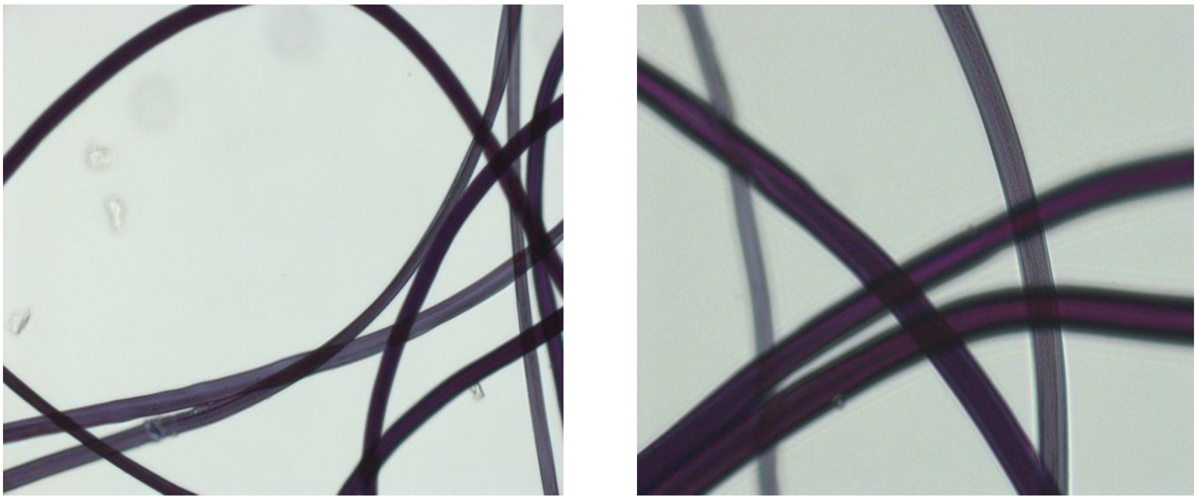


Figure 1: Microscope longitudinal analysis of sample **304** (28% PTT - 5% elastane - 68% modal) 200x (left) and 400x (right)

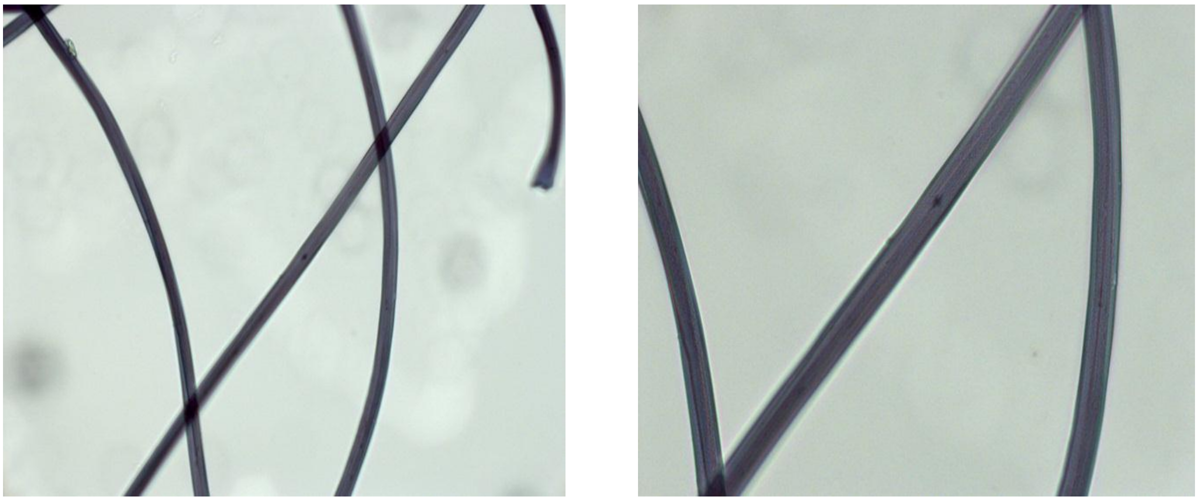


Figure 2: Microscope longitudinal analysis of sample's **304** remaining residue after applying the chemical dissolution methods 7 and 8 (28% PTT - 5% elastane - 68% modal) 200x (left) and 400x (right)

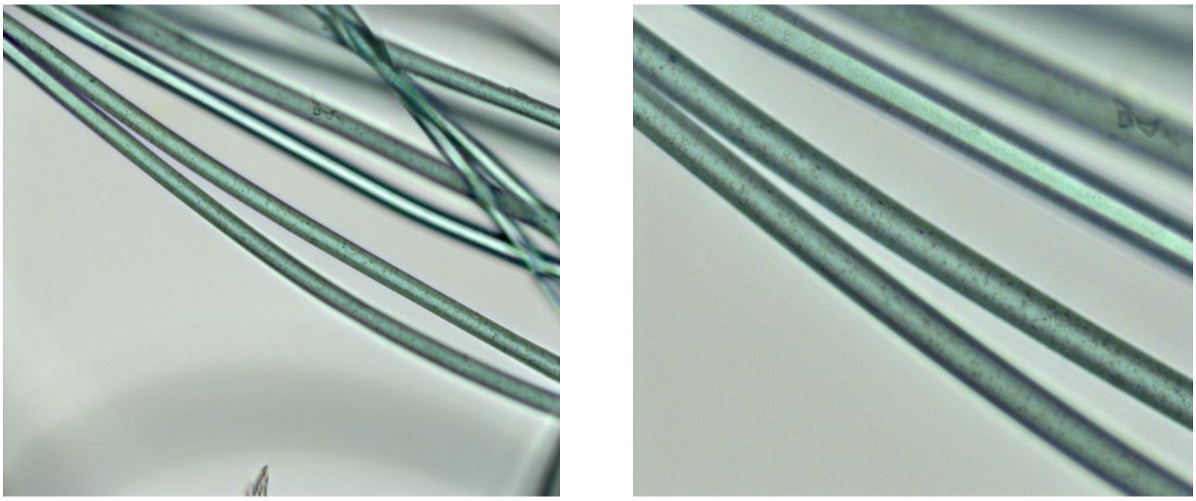


Figure 3: Microscope longitudinal analysis of sample **305** (80% PTT - 20% elastane) 200x (left) and 400x (right)

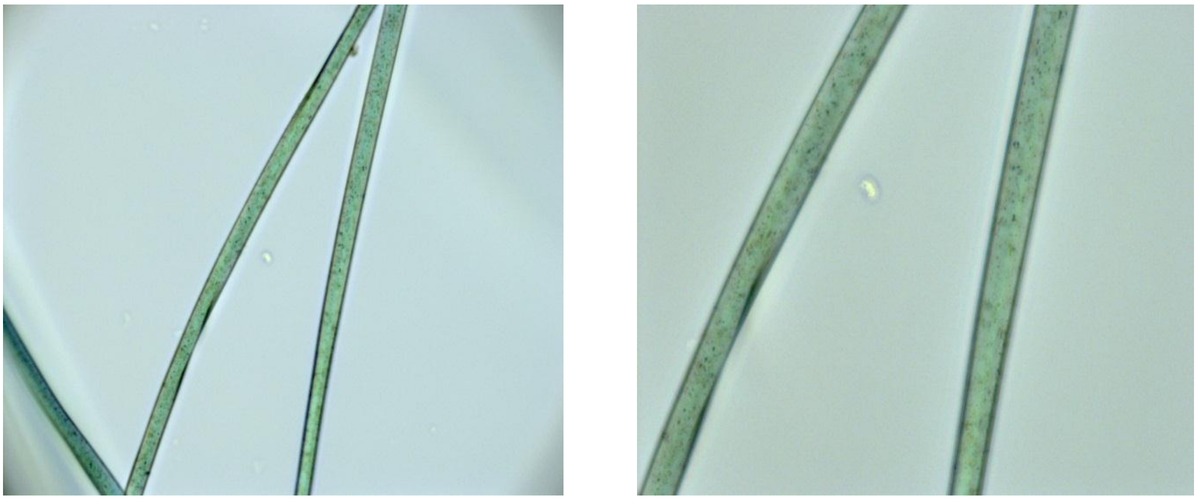


Figure 4: Microscope longitudinal analysis of sample's **305** remaining residue after applying the chemical dissolution method 7 (80% PTT - 20% elastane) 200x (left) and 400x (right)

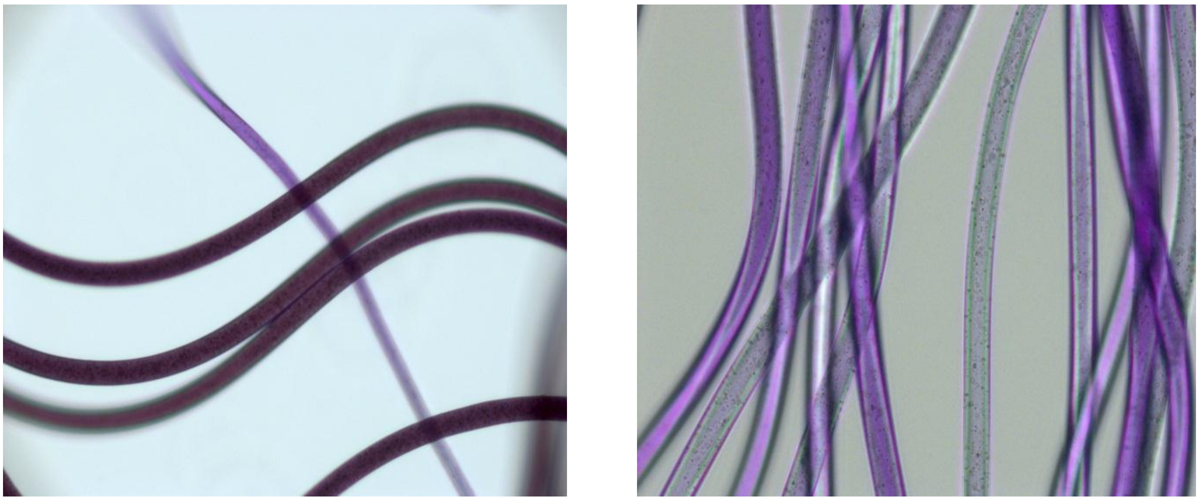


Figure 5: Microscope longitudinal analysis of sample **306** (41% PTT - 58% polyamide) 200x (left) and 400x (right)

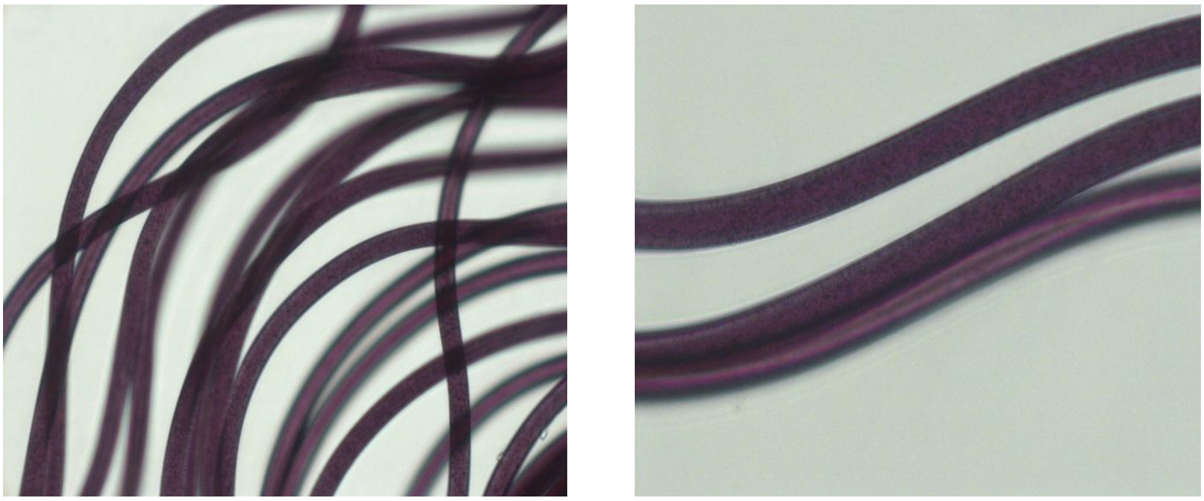


Figure 6: Microscope longitudinal analysis of sample's **306** remaining residue after applying the chemical dissolution method 4 (41% PTT - 58% polyamide) 200x (left) and 400x (right)

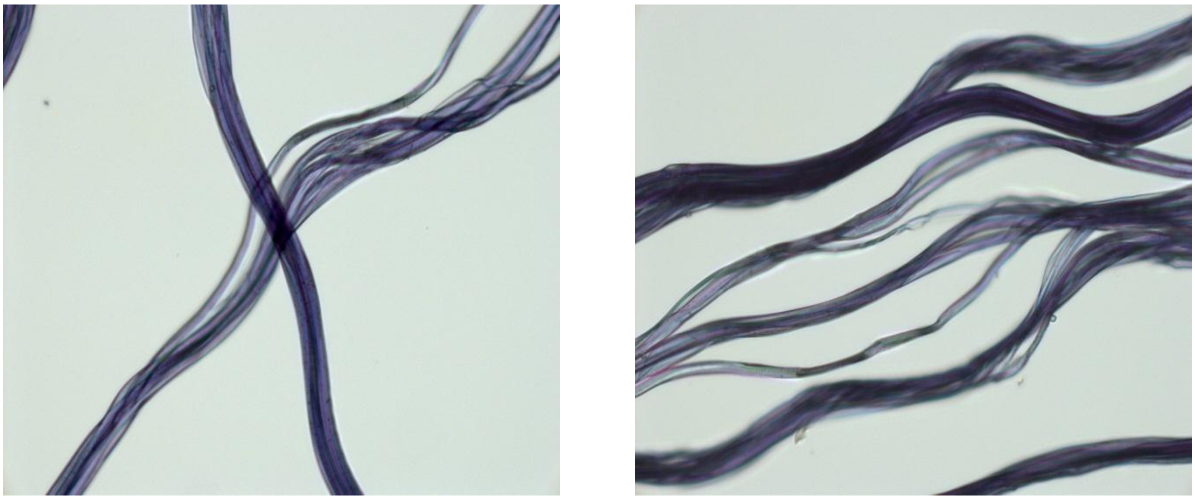


Figure 7: Microscope longitudinal analysis of sample **307** (76% PTT - 17% PET - 7% polyamide) 200x (left) and 400x (right)

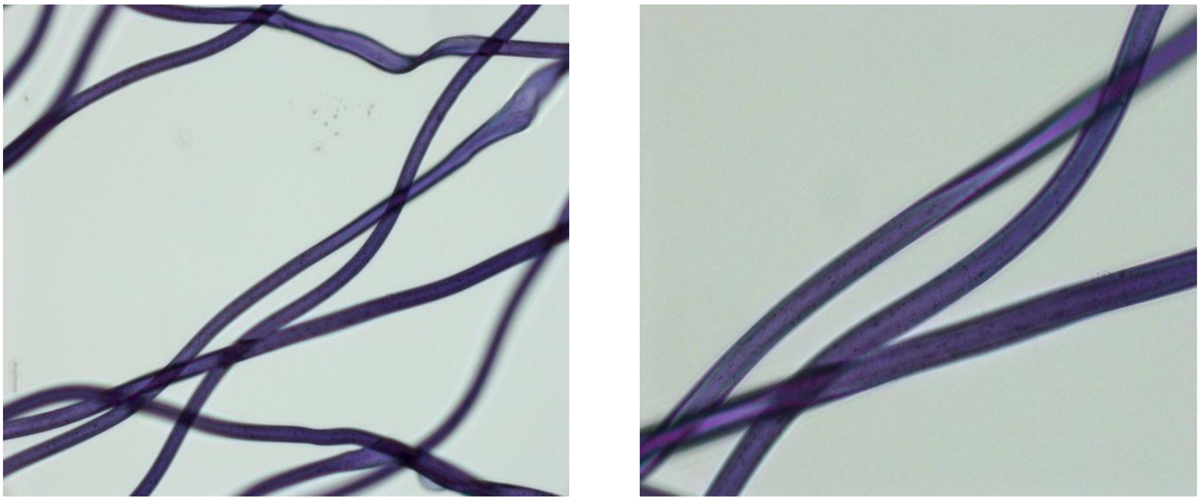


Figure 8: Microscope longitudinal analysis of sample's **307** remaining residue after applying the chemical dissolution method 4 (76% PTT - 17% PET - 7% polyamide) 200x (left) and 400x (right)

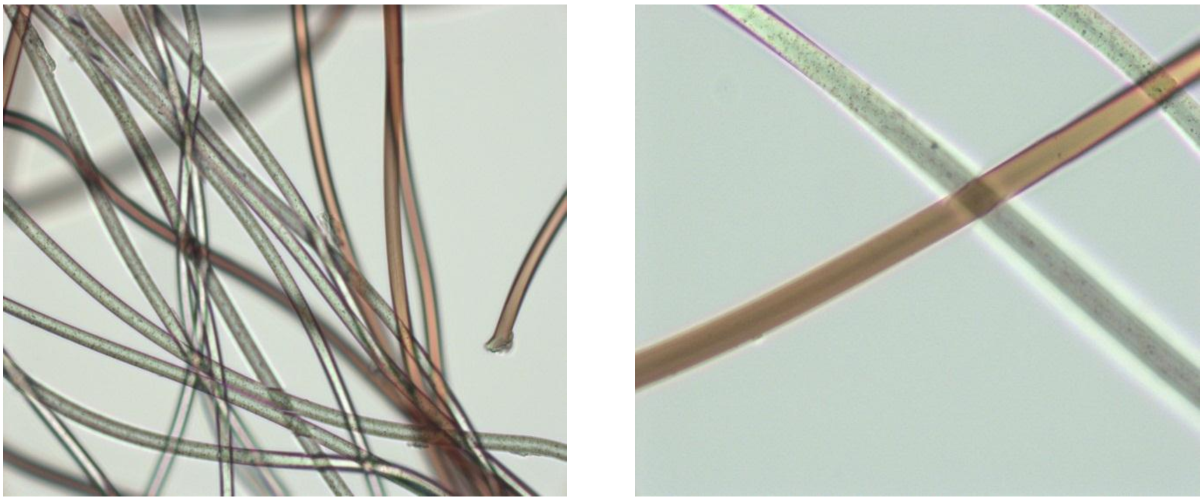


Figure 9: Microscope longitudinal analysis of sample **312** (60% PTT - 40% PET) 200x (left) and 400x (right)

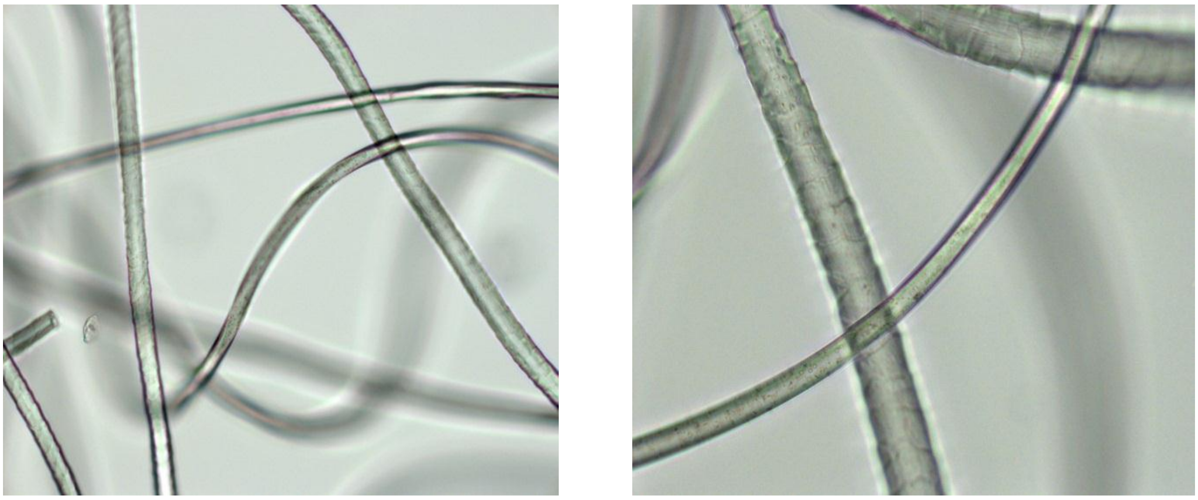


Figure 10: Microscope longitudinal analysis of sample **322** (76% PTT - 24% wool) 200x (left) and 400x (right)

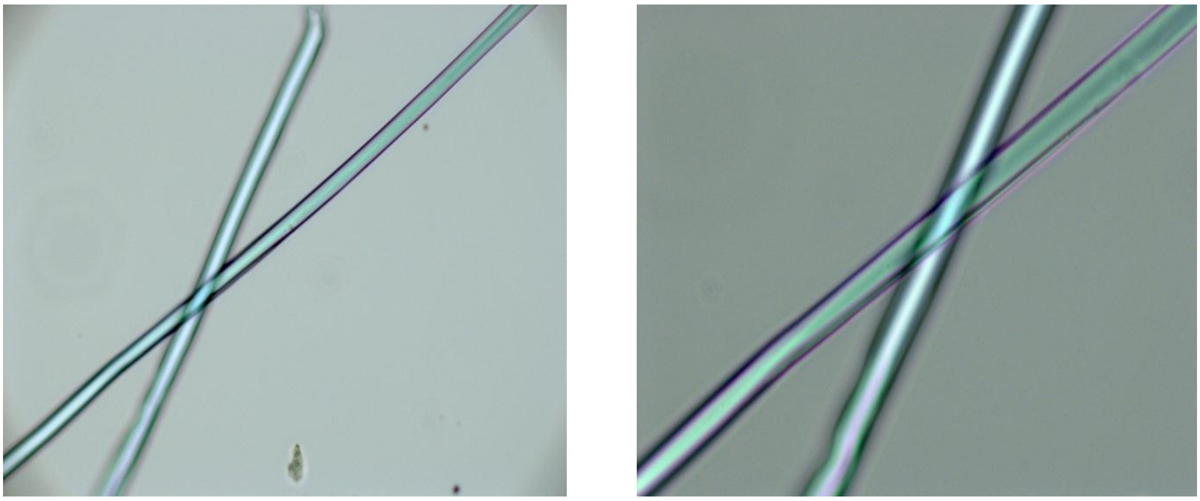


Figure 11: Microscope longitudinal analysis of sample's **322** remaining residue after applying the chemical dissolution method 2 (76% PTT - 24% wool) 200x (left) and 400x (right)

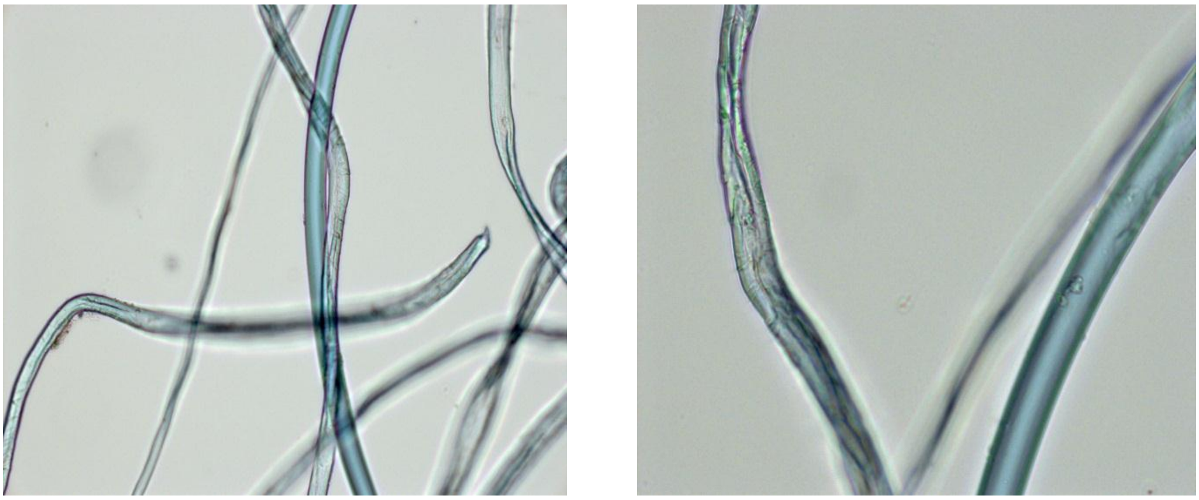


Figure 12: Microscope longitudinal analysis of sample **323** (40% PTT - 60% cotton) 200x (left) and 400x (right)

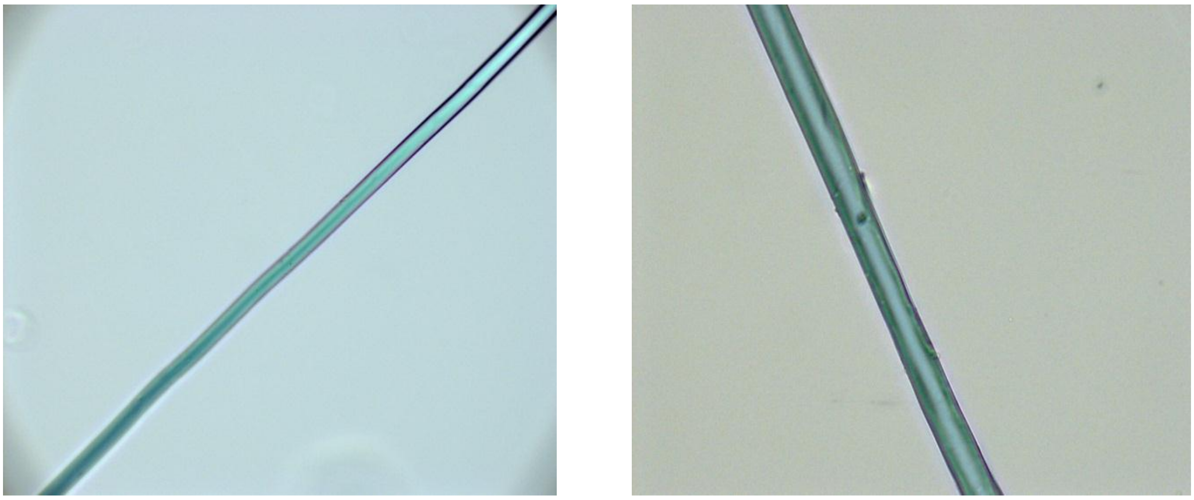


Figure 13: Microscope longitudinal analysis of sample's **323** remaining residue after applying the chemical dissolution method 7 (40% PTT - 60% cotton) 200x (left) and 400x (right)

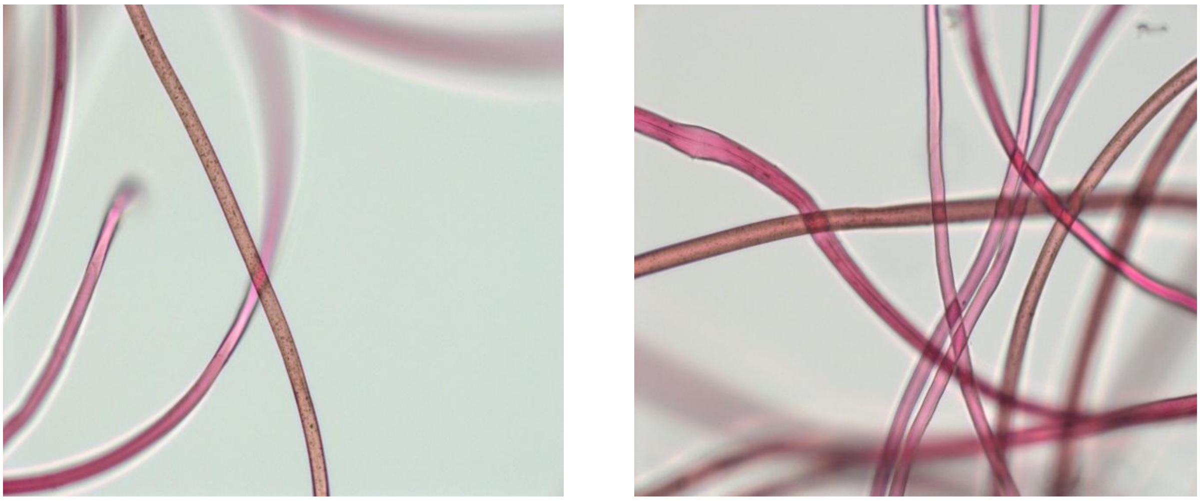


Figure 14: Microscope longitudinal analysis of sample **324** (37% PTT -58% modal – 5% elastane) 200x (left) and 400x (right)

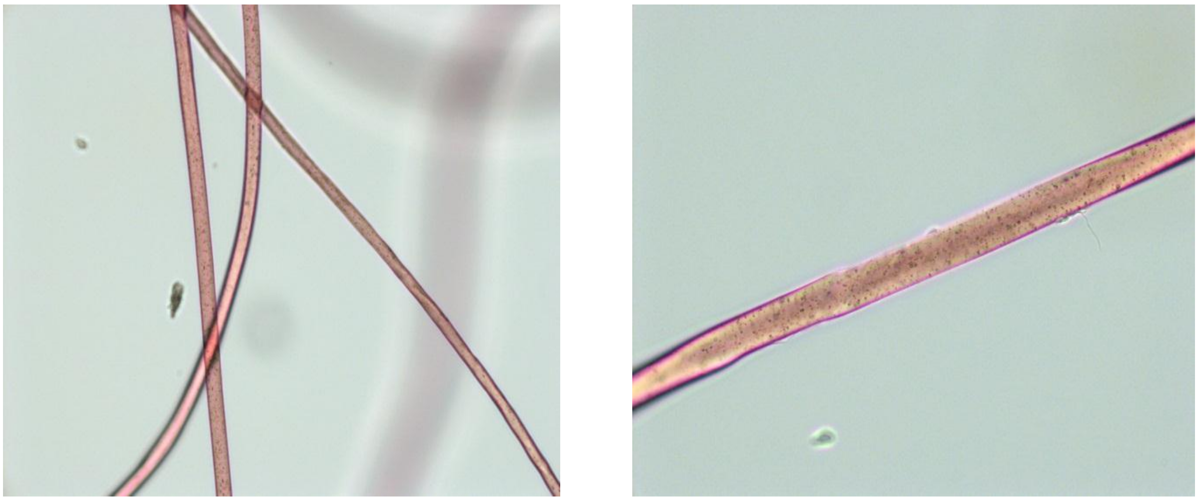


Figure 15: Microscope longitudinal analysis of the sample's **324** remaining residue after applying the chemical dissolution methods 7 and 8 (37% PTT -58% modal – 5% elastane) 200x (left) and 400x (right)

Annex III

Spectroscopic analysis FT-IR

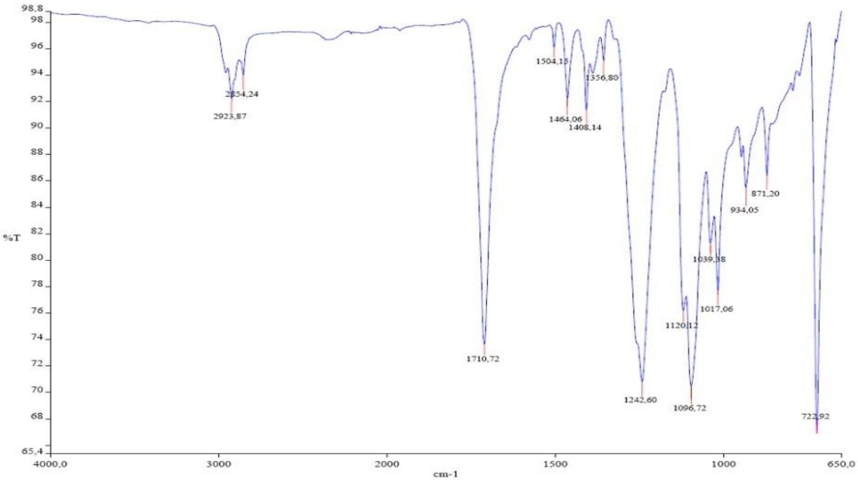


Figure 1: FT-IR spectrum of not-treated PTT (sample **297**).

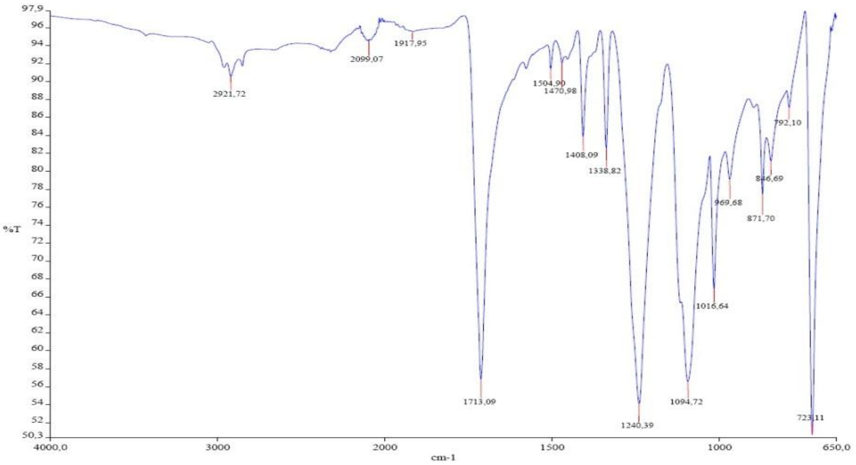


Figure 2: FT-IR spectrum of not-treated PET (sample **015**).

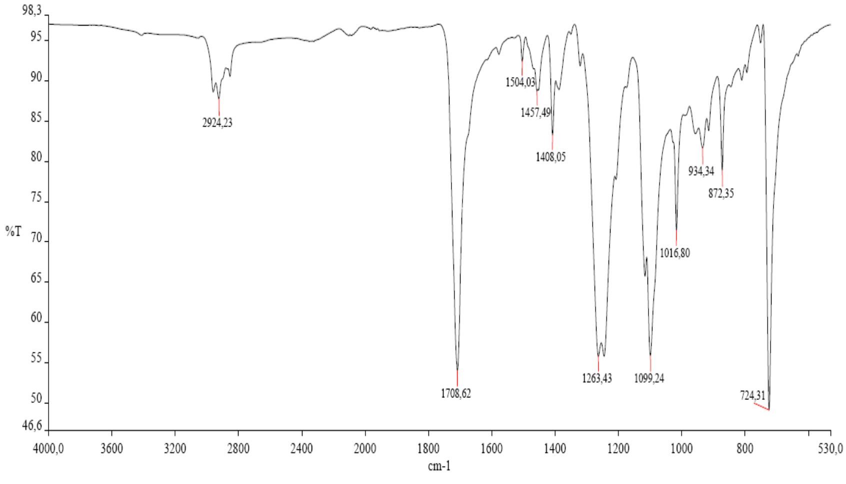


Figure 3: FT-IR spectrum of not-treated PBT (sample **086**).

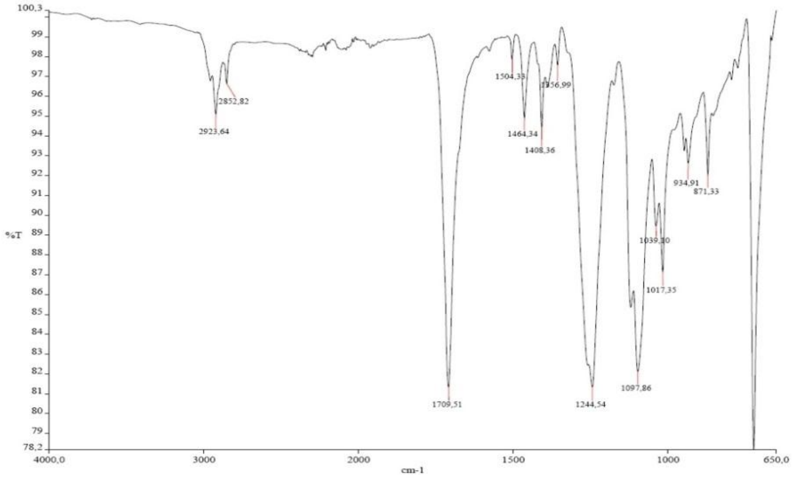


Figure 4: FT-IR spectrum of PTT pre-treated with petroleum ether in Soxhlet (sample **297**).

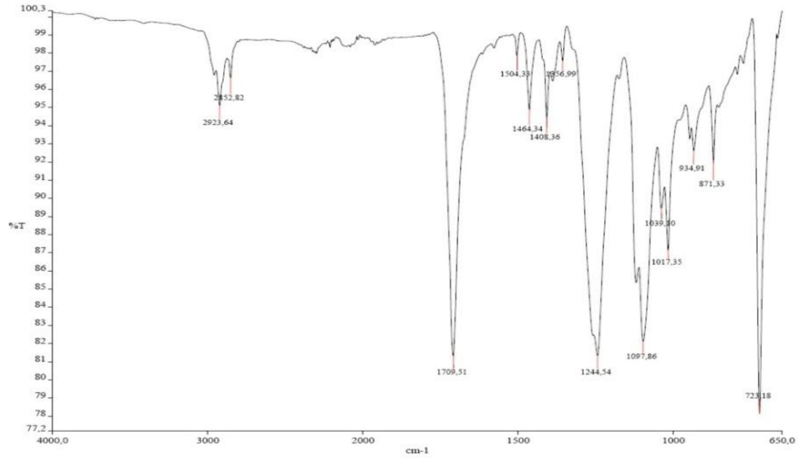


Figure 5: FT-IR spectrum of PTT pre-treated with petroleum ether in Soxhtec (sample **297**).

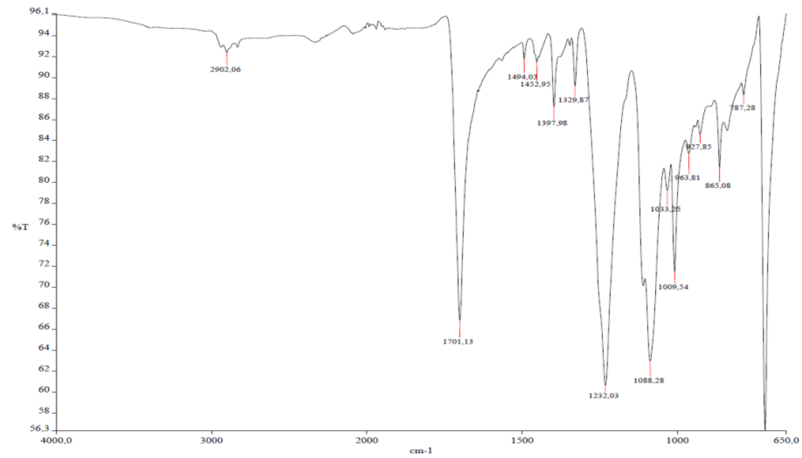


Figure 6: FT-IR spectrum of binary mixture PET/PTT (sample **315**).

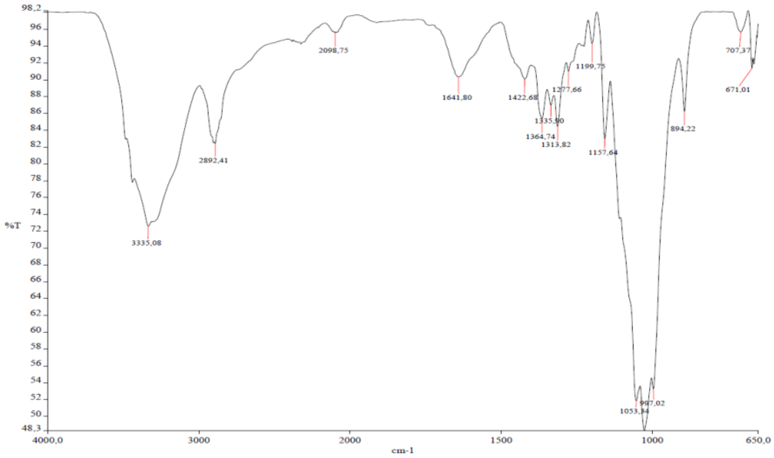


Figure 7: FT-IR spectrum of cotton (sample **005**).

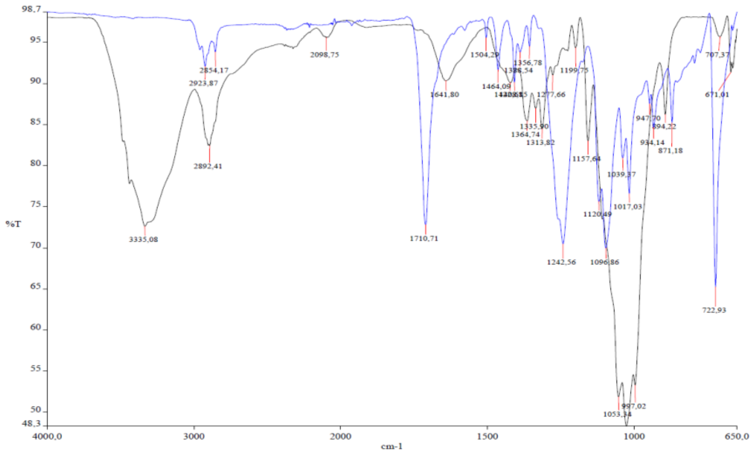


Figure 8: Overlay of FT-IR spectra of cotton (sample **005**, black) and PTT (sample **297**, blue).

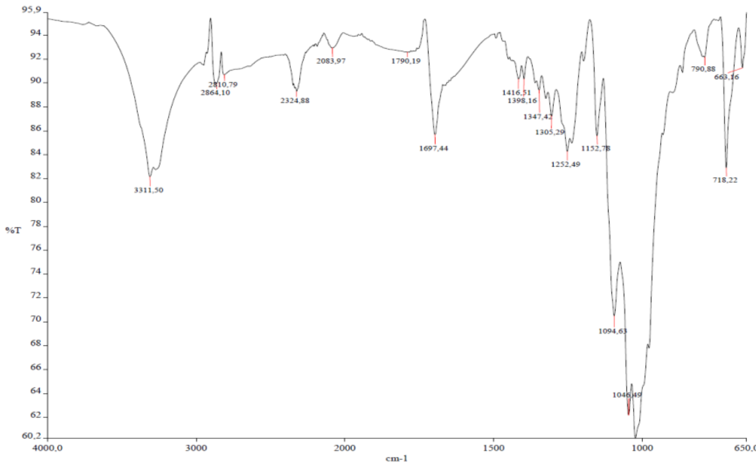


Figure 9: FT-IR spectrum of binary mixture cotton/PTT (sample **321**).

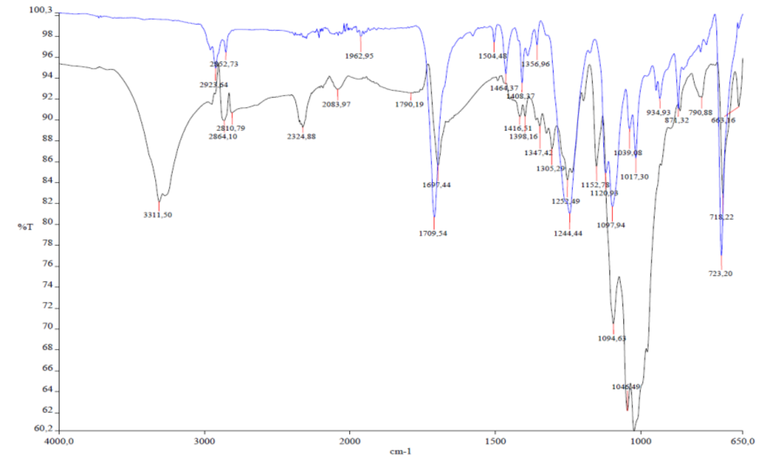


Figure 10: Overlay of FT-IR spectra of PTT (sample **297**, blue) and binary mixture cotton/PTT (sample **321, black**).

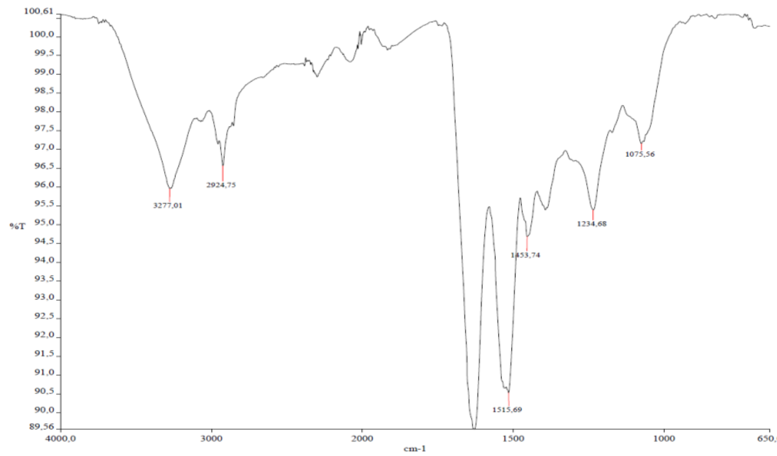


Figure 11: FT-IR spectrum of wool (sample **013**).

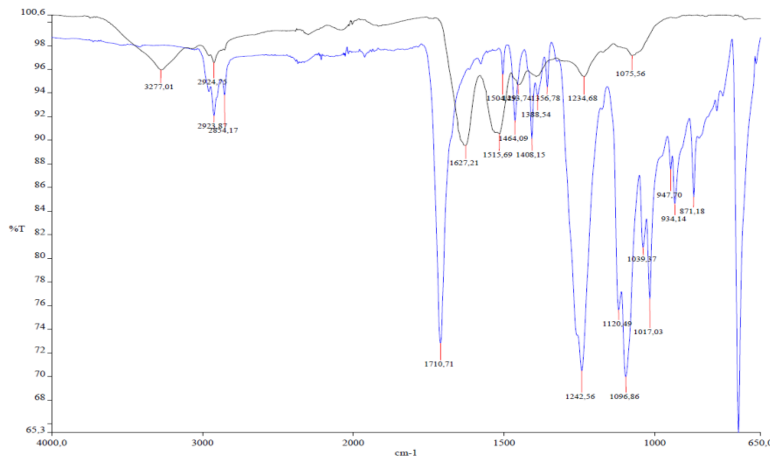


Figure 12: Overlay of FT-IR spectra of wool (sample **013**, black) and PTT (sample **297**, blue).

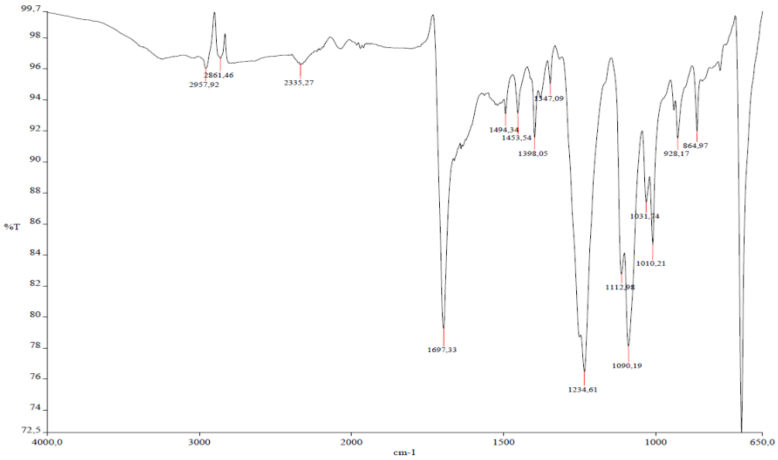


Figure 13: FT-IR spectrum of binary mixture wool/PTT (sample **322**).

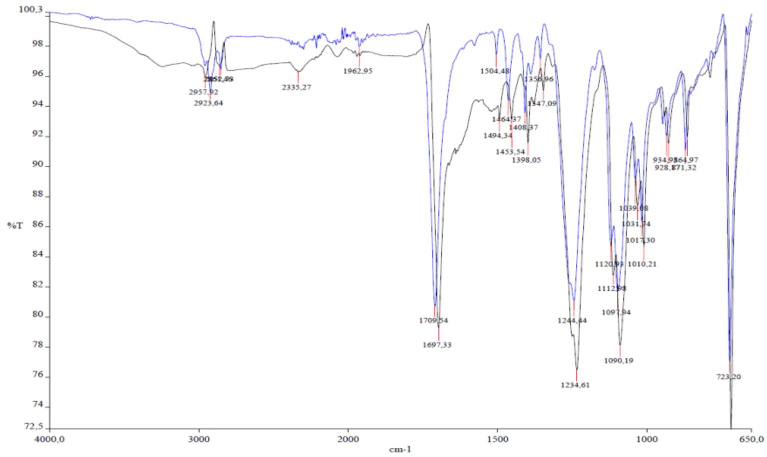


Figure 14: Overlay of FT-IR spectra of PTT (sample **297**, blue) and binary mixture wool/PTT (sample **322, black**).

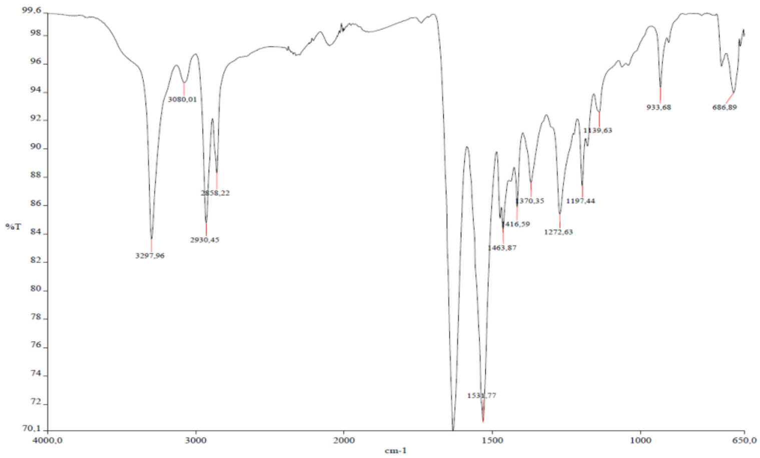


Figure 15: FT-IR spectrum of polyamide (sample **038**).

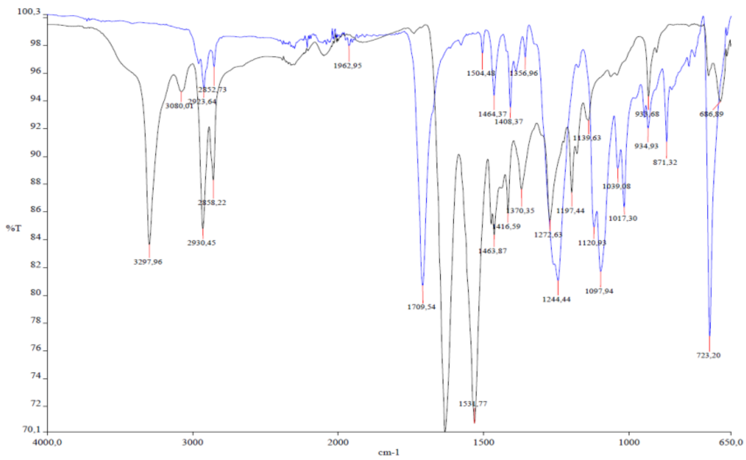


Figure 16: Overlay of FT-IR spectra of polyamide (sample **038**, black) and PTT (sample **297**, blue).

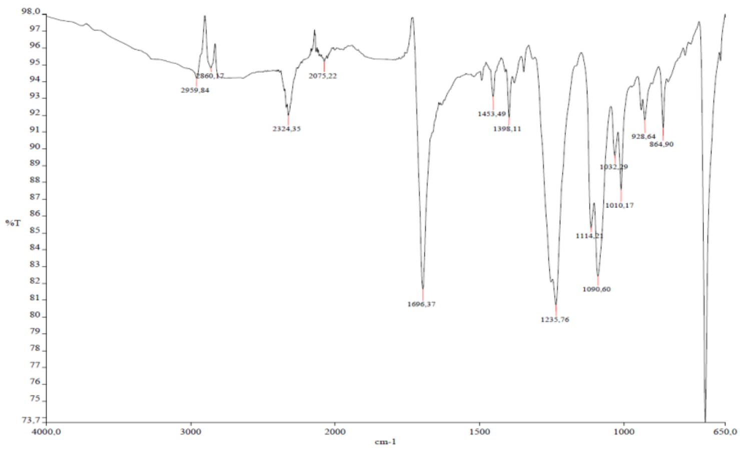


Figure 17: FT-IR spectrum of binary mixture polyamide/PTT (sample **306**).

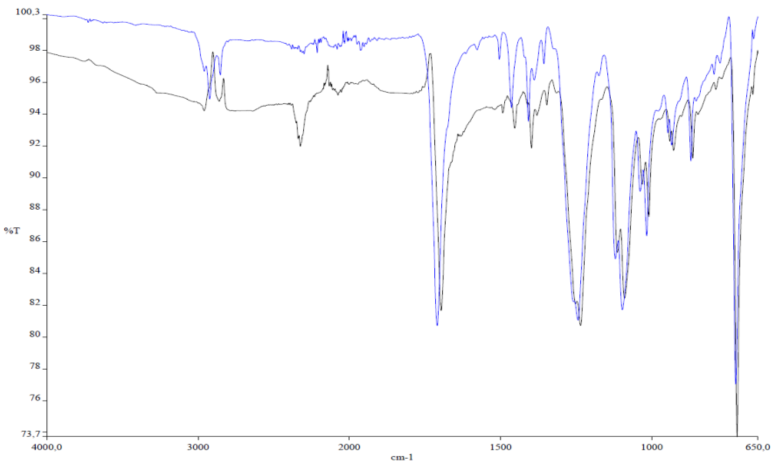


Figure 18: Overlay of FT-IR spectra of PTT (sample **297**, blue) and binary mixture polyamide/PTT (sample **306, black**).

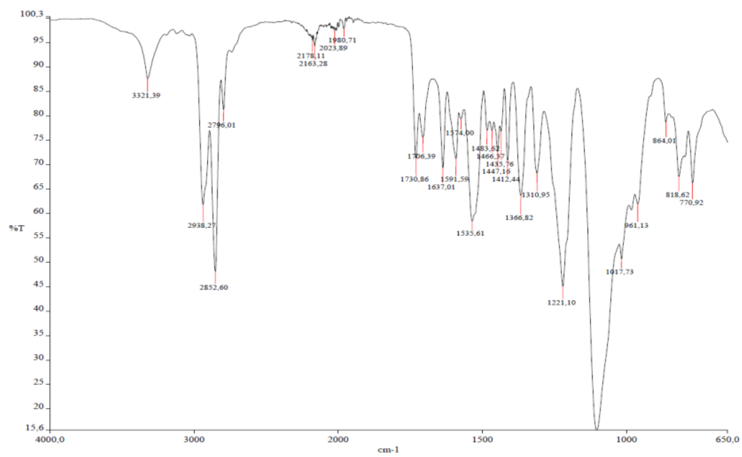


Figure 19: FT-IR spectrum of elastane (sample **064**).

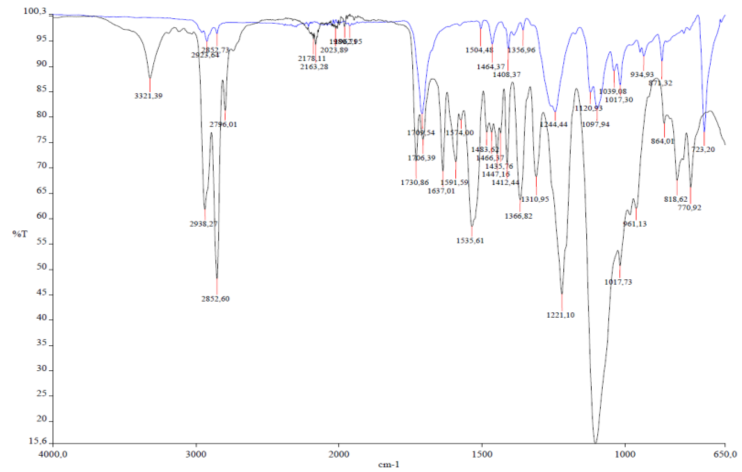


Figure 20: Overlay of FT-IR spectra of elastane (sample **064**, black) and PTT (sample **297**, blue).

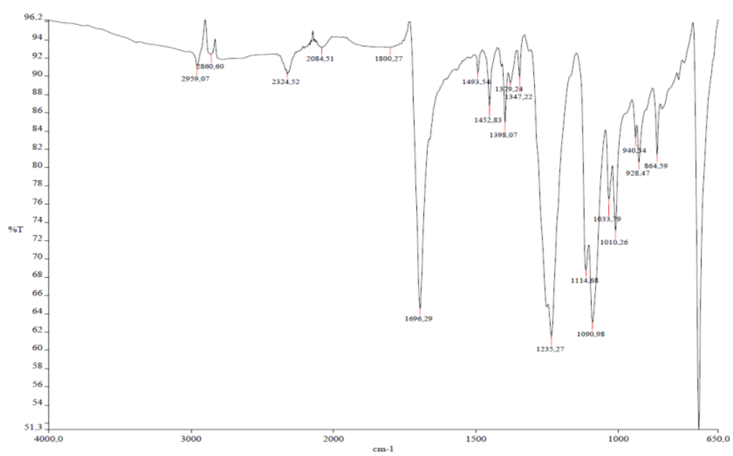


Figure 21: FT-IR spectrum of binary mixture elastane/PTT (sample **305**).

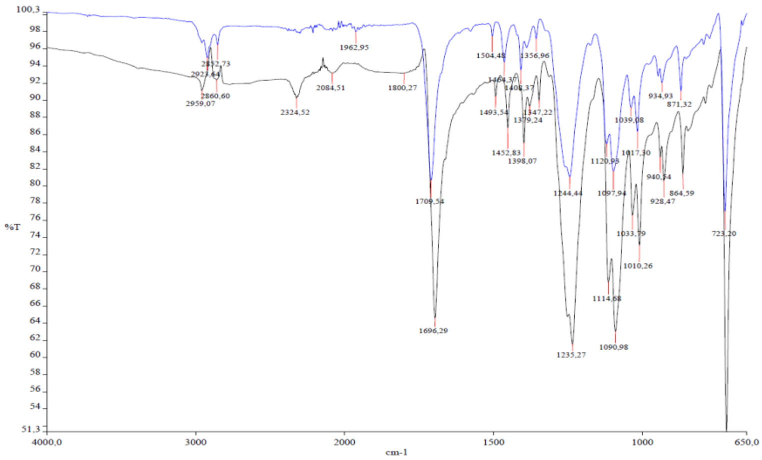


Figure 22: Overlay of FT-IR spectra of PTT (sample **297**, blue) and binary mixture elastane/PTT (sample **324, black**).

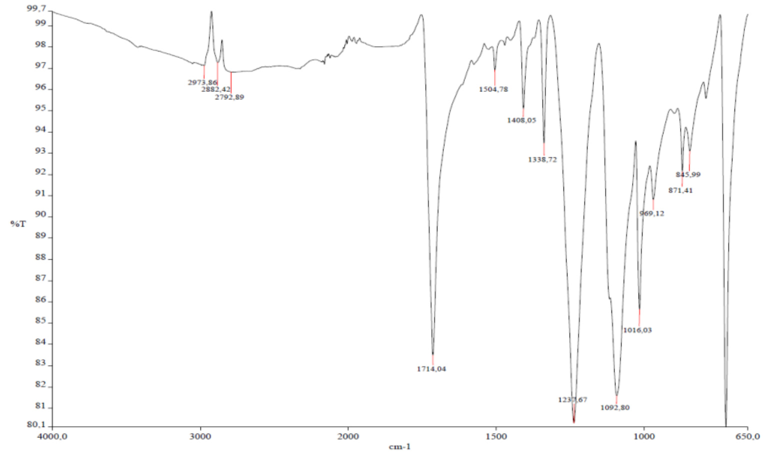


Figure 23: FT-IR spectrum of modal (sample **128**).

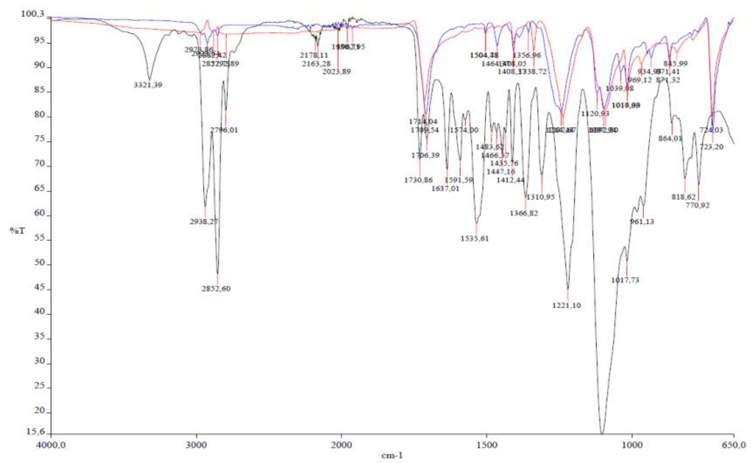


Figure 24: Overlay of FT-IR spectra of modal (sample **128**, pink), elastane (sample **064**, black) and PTT (sample **297**, blue).

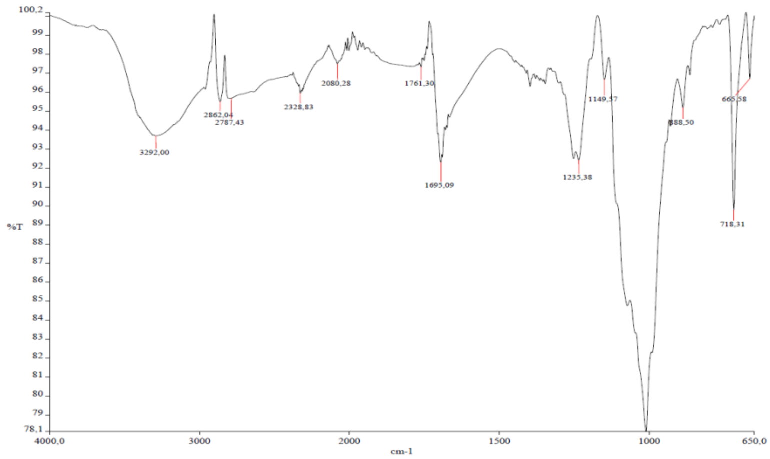


Figure 25: FT-IR spectrum of ternary mixture modal/elastane/PTT (sample **324**).

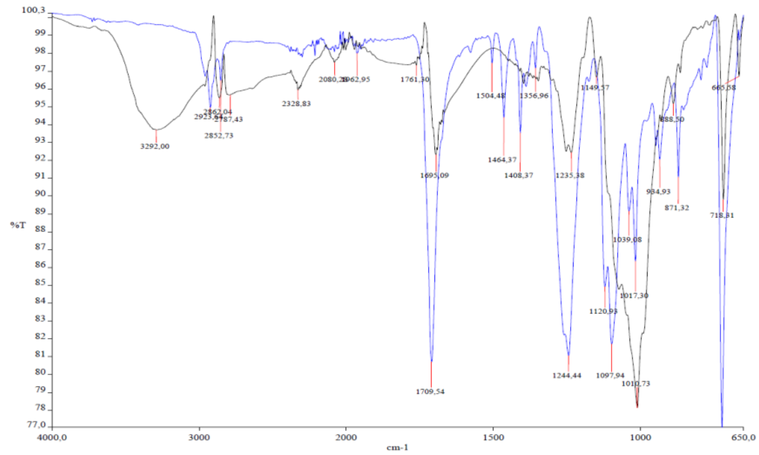


Figure 26: Overlay of FT-IR spectra of PTT (sample **297**, blue) and ternary mixture modal/elastane/PTT (sample **324, black**).

Annex IV

Analysis of composition

Pre-treatment with petroleum ether in Soxhlet

JRC code	untreated sample mass g	pre-treated sample mass g	mass loss g	mass loss %
296-1	2.0536	2.0387	0.0149	0.73
296-2	2.0535	2.0369	0.0166	0.81
296-3	2.0471	2.0333	0.0138	0.67
296-4	2.0492	2.0345	0.0147	0.72
296-5	2.0482	2.0338	0.0144	0.70
296-6	2.0550	2.0390	0.0160	0.78
			average	**0.73**
			confidence limit	**0.05**
			SD	**0.05**
			CV %	**6.78**

Pre-treatment with petroleum ether in Soxhtec

JRC code	untreated sample mass g	pre-treated sample mass g	mass loss g	mass loss %
293-1	2.1522	2.1377	0.0145	0.67
293-2	2.1523	2.1383	0.0140	0.65
293-3	2.1528	2.1415	0.0113	0.52
293-4	2.1583	2.1464	0.0119	0.55
293-5	2.1476	2.1331	0.0145	0.68
293-6	2.1515	2.1383	0.0132	0.61
			average	**0.61**
			confidence limit	**0.07**
			SD	**0.06**
			CV %	**10.41**

JRC code	untreated sample mass g	pre-treated sample mass g	mass loss g	mass loss %
296-1	2.1414	2.1294	0.0120	0.56
296-2	1.9965	1.9842	0.0123	0.62
296-3	2.0617	2.0494	0.0123	0.60
296-4	1.9780	1.9633	0.0147	0.74
296-5	1.9280	1.9160	0.0120	0.62
296-6	1.9425	1.9305	0.0120	0.62
			average	**0.63**
			confidence limit	**0.06**
			SD	**0.06**
			CV %	**9.87**

Mass loss due to consecutive pre-treatments with petroleum ether in Soxhtec

1st pre-treatment

JRC code	untreated sample mass g	pre-treated sample mass g	mass loss g	mass loss %
296-1	2.0675	2.0554	0.0121	0.59
296-2	2.0532	2.0388	0.0144	0.70
296-3	2.0541	2.0419	0.0122	0.59
296-4	2.0449	2.0293	0.0156	0.76
296-5	2.0468	2.0344	0.0124	0.61
296-6	2.0599	2.0473	0.0126	0.61
			average	**0.64**
			confidence limit	**0.08**
			SD	**0.07**
			CV %	**11.18**

2nd pre-treatment

JRC code	untreated sample mass g	pre-treated sample mass g	mass loss g	mass loss %
296-1	2.0554	2.0527	0.0027	0.13
296-2	2.0388	2.0365	0.0023	0.11
296-3	2.0419	2.0386	0.0033	0.16
296-4	2.0293	2.0262	0.0031	0.15
296-5	2.0344	2.0322	0.0022	0.11
296-6	2.0473	2.0457	0.0016	0.08
			average	**0.12**
			confidence limit	**0.03**
			SD	**0.03**
			CV %	**24.89**

3rd pre-treatment

JRC code	untreated sample mass g	pre-treated sample mass g	mass loss g	mass loss %
296-1	2.0527	2.0493	0.0034	0.17
296-2	2.0365	2.0336	0.0029	0.14
296-3	2.0386	2.0361	0.0025	0.12
296-4	2.0262	2.0241	0.0021	0.10
296-5	2.0322	2.0297	0.0025	0.12
296-6	2.0457	2.0434	0.0023	0.11
			average	**0.13**
			confidence limit	**0.02**
			SD	**0.02**
			CV %	**17.47**

Agreed allowance

Untreated PTT (samples 296, 297, 299)

JRC code	dried sample mass g	wet sample mass g	water mass g	agreed allowance %
296-1	1.9913	1.9983	0.0070	0.35
296-2	2.0470	2.0543	0.0073	0.36
296-3	2.0437	2.0492	0.0055	0.27
296-4	2.0461	2.0524	0.0063	0.31
296-5	2.0465	2.0561	0.0096	0.47
296-6	2.0465	2.0541	0.0076	0.37
296-7	2.0521	2.0599	0.0078	0.38
296-8	2.0498	2.0583	0.0085	0.41
296-9	2.0421	2.0512	0.0091	0.45
296-10	2.0498	2.0582	0.0084	0.41
			average	**0.38**
			confidence limit	**0.04**
			SD	**0.06**
			CV %	**16.09**

JRC code	dried sample mass g	wet sample mass g	water mass g	agreed allowance %
297-1	2.0123	2.0178	0.0055	0.27
297-2	2.0138	2.0217	0.0079	0.39
297-3	1.9843	1.9899	0.0056	0.28
297-4	1.9868	1.9935	0.0067	0.34
297-5	1.9925	1.9975	0.0050	0.25
297-6	2.0342	2.0406	0.0064	0.31
297-7	2.0237	2.0292	0.0055	0.27
297-8	2.0208	2.0258	0.0050	0.25
297-9	2.0173	2.0252	0.0079	0.39
297-10	1.9983	2.0047	0.0064	0.32
			average	**0.31**
			confidence limit	**0.04**
			SD	**0.05**
			CV %	**17.19**

JRC code	dried sample mass g	wet sample mass g	water mass g	agreed allowance %
299-1	1.9822	1.9894	0.0072	0.36
299-2	1.8366	1.8446	0.0080	0.44
299-3	1.9479	1.9558	0.0079	0.41
299-4	1.9234	1.9312	0.0078	0.41
299-5	2.1452	2.1529	0.0077	0.36
299-6	1.9621	1.9712	0.0091	0.46
299-7	2.1119	2.1200	0.0081	0.38
299-8	1.9800	1.9880	0.0080	0.40
299-9	2.0446	2.0534	0.0088	0.43
299-10	1.9017	1.9081	0.0064	0.34
			average	**0.40**
			confidence limit	**0.03**
			SD	**0.04**
			CV %	**9.77**

Agreed allowance

Treated PTT in Soxhlet (samples 296, 297, 299)

JRC code	dried sample mass g	wet sample mass g	water mass g	agreed allowance %
296-1	2.0208	2.0263	0.0055	0.27
296-2	2.0960	2.1025	0.0065	0.31
296-3	2.0856	2.0940	0.0084	0.40
296-4	2.0356	2.0417	0.0061	0.30
296-5	2.0590	2.0645	0.0055	0.27
296-6	2.0714	2.0783	0.0069	0.33
296-7	2.0353	2.0422	0.0069	0.34
296-8	2.0135	2.0191	0.0056	0.28
296-9	2.0113	2.0192	0.0079	0.39
296-10	1.9877	1.9928	0.0051	0.26
			average	**0.32**
			confidence limit	**0.04**
			SD	**0.05**
			CV %	**16.32**

JRC code	dried sample mass g	wet sample mass g	water mass g	agreed allowance %
297-1	2.0642	2.0689	0.0047	0.23
297-2	2.1118	2.1186	0.0068	0.32
297-3	2.0835	2.0904	0.0069	0.33
297-4	2.0291	2.0352	0.0061	0.30
297-5	2.1227	2.1290	0.0063	0.30
297-6	1.9938	1.9989	0.0051	0.26
297-7	1.9820	1.9874	0.0054	0.27
297-8	1.9950	1.9997	0.0047	0.24
297-9	2.1716	2.1762	0.0046	0.21
297-10	2.0948	2.1014	0.0066	0.32
			average	**0.28**
			confidence limit	**0.03**
			SD	**0.04**
			CV %	**15.33**

JRC code	dried sample mass g	wet sample mass g	water mass g	agreed allowance %
299-1	2.0342	2.0413	0.0071	0.35
299-2	1.8981	1.9052	0.0071	0.37
299-3	1.8776	1.8849	0.0073	0.39
299-4	1.8951	1.9016	0.0065	0.34
299-5	1.7716	1.7795	0.0079	0.45
299-6	1.9633	1.9703	0.0070	0.36
299-7	2.0356	2.0423	0.0067	0.33
299-8	1.9764	1.9843	0.0079	0.40
299-9	1.9082	1.9162	0.0080	0.42
299-10	1.9439	1.9526	0.0087	0.45
			average	**0.39**
			confidence limit	**0.03**
			SD	**0.04**
			CV %	**10.99**

Agreed allowance - Dupont's results

DuPont code	dried sample mass g	wet sample mass g	water mass g	agreed allowance %
PTT-04-1	2.3632	2.3569	0.0063	0.27
PTT-04-2	2.2506	2.2448	0.0058	0.26
PTT-04-3	2.4561	2.4498	0.0063	0.26
PTT-04-4	2.4505	2.4442	0.0063	0.26
PTT-04-5	2.4638	2.4566	0.0072	0.29
PTT-04-6	2.4562	2.4481	0.0081	0.33
			average	**0.28**
			confidence limit	**0.03**
			SD	**0.03**
			CV %	**10.65**

Solubility properties - PTT - *d* correction factors

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M1-296-1	1.0517	1.0450	99.36	0.6371	1.006
M1-296-2	1.0128	1.0054	99.27	0.7306	1.007
M1-296-3	1.1465	1.1376	99.22	0.7763	1.008
M1-296-4	1.1138	1.1058	99.28	0.7183	1.007
M1-296-5	1.0002	0.9923	99.21	0.7898	1.008
M1-296-6	1.0650	1.0566	99.21	0.7887	1.008
M1-296-7	1.0740	1.0663	99.28	0.7169	1.007
M1-296-8	1.0213	1.0155	99.43	0.5679	1.006
M1-296-9	1.2884	1.2795	99.31	0.6908	1.007
M1-296-10	1.0201	1.0147	99.47	0.5294	1.005
M1-296-11	0.9632	0.9536	99.00	0.9967	1.010
M1-296-12	0.9867	0.9764	98.96	1.0439	1.011
M1-296-13	0.9835	0.9739	99.02	0.9761	1.010
M1-296-14	0.9996	0.9895	98.99	1.0104	1.010
M1-296-15	0.9626	0.9517	98.87	1.1323	1.011
M1-296-16	0.9819	0.9725	99.04	0.9573	1.010
M1-296-17	0.9047	0.8939	98.81	1.1938	1.012
M1-296-18	0.9900	0.9819	99.18	0.8182	1.008
M1-296-19	0.9751	0.9663	99.10	0.9025	1.009
M1-296-20	0.9557	0.9462	99.01	0.9940	1.010
		average	**99.15**	**0.85**	**1.009**
		confidence limit	**0.09**	**0.09**	**0.001**
		SD	**0.18**	**0.18**	**0.002**
		CV %	**0.19**	**21.79**	**0.187**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M2-296-1	1.0373	1.0422	-0.47	0.9953	0.995
M2-296-2	1.0706	1.0752	-0.43	0.9957	0.996
M2-296-3	0.9997	1.0016	-0.19	0.9981	0.998
M2-296-4	1.0255	1.0283	-0.27	0.9973	0.997
M2-296-5	1.0225	1.0252	-0.26	0.9974	0.997
M2-296-6	1.0340	1.0364	-0.23	0.9977	0.998
M2-296-7	0.9997	1.0015	-0.18	0.9982	0.998
M2-296-8	1.0078	1.0144	-0.65	0.9935	0.993
M2-296-9	1.0393	1.0459	-0.64	0.9937	0.994
M2-296-10	0.9966	0.9984	-0.18	0.9982	0.998
M2-296-11	1.0508	1.0523	-0.14	0.9986	0.999
M2-296-12	1.0487	1.0490	-0.03	0.9997	1.000
M2-296-13	1.0129	1.0110	0.19	1.0019	1.002
M2-296-14	0.9975	0.9982	-0.07	0.9993	0.999
M2-296-15	1.0868	1.0893	-0.23	0.9977	0.998
M2-296-16	1.0652	1.0651	0.01	1.0001	1.000
M2-296-17	1.0408	1.0431	-0.22	0.9978	0.998
M2-296-18	1.0751	1.0736	0.14	1.0014	1.001
M2-296-19	1.0391	1.0385	0.06	1.0006	1.001
M2-296-20	1.0714	1.0699	0.14	1.0014	1.001
		average	**100.18**	**-0.18**	**0.998**
		confidence limit	**0.11**	**0.11**	**0.001**
		SD	**0.24**	**0.24**	**0.002**
		CV %	**0.24**	**-129.39**	**0.237**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M3-296-1	1.0951	1.0925	99.76	0.24	1.002
M3-296-2	0.9911	0.9890	99.79	0.21	1.002
M3-296-3	1.0414	1.0388	99.75	0.25	1.003
M3-296-4	1.0597	1.0574	99.78	0.22	1.002
M3-296-5	1.0273	1.0232	99.60	0.40	1.004
M3-296-6	1.0511	1.0473	99.64	0.36	1.004
M3-296-7	1.0420	1.0390	99.71	0.29	1.003
M3-296-8	1.0349	1.0336	99.87	0.13	1.001
M3-296-9	1.0469	1.0436	99.68	0.32	1.003
M3-296-10	1.0512	1.0492	99.81	0.19	1.002
M3-296-11	0.9505	0.9456	99.48	0.52	1.005
M3-296-12	0.9667	0.9634	99.66	0.34	1.003
M3-296-13	0.9727	0.9685	99.57	0.43	1.004
M3-296-14	1.0333	1.0276	99.45	0.55	1.006
M3-296-15	1.1056	1.0994	99.44	0.56	1.006
M3-296-16	1.0662	1.0600	99.42	0.58	1.006
M3-296-17	1.0404	1.0363	99.61	0.39	1.004
M3-296-18	1.0241	1.0220	99.79	0.21	1.002
M3-296-19	0.9661	0.9647	99.86	0.14	1.001
M3-296-20	1.0468	1.0449	99.82	0.18	1.002
		average	**99.67**	**0.33**	**1.003**
		confidence limit	**0.07**	**0.07**	**0.001**
		SD	**0.14**	**0.14**	**0.001**
		CV %	**0.15**	**44.45**	**0.145**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M4-296-1	1.1204	1.1128	99.32	0.6783	1.007
M4-296-2	1.0715	1.0691	99.78	0.2240	1.002
M4-296-3	1.0303	1.0238	99.37	0.6309	1.006
M4-296-4	1.0136	1.0065	99.30	0.7005	1.007
M4-296-5	1.0423	1.0383	99.62	0.3838	1.004
M4-296-6	1.0269	1.0224	99.56	0.4382	1.004
M4-296-7	1.0825	1.0793	99.70	0.2956	1.003
M4-296-8	1.1153	1.1075	99.30	0.6994	1.007
M4-296-9	1.0068	0.9989	99.22	0.7847	1.008
M4-296-10	0.9936	0.9860	99.24	0.7649	1.008
M4-296-11	0.9581	0.9504	99.20	0.8037	1.008
M4-296-12	0.9986	0.9928	99.42	0.5808	1.006
M4-296-13	0.9762	0.9676	99.12	0.8810	1.009
M4-296-14	0.9543	0.9454	99.07	0.9326	1.009
M4-296-15	0.9134	0.9072	99.32	0.6788	1.007
M4-296-16	0.9688	0.9592	99.01	0.9909	1.010
M4-296-17	0.9555	0.9491	99.33	0.6698	1.007
M4-296-18	0.9423	0.9361	99.34	0.6580	1.007
M4-296-19	0.9188	0.9120	99.26	0.7401	1.007
M4-296-20	0.8497	0.8398	98.83	1.1651	1.012
		average	**99.31**	**0.69**	**1.007**
		confidence limit	**0.11**	**0.11**	**0.001**
		SD	**0.23**	**0.23**	**0.002**
		CV %	**0.23**	**33.29**	**0.230**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M5-296-1	1.0388	1.0320	99.35	0.6546	1.007
M5-296-2	1.0226	1.0156	99.32	0.6845	1.007
M5-296-3	1.1020	1.0916	99.06	0.9437	1.010
M5-296-4	1.0309	1.0204	98.98	1.0185	1.010
M5-296-5	1.0491	1.0403	99.16	0.8388	1.008
M5-296-6	1.0426	1.0315	98.94	1.0646	1.011
M5-296-7	1.0609	1.0541	99.36	0.6410	1.006
M5-296-8	1.0742	1.0639	99.04	0.9589	1.010
M5-296-9	1.0476	1.0367	98.96	1.0405	1.011
M5-296-10	1.0946	1.0842	99.05	0.9501	1.010
M5-296-11	0.9677	0.9543	98.62	1.3847	1.014
M5-296-12	0.9854	0.9729	98.73	1.2685	1.013
M5-296-13	0.9977	0.9848	98.71	1.2930	1.013
M5-296-14	0.9631	0.9493	98.57	1.4329	1.015
M5-296-15	1.0322	1.0188	98.70	1.2982	1.013
M5-296-16	1.1024	1.0876	98.66	1.3425	1.014
M5-296-17	1.0817	1.0693	98.85	1.1463	1.012
M5-296-18	1.0836	1.0700	98.74	1.2551	1.013
M5-296-19	1.0543	1.0399	98.63	1.3658	1.014
M5-296-20	1.0635	1.0516	98.88	1.1189	1.011
		average	**98.91**	**1.09**	**1.011**
		confidence limit	**0.12**	**0.12**	**0.001**
		SD	**0.25**	**0.25**	**0.003**
		CV %	**0.25**	**22.93**	**0.251**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M6-296-1	1.0152	0.9864	97.16	2.8369	1.029
M6-296-2	0.9914	0.9631	97.15	2.8545	1.029
M6-296-3	1.0665	1.0369	97.22	2.7754	1.029
M6-296-4	0.9975	0.9684	97.08	2.9173	1.030
M6-296-5	0.9887	0.9597	97.07	2.9331	1.030
M6-296-6	1.0382	1.0071	97.00	2.9956	1.031
M6-296-7	1.0485	1.0188	97.17	2.8326	1.029
M6-296-8	1.0020	0.9744	97.25	2.7545	1.028
M6-296-9	0.9932	0.9661	97.27	2.7286	1.028
M6-296-10	1.0425	1.0144	97.30	2.6954	1.028
M6-296-11	0.9551	0.9283	97.19	2.8060	1.029
M6-296-12	0.9719	0.9434	97.07	2.9324	1.030
M6-296-13	1.0131	0.9841	97.14	2.8625	1.029
M6-296-14	1.0244	0.9964	97.27	2.7333	1.028
M6-296-15	1.0240	0.9956	97.23	2.7734	1.029
M6-296-16	0.9345	0.9077	97.13	2.8678	1.030
M6-296-17	1.0355	1.0064	97.19	2.8102	1.029
M6-296-18	0.9709	0.9442	97.25	2.7500	1.028
M6-296-19	1.0298	1.0012	97.22	2.7772	1.029
M6-296-20	1.0587	1.0298	97.27	2.7298	1.028
		average	**97.18**	**2.82**	**1.029**
		confidence limit	**0.04**	**0.04**	**0.000**
		SD	**0.08**	**0.08**	**0.001**
		CV %	**0.08**	**2.89**	**0.084**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M7-293-1	1.4097	1.3903	98.62	1.3762	1.014
M7-293-2	1.0653	1.0494	98.51	1.4925	1.015
M7-293-3	1.1518	1.1345	98.50	1.5020	1.015
M7-293-4	1.3700	1.3491	98.47	1.5255	1.015
M7-293-5	1.2244	1.2065	98.54	1.4619	1.015
M7-293-6	1.0393	1.0228	98.41	1.5876	1.016
M7-293-7	1.5874	1.5699	98.90	1.1024	1.011
M7-293-8	1.0991	1.0774	98.03	1.9743	1.020
M7-293-9	1.0965	1.0751	98.05	1.9517	1.020
M7-293-10	1.1549	1.1423	98.91	1.0910	1.011
M7-293-11	0.9412	0.9139	97.10	2.9006	1.030
M7-293-12	0.9580	0.9367	97.78	2.2234	1.023
M7-293-13	0.9351	0.9145	97.80	2.2030	1.023
M7-293-14	0.9655	0.9502	98.42	1.5847	1.016
M7-293-15	0.9297	0.9169	98.62	1.3768	1.014
		average	98.31	1.69	1.017
		confidence limit	0.27	0.27	0.003
		SD	0.48	0.48	0.005
		CV %	0.49	28.45	0.492

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M7-294-1	1.0780	1.0757	99.79	0.2134	1.002
M7-294-2	1.0293	1.0277	99.84	0.1554	1.002
M7-294-3	1.0306	1.0282	99.77	0.2329	1.002
M7-294-4	1.0083	1.0069	99.86	0.1388	1.001
M7-294-5	1.0444	1.0410	99.67	0.3255	1.003
		average	99.79	0.21	1.002
		confidence limit	0.09	0.09	0.001
		SD	0.07	0.07	0.001
		CV %	0.07	34.68	0.074

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M7-295-1	1.0773	1.0740	99.69	0.3063	1.003
M7-295-2	1.0099	1.0068	99.69	0.3070	1.003
M7-295-3	1.0297	1.0226	99.31	0.6895	1.007
M7-295-4	1.0191	1.0160	99.70	0.3042	1.003
M7-295-5	1.0461	1.0412	99.53	0.4684	1.005
		average	99.58	0.42	1.004
		confidence limit	0.21	0.21	0.002
		SD	0.17	0.17	0.002
		CV %	0.17	40.67	0.170

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M7-296-1	0.9904	0.9850	99.45	0.5452	1.005
M7-296-2	0.9765	0.9744	99.78	0.2151	1.002
M7-296-3	0.9601	0.9569	99.67	0.3333	1.003
M7-296-4	0.9864	0.9822	99.57	0.4258	1.004
M7-296-5	0.9496	0.9480	99.83	0.1685	1.002
M7-296-6	1.0012	0.9970	99.58	0.4195	1.004
M7-296-7	0.9990	0.9968	99.78	0.2202	1.002
M7-296-8	1.0090	1.0069	99.79	0.2081	1.002
M7-296-9	1.0065	1.0046	99.81	0.1888	1.002
M7-296-10	1.0146	1.0125	99.79	0.2070	1.002
M7-296-11	0.9693	0.9665	99.71	0.2889	1.003
M7-296-12	0.9188	0.9182	99.93	0.0653	1.001
M7-296-13	0.9690	0.9674	99.83	0.1651	1.002
M7-296-14	0.9314	0.9270	99.53	0.4724	1.005
M7-296-15	0.9046	0.9045	99.99	0.0111	1.000
M7-296-16	1.0328	1.0290	99.63	0.3679	1.004
M7-296-17	0.9056	0.9026	99.67	0.3313	1.003
M7-296-18	0.9754	0.9739	99.85	0.1538	1.002
M7-296-19	0.9619	0.9585	99.65	0.3535	1.004
M7-296-20	0.9549	0.9547	99.98	0.0209	1.000
		average	**99.74**	**0.26**	**1.003**
		confidence limit	**0.07**	**0.07**	**0.001**
		SD	**0.15**	**0.15**	**0.001**
		CV %	**0.15**	**56.98**	**0.147**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M7-297-1	1.0625	1.0568	99.46	0.5365	1.005
M7-297-2	1.0409	1.0371	99.63	0.3651	1.004
M7-297-3	1.0152	1.0107	99.56	0.4433	1.004
M7-297-4	1.0130	1.0076	99.47	0.5331	1.005
M7-297-5	1.0284	1.0240	99.57	0.4278	1.004
		average	**99.54**	**0.46**	**1.005**
		confidence limit	**0.09**	**0.09**	**0.001**
		SD	**0.07**	**0.07**	**0.001**
		CV %	**0.07**	**15.90**	**0.074**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M7-299-1	1.0581	1.0526	99.48	0.5198	1.005
M7-299-2	1.0601	1.0531	99.34	0.6603	1.007
M7-299-3	1.0099	1.0053	99.54	0.4555	1.005
M7-299-4	1.0223	1.0161	99.39	0.6065	1.006
M7-299-5	1.0210	1.0164	99.55	0.4505	1.005
		average	**99.46**	**0.54**	**1.005**
		confidence limit	**0.12**	**0.12**	**0.001**
		SD	**0.09**	**0.09**	**0.001**
		CV %	**0.09**	**17.23**	**0.093**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M7-300-1	1.0993	1.0966	99.75	0.2456	1.002
M7-300-2	1.0695	1.0660	99.67	0.3273	1.003
M7-300-3	1.0161	1.0138	99.77	0.2264	1.002
M7-300-4	1.0396	1.0363	99.68	0.3174	1.003
M7-300-5	1.0370	1.0365	99.95	0.0482	1.000
		average	**99.77**	**0.23**	**1.002**
		confidence limit	**0.14**	**0.14**	**0.001**
		SD	**0.11**	**0.11**	**0.001**
		CV %	**0.11**	**48.16**	**0.112**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M7-301-1	1.0177	1.0186	100.09	-0.0884	0.999
M7-301-2	0.9683	0.9702	100.20	-0.1962	0.998
M7-301-3	1.0493	1.0526	100.31	-0.3145	0.997
M7-301-4	0.9953	0.9982	100.29	-0.2914	0.997
M7-301-5	1.0796	1.0838	100.39	-0.3890	0.996
		average	**100.26**	**-0.26**	**0.997**
		confidence limit	**0.14**	**0.14**	**0.001**
		SD	**0.12**	**0.12**	**0.001**
		CV %	**0.12**	**-45.41**	**0.116**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M7-317-1	0.9042	0.8973	99.24	0.7631	1.008
M7-317-2	0.9917	0.9843	99.25	0.7462	1.008
M7-317-3	1.0011	0.9937	99.26	0.7392	1.007
M7-317-4	1.1447	1.1377	99.39	0.6115	1.006
M7-317-5	1.0345	1.0251	99.09	0.9087	1.009
		average	**99.25**	**0.75**	**1.008**
		confidence limit	**0.13**	**0.13**	**0.001**
		SD	**0.11**	**0.11**	**0.001**
		CV %	**0.11**	**14.01**	**0.106**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M8-296-1	0.9896	0.9616	97.17	2.8294	1.029
M8-296-2	1.0188	0.9868	96.86	3.1410	1.032
M8-296-3	0.9838	0.9531	96.88	3.1206	1.032
M8-296-4	1.0228	0.9917	96.96	3.0407	1.031
M8-296-5	1.0464	1.0173	97.22	2.7810	1.029
M8-296-6	1.0000	0.9698	96.98	3.0200	1.031
M8-296-7	1.0099	0.9784	96.88	3.1191	1.032
M8-296-8	0.9891	0.9568	96.73	3.2656	1.034
M8-296-9	1.0208	0.9880	96.79	3.2132	1.033
M8-296-10	1.0293	0.9979	96.95	3.0506	1.031
M8-296-11	1.0278	0.9981	97.11	2.8897	1.030
M8-296-12	0.9996	0.9678	96.82	3.1813	1.033
M8-296-13	1.0251	0.9951	97.07	2.9265	1.030
M8-296-14	1.0763	1.0446	97.05	2.9453	1.030
M8-296-15	1.0382	1.0067	96.97	3.0341	1.031
M8-296-16	1.0004	0.9708	97.04	2.9588	1.030
M8-296-17	1.0145	0.9853	97.12	2.8783	1.030
M8-296-18	1.0419	1.0110	97.03	2.9657	1.031
M8-296-19	1.0559	1.0257	97.14	2.8601	1.029
M8-296-20	0.9947	0.9646	96.97	3.0260	1.031
		average	96.99	3.01	1.031
		confidence limit	0.06	0.06	0.001
		SD	0.13	0.13	0.001
		CV %	0.14	4.41	0.137

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M9-296-1	1.0610	1.0516	99.11	0.8860	1.009
M9-296-2	0.9701	0.9619	99.15	0.8453	1.009
M9-296-3	0.9781	0.9692	99.09	0.9099	1.009
M9-296-4	1.0075	0.9971	98.97	1.0323	1.010
M9-296-5	1.0040	0.9942	99.02	0.9761	1.010
M9-296-6	1.0424	1.0321	99.01	0.9881	1.010
M9-296-7	0.9755	0.9651	98.93	1.0661	1.011
M9-296-8	0.9950	0.9860	99.10	0.9045	1.009
M9-296-9	1.0094	0.9996	99.03	0.9709	1.010
M9-296-10	1.0037	0.9928	98.91	1.0860	1.011
M9-296-11	1.0608	1.0502	99.00	0.9992	1.010
M9-296-12	1.0085	0.9898	98.15	1.8542	1.019
M9-296-13	0.9937	0.9917	99.80	0.2013	1.002
M9-296-14	1.0456	1.0359	99.07	0.9277	1.009
M9-296-15	1.0735	1.0544	98.22	1.7792	1.018
M9-296-16	1.0051	0.9908	98.58	1.4227	1.014
M9-296-17	1.0002	0.9887	98.85	1.1498	1.012
M9-296-18	1.0032	0.9921	98.89	1.1065	1.011
M9-296-19	1.0787	1.0671	98.92	1.0754	1.011
M9-296-20	0.9792	0.9705	99.11	0.8885	1.009
		average	98.95	1.05	1.011
		confidence limit	0.16	0.16	0.002
		SD	0.34	0.34	0.003
		CV %	0.35	32.46	0.346

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M10-296-1	1.0702	1.0624	99.27	0.7288	1.007
M10-296-2	1.0425	1.0373	99.50	0.4988	1.005
M10-296-3	1.0532	1.0469	99.40	0.5982	1.006
M10-296-4	1.0517	1.0456	99.42	0.5800	1.006
M10-296-5	1.0596	1.0529	99.37	0.6323	1.006
M10-296-6	1.0092	1.0007	99.16	0.8423	1.008
M10-296-7	1.0574	1.0506	99.36	0.6431	1.006
M10-296-8	1.0923	1.0847	99.30	0.6958	1.007
M10-296-9	1.0346	1.0271	99.28	0.7249	1.007
M10-296-10	1.0210	1.0127	99.19	0.8129	1.008
M10-296-11	1.0269	1.0169	99.03	0.9738	1.010
M10-296-12	1.0118	1.0011	98.94	1.0575	1.011
M10-296-13	1.0232	1.0137	99.07	0.9285	1.009
M10-296-14	1.0699	1.0588	98.96	1.0375	1.010
M10-296-15	1.0797	1.0694	99.05	0.9540	1.010
M10-296-16	1.0834	1.0721	98.96	1.0430	1.011
M10-296-17	1.0017	0.9911	98.94	1.0582	1.011
M10-296-18	1.0153	1.0065	99.13	0.8667	1.009
M10-296-19	1.0848	1.0744	99.04	0.9587	1.010
M10-296-20	1.0215	1.0091	98.79	1.2139	1.012
		average	**99.16**	**0.84**	**1.008**
		confidence limit	**0.09**	**0.09**	**0.001**
		SD	**0.20**	**0.20**	**0.002**
		CV %	**0.20**	**23.42**	**0.199**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M11-296-1	0.9862	0.9842	99.80	0.2028	1.002
M11-296-2	0.9912	0.9880	99.68	0.3228	1.003
M11-296-3	0.9809	0.9790	99.81	0.1937	1.002
M11-296-4	0.9933	0.9903	99.70	0.3020	1.003
M11-296-5	1.0052	1.0006	99.54	0.4576	1.005
M11-296-6	1.0069	1.0028	99.59	0.4072	1.004
M11-296-7	0.9897	0.9839	99.41	0.5860	1.006
M11-296-8	1.0425	1.0363	99.41	0.5947	1.006
M11-296-9	0.9776	0.9744	99.67	0.3273	1.003
M11-296-10	1.0012	0.9960	99.48	0.5194	1.005
M11-296-11	0.9911	0.9845	99.33	0.6659	1.007
M11-296-12	0.9086	0.9020	99.27	0.7264	1.007
M11-296-13	1.0819	1.0778	99.62	0.3790	1.004
M11-296-14	1.0196	1.0173	99.77	0.2256	1.002
M11-296-15	1.1199	1.1181	99.84	0.1607	1.002
M11-296-16	0.9941	0.9905	99.64	0.3621	1.004
M11-296-17	1.1173	1.1117	99.50	0.5012	1.005
M11-296-18	1.0601	1.0571	99.72	0.2830	1.003
M11-296-19	0.9859	0.9815	99.55	0.4463	1.004
M11-296-20	1.0065	1.0003	99.38	0.6160	1.006
		average	**99.59**	**0.41**	**1.004**
		confidence limit	**0.08**	**0.08**	**0.001**
		SD	**0.17**	**0.17**	**0.002**
		CV %	**0.17**	**40.27**	**0.167**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M13-296-1	1.0077	0.9875	98.00	2.00	1.020
M13-296-2	1.0247	1.0052	98.10	1.90	1.019
M13-296-3	0.9805	0.9639	98.31	1.69	1.017
M13-296-4	0.9975	0.9796	98.21	1.79	1.018
M13-296-5	1.0106	0.9922	98.18	1.82	1.019
M13-296-6	0.9677	0.9488	98.05	1.95	1.020
M13-296-7	0.9322	0.9135	97.99	2.01	1.020
M13-296-8	1.0048	0.9844	97.97	2.03	1.021
M13-296-9	0.9818	0.9631	98.10	1.90	1.019
M13-296-10	1.0243	1.0052	98.14	1.86	1.019
M13-296-11	0.9500	0.9314	98.04	1.96	1.020
M13-296-12	1.0096	0.9885	97.91	2.09	1.021
M13-296-13	1.0090	0.9885	97.97	2.03	1.021
M13-296-14	0.9690	0.9507	98.11	1.89	1.019
M13-296-15	0.9662	0.9477	98.09	1.91	1.020
M13-296-16	0.9894	0.9715	98.19	1.81	1.018
M13-296-17	1.0437	1.0241	98.12	1.88	1.019
M13-296-18	1.0148	0.9953	98.08	1.92	1.020
M13-296-19	1.0066	0.9879	98.14	1.86	1.019
M13-296-20	1.0058	0.9864	98.07	1.93	1.020
		average	**98.09**	**1.91**	**1.020**
		confidence limit	**0.04**	**0.04**	**0.000**
		SD	**0.09**	**0.09**	**0.001**
		CV %	**0.10**	**4.93**	**0.096**

JRC code	sample mass g	residue mass g	PTT %	soluble %
M14-301-1	0.9715	-0.0004	-0.04	100.04
M14-301-2	0.9998	0.0018	0.18	99.82
M14-301-3	1.0101	-0.0004	-0.04	100.04
M14-301-4	0.9647	0.0030	0.31	99.69
M14-301-5	1.0000	-0.0002	-0.02	100.02
M14-301-6	0.9752	-0.0011	-0.11	100.11
M14-301-7	0.9334	-0.0009	-0.10	100.10
M14-301-8	0.9036	0.0005	0.06	99.94
M14-301-9	0.9393	0.0029	0.31	99.69
M14-301-10	0.8806	-0.0009	-0.10	100.10
		average	**0.04**	**99.96**
		confidence limit	**0.12**	**0.12**
		SD	**0.16**	**0.16**
		CV %	**371.78**	**0.16**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M15-296-1	0.9979	0.9470	94.90	5.10	1.054
M15-296-2	0.9762	0.9275	95.01	4.99	1.053
M15-296-3	0.8839	0.8463	95.75	4.25	1.044
M15-296-4	0.9349	0.8937	95.59	4.41	1.046
M15-296-5	0.7597	0.7157	94.21	5.79	1.061
M15-296-6	1.0362	0.9868	95.23	4.77	1.050
M15-296-7	0.7963	0.7645	96.01	3.99	1.042
M15-296-8	0.9758	0.9196	94.24	5.76	1.061
M15-296-9	0.9604	0.9033	94.05	5.95	1.063
M15-296-10	0.9650	0.9230	95.65	4.35	1.046
		average	**95.06**	**4.94**	**1.052**
		confidence limit	**0.50**	**0.50**	**0.006**
		SD	**0.70**	**0.70**	**0.008**
		CV %	**0.74**	**14.26**	**0.742**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M16-293-1	0.9803	0.9515	97.06	2.94	1.030
M16-293-2	0.9422	0.9156	97.18	2.82	1.029
M16-293-3	0.9860	0.9576	97.12	2.88	1.030
M16-293-4	0.8600	0.8353	97.13	2.87	1.030
M16-293-5	0.7114	0.6904	97.05	2.95	1.030
M16-293-6	1.0259	0.9966	97.14	2.86	1.029
M16-293-7	0.9832	0.9555	97.18	2.82	1.029
M16-293-8	1.0644	1.0340	97.14	2.86	1.029
M16-293-9	1.0877	1.0566	97.14	2.86	1.029
M16-293-10	0.9908	0.9616	97.05	2.95	1.030
		average	**97.12**	**2.88**	**1.030**
		confidence limit	**0.04**	**0.04**	**0.000**
		SD	**0.05**	**0.05**	**0.001**
		CV %	**0.05**	**1.71**	**0.051**

JRC code	sample mass g	residue mass g	PTT %	soluble %	d
M16-296-1	0.9916	0.9642	97.24	2.76	1.028
M16-296-2	1.0227	0.9961	97.40	2.60	1.027
M16-296-3	0.9971	0.9710	97.38	2.62	1.027
M16-296-4	0.9831	0.9572	97.37	2.63	1.027
M16-296-5	1.0092	0.9826	97.36	2.64	1.027
M16-296-6	1.0077	0.9813	97.38	2.62	1.027
M16-296-7	0.9963	0.9698	97.34	2.66	1.027
M16-296-8	1.0195	0.9927	97.37	2.63	1.027
M16-296-9	1.0489	1.0210	97.34	2.66	1.027
M16-296-10	0.9984	0.9719	97.35	2.65	1.027
M16-296-11	1.0152	0.9864	97.16	2.84	1.029
M16-296-12	1.0384	1.0072	97.00	3.00	1.031
M16-296-13	1.0487	1.0188	97.15	2.85	1.029
M16-296-14	1.0020	0.9746	97.27	2.73	1.028
M16-296-15	0.9932	0.9656	97.22	2.78	1.029
M16-296-16	0.9555	0.9283	97.15	2.85	1.029
M16-296-17	0.9713	0.9434	97.13	2.87	1.030
M16-296-18	1.0131	0.9875	97.47	2.53	1.026
M16-296-19	1.0355	1.0044	97.00	3.00	1.031
M16-296-20	0.9719	0.9435	97.08	2.92	1.030
		average	**97.26**	**2.74**	**1.028**
		confidence limit	**0.07**	**0.07**	**0.001**
		SD	**0.14**	**0.14**	**0.001**
		CV %	**0.14**	**5.11**	**0.144**

Solubility properties - PTT - *d* correction factors

DuPont's results

Code	sample mass g	residue mass g	PTT %	soluble %	d
M1-1	1.4876	1.4837	99.74	0.2622	1.003
M1-2	1.5049	1.5039	99.93	0.0664	1.001
M1-3	1.4683	1.4650	99.78	0.2247	1.002
M1-4	1.4771	1.4719	99.65	0.3520	1.004
M1-5	1.4722	1.4698	99.84	0.1630	1.002
M1-6	1.4703	1.4643	99.59	0.4081	1.004
		average	**99.75**	**0.25**	**1.00**
		confidence limit	**0.13**	**0.13**	**0.00**
		SD	**0.12**	**0.12**	**0.00**
		CV %	**0.12**	**50.54**	**0.12**

Code	sample mass g	residue mass g	PTT %	soluble %	d
M2-1	1.3338	1.3276	99.54	0.4648	1.005
M2-2	1.3226	1.3149	99.42	0.5822	1.006
M2-3	1.2948	1.2878	99.46	0.5406	1.005
M2-4	1.3871	1.3890	100.14	-0.1370	0.999
M2-5	1.4463	1.4459	99.97	0.0270	1.000
M2-6	1.4020	1.4029	100.06	-0.0642	0.999
		average	**99.76**	**0.24**	**1.002**
		confidence limit	**0.34**	**0.34**	**0.003**
		SD	**0.33**	**0.33**	**0.003**
		CV %	**0.33**	**139.24**	**0.329**

Code	sample mass g	residue mass g	PTT %	soluble %	d
M3-1	1.4675	1.4572	99.30	0.70	1.007
M3-2	1.4109	1.3964	98.97	1.03	1.010
M3-3	1.3841	1.3744	99.30	0.70	1.007
M3-4	1.4898	1.4814	99.44	0.56	1.006
M3-5	1.2772	1.2623	98.83	1.17	1.012
M3-6	1.3808	1.3621	98.65	1.35	1.014
		average	**99.08**	**0.92**	**1.009**
		confidence limit	**0.33**	**0.33**	**0.003**
		SD	**0.31**	**0.31**	**0.003**
		CV %	**0.31**	**33.83**	**0.314**

Code	sample mass g	residue mass g	PTT %	soluble %	d
M4-1	1.7006	1.6843	99.04	0.9585	1.010
M4-2	1.8414	1.8242	99.07	0.9341	1.009
M4-3	1.7160	1.6994	99.03	0.9674	1.010
M4-4	1.5961	1.5799	98.99	1.0150	1.010
M4-5	1.3515	1.3380	99.00	0.9989	1.010
M4-6	1.5302	1.5121	98.82	1.1829	1.012
		average	**98.99**	**1.01**	**1.010**
		confidence limit	**0.09**	**0.09**	**0.001**
		SD	**0.09**	**0.09**	**0.001**
		CV %	**0.09**	**8.89**	**0.091**

Code	sample mass g	residue mass g	PTT %	soluble %	d
M5-1	1.6035	1.5701	97.92	2.0829	1.021
M5-2	1.5599	1.5321	98.22	1.7822	1.018
M5-3	1.5723	1.5442	98.21	1.7872	1.018
M5-4	1.5064	1.4780	98.11	1.8853	1.019
M5-5	1.5434	1.5251	98.81	1.1857	1.012
M5-6	1.5055	1.4759	98.03	1.9661	1.020
		average	**98.22**	**1.78**	**1.018**
		confidence limit	**0.33**	**0.33**	**0.003**
		SD	**0.31**	**0.31**	**0.003**
		CV %	**0.32**	**17.58**	**0.318**

Code	sample mass g	residue mass g	PTT %	soluble %	d
M6-1	1.4833	1.4589	98.36	1.6450	1.017
M6-2	1.4215	1.3953	98.16	1.8431	1.019
M6-3	1.4386	1.4081	97.88	2.1201	1.022
M6-4	1.4791	1.4567	98.49	1.5144	1.015
M6-5	1.4279	1.4032	98.27	1.7298	1.018
M6-6	1.3374	1.3139	98.24	1.7571	1.018
		average	**98.23**	**1.77**	**1.018**
		confidence limit	**0.22**	**0.22**	**0.002**
		SD	**0.21**	**0.21**	**0.002**
		CV %	**0.21**	**11.61**	**0.209**

Code	sample mass g	residue mass g	PTT %	soluble %	d
M7-1	1.4569	1.4519	99.66	0.3432	1.003
M7-2	1.4574	1.4468	99.27	0.7273	1.007
M7-3	1.4637	1.4499	99.06	0.9428	1.010
M7-4	1.3703	1.3602	99.26	0.7371	1.007
M7-5	1.4356	1.4193	98.86	1.1354	1.011
M7-6	1.3827	1.3759	99.51	0.4918	1.005
		average	**99.27**	**0.73**	**1.007**
		confidence limit	**0.30**	**0.30**	**0.003**
		SD	**0.29**	**0.29**	**0.003**
		CV %	**0.29**	**39.54**	**0.291**

Code	sample mass g	residue mass g	PTT %	soluble %	d
M8-1	1.3541	1.3130	96.96	3.0352	1.031
M8-2	1.5191	1.4746	97.07	2.9294	1.030
M8-3	1.3468	1.3072	97.06	2.9403	1.030
M8-4	1.3224	1.2825	96.98	3.0172	1.031
M8-5	1.4400	1.4005	97.26	2.7431	1.028
M8-6	1.3304	1.2904	96.99	3.0066	1.031
		average	**97.05**	**2.95**	**1.030**
		confidence limit	**0.11**	**0.11**	**0.001**
		SD	**0.11**	**0.11**	**0.001**
		CV %	**0.11**	**3.66**	**0.111**

Code	sample mass g	residue mass g	PTT %	soluble %	d
M9-1	1.4133	1.3996	99.03	0.9694	1.010
M9-2	1.3449	1.3305	98.93	1.0707	1.011
M9-3	1.2957	1.2867	99.31	0.6946	1.007
M9-4	1.4336	1.4264	99.50	0.5022	1.005
M9-5	1.3983	1.3821	98.84	1.1585	1.012
		average	99.12	0.88	1.009
		confidence limit	0.34	0.34	0.00
		SD	0.27	0.27	0.003
		CV %	0.28	31.10	0.276

Code	sample mass g	residue mass g	PTT %	soluble %	d
M10-1	1.8560	1.8393	99.10	0.8998	1.009
M10-2	1.5177	1.5141	99.76	0.2372	1.002
M10-3	1.5718	1.5709	99.94	0.0573	1.001
M10-4	1.6429	1.6278	99.08	0.9191	1.009
M10-5	1.6930	1.6666	98.44	1.5594	1.016
M10-6	1.5859	1.5620	98.49	1.5070	1.015
		average	99.14	0.86	1.009
		confidence limit	0.65	0.65	0.007
		SD	0.62	0.62	0.006
		CV %	0.63	72.25	0.629

Code	sample mass g	residue mass g	PTT %	soluble %	d
M11-1	1.9432	1.9044	98.00	1.9967	1.020
M11-2	1.8356	1.8083	98.51	1.4873	1.015
M11-3	1.6970	1.6464	97.02	2.9817	1.031
M11-4	1.8284	1.8025	98.58	1.4165	1.014
M11-5	1.9192	1.8830	98.11	1.8862	1.019
M11-6	1.8131	1.7822	98.30	1.7043	1.017
		average	98.09	1.91	1.020
		confidence limit	0.60	0.60	0.006
		SD	0.57	0.57	0.006
		CV %	0.58	29.78	0.584

Code	sample mass g	residue mass g	PTT %	soluble %	d
M13-1	1.3952	1.3577	97.31	2.69	1.028
M13-2	1.4591	1.4151	96.98	3.02	1.031
M13-3	1.3159	1.2787	97.17	2.83	1.029
M13-4	1.6909	1.6420	97.11	2.89	1.030
M13-5	1.6572	1.6068	96.96	3.04	1.031
M13-6	1.3738	1.3323	96.98	3.02	1.031
		average	97.09	2.91	1.030
		confidence limit	0.15	0.15	0.002
		SD	0.14	0.14	0.001
		CV %	0.14	4.78	0.143

Code	sample mass g	residue mass g	PTT %	soluble %	d
M15-1	1.4175	1.3689	96.57	3.43	1.036
M15-2	1.5295	1.4814	96.86	3.14	1.032
M15-3	1.3517	1.3028	96.38	3.62	1.038
M15-4	1.4342	1.3884	96.81	3.19	1.033
M15-5	1.4671	1.4122	96.26	3.74	1.039
M15-6	1.5287	1.4688	96.08	3.92	1.041
		average	**96.49**	**3.51**	**1.036**
		confidence limit	**0.32**	**0.32**	**0.003**
		SD	**0.31**	**0.31**	**0.003**
		CV %	**0.32**	**8.76**	**0.319**

Code	sample mass g	residue mass g	PTT %	soluble %	d
M16-1	1.8623	1.7949	96.38	3.62	1.038
M16-2	1.5816	1.5347	97.03	2.97	1.031
M16-3	1.5558	1.5084	96.95	3.05	1.031
M16-4	1.8256	1.7717	97.05	2.95	1.030
M16-5	1.6225	1.5750	97.07	2.93	1.030
M16-6	1.7892	1.7357	97.01	2.99	1.031
		average	**96.92**	**3.08**	**1.032**
		confidence limit	**0.28**	**0.28**	**0.003**
		SD	**0.27**	**0.27**	**0.003**
		CV %	**0.27**	**8.61**	**0.275**

Solubility properties - elastane

JRC code	sample mass g	residue mass g	elastane %	soluble %
M3-095-1	0.9728	0.0041	0.42	99.58
M3-095-2	1.1558	0.0047	0.41	99.59
M3-095-3	1.1154	0.0047	0.42	99.58
M3-095-4	1.1241	0.0056	0.50	99.50
M3-095-5	1.1459	0.0054	0.47	99.53
M3-095-6	0.9356	0.0033	0.35	99.65
M3-095-7	1.0531	0.0052	0.49	99.51
M3-095-8	1.0679	0.0021	0.20	99.80
M3-095-9	1.0521	0.0064	0.61	99.39
M3-095-10	1.0894	0.0048	0.44	99.56
		average	0.43	99.57
		confidence limit	0.08	0.08
		SD	0.11	0.11
		CV %	24.87	0.11

JRC code	sample mass g	residue mass g	elastane %	soluble %	d
M4-095-1	0.9927	0.9794	98.66	1.34	1.014
M4-095-2	1.0161	0.9990	98.32	1.68	1.017
M4-095-3	0.9840	0.9673	98.30	1.70	1.017
M4-095-4	0.9385	0.9260	98.67	1.33	1.013
M4-095-5	0.9824	0.9668	98.41	1.59	1.016
M4-095-6	0.9508	0.9373	98.58	1.42	1.014
M4-095-7	0.9533	0.9363	98.22	1.78	1.018
M4-095-8	0.9896	0.9692	97.94	2.06	1.021
M4-095-9	0.9174	0.9013	98.25	1.75	1.018
M4-095-10	0.9398	0.9242	98.34	1.66	1.017
M4-095-11	0.9987	0.9822	98.35	1.65	1.017
M4-095-12	0.9745	0.9539	97.89	2.11	1.020
M4-095-13	0.9685	0.9546	98.56	1.44	1.014
M4-095-14	0.9874	0.9723	98.47	1.53	1.015
M4-095-15	0.9985	0.9810	98.25	1.75	1.018
M4-095-16	0.9998	0.9871	98.73	1.27	1.013
M4-095-17	0.9365	0.9240	98.67	1.33	1.013
M4-095-18	0.9756	0.9548	97.87	2.13	1.022
M4-095-19	0.9999	0.9867	98.68	1.32	1.013
M4-095-20	0.9877	0.9760	98.82	1.18	1.012
		average	98.40	1.60	1.016
		uncertainty	0.13	0.13	0.001
		SD	0.28	0.28	0.003
		CV %	0.29	17.52	0.282

JRC code	sample mass g	residue mass g	elastane %	soluble %
M7-095-1	0.9805	0.0000	0.00	100.00
M7-095-2	1.1750	-0.0001	-0.01	100.01
M7-095-3	0.9840	0.0010	0.10	99.90
M7-095-4	0.9765	-0.0005	-0.05	100.05
M7-095-5	1.0103	0.0017	0.17	99.83
M7-095-6	1.1478	0.0015	0.13	99.87
M7-095-7	1.0059	0.0002	0.02	99.98
M7-095-8	1.0024	0.0002	0.02	99.98
M7-095-9	1.0482	-0.0002	-0.02	100.02
M7-095-10	1.0232	0.0008	0.08	99.92
		average	0.04	99.96
		confidence limit	0.05	0.05
		SD	0.07	0.07
		CV %	163.16	0.07

JRC code	sample mass g	residue mass g	elastane %	soluble %
M8-095-1	1.0117	0.0007	0.07	99.93
M8-095-2	1.1170	-0.0002	-0.02	100.02
M8-095-3	1.1196	0.0002	0.02	99.98
M8-095-4	0.9805	-0.0004	-0.04	100.04
M8-095-5	1.0125	-0.0002	-0.02	100.02
M8-095-6	0.9974	-0.0007	-0.07	100.07
M8-095-7	1.0326	0.0002	0.02	99.98
M8-095-8	1.0022	-0.0009	-0.09	100.09
M8-095-9	1.0434	-0.0004	-0.04	100.04
M8-095-10	0.9977	-0.0009	-0.09	100.09
		average	**-0.03**	**100.03**
		confidence limit	**0.04**	**0.04**
		SD	**0.05**	**0.05**
		CV %	**-196.80**	**0.05**

JRC code	sample mass g	residue mass g	elastane %	soluble %
M14-095-1	1.0110	0.0004	0.04	99.96
M14-095-2	1.1567	0.0014	0.12	99.88
M14-095-3	1.0322	0.0006	0.06	99.94
M14-095-4	1.0847	0.0003	0.03	99.97
M14-095-5	1.0465	-0.0002	-0.02	100.02
M14-095-6	0.9581	-0.0001	-0.01	100.01
M14-095-7	1.0410	0.0005	0.05	99.95
M14-095-8	1.0152	0.0017	0.17	99.83
M14-095-9	1.0273	0.0006	0.06	99.94
M14-095-10	1.0061	0.0011	0.11	99.89
		average	**0.06**	**99.94**
		confidence limit	**0.04**	**0.04**
		SD	**0.06**	**0.06**
		CV %	**97.87**	**0.06**

Chemical quantification

68 % modal - 28 % PTT - 4 % elastane (sample 304)

Variant 4

JRC code	sample mass g	PTT+modal mass g	PTT mass g	PTT %	modal %	elastane %
M8+7-304-1	0.9323	0.8679	0.2388	24.49	70.44	5.07
M8+7-304-2	0.9877	0.9173	0.2589	25.09	69.64	5.27
M8+7-304-3	0.9650	0.8965	0.2526	25.05	69.71	5.25
M8+7-304-4	0.9577	0.8914	0.2493	24.90	70.02	5.08
M8+7-304-5	0.9555	0.8870	0.2512	25.16	69.53	5.31
M8+7-304-6	0.9020	0.8394	0.2355	24.98	69.92	5.10
M8+7-304-7	0.9154	0.8507	0.2352	24.57	70.20	5.22
M8+7-304-8	0.9896	0.9196	0.2568	24.83	69.95	5.22
M8+7-304-9	0.9422	0.8779	0.2464	25.02	70.00	4.99
M8+7-304-10	0.8959	0.8327	0.2359	25.20	69.60	5.20
			average	**24.93**	**69.90**	**5.17**
			confidence limit	**0.17**	**0.21**	**0.07**
			SD	**0.24**	**0.29**	**0.10**
			CV %	**0.95**	**0.41**	**2.03**

Variant 2

JRC code	sample mass g	PTT mass g	PTT %
M7-304-1	0.9348	0.2533	25.17
M7-304-2	0.9569	0.2636	25.61
M7-304-3	0.9734	0.2654	25.34
M7-304-4	0.9500	0.2591	25.34
M7-304-5	0.9912	0.2728	25.59
M7-304-6	1.0361	0.2869	25.74
M7-304-7	0.9944	0.2743	25.64
M7-304-8	1.0024	0.2747	25.47
M7-304-9	0.9365	0.2570	25.50
M7-304-10	0.9309	0.2518	25.13
		average	**25.45**
		confidence limit	**0.15**
		SD	**0.20**
		CV %	**0.80**

JRC code	sample mass g	PTT + modal mass g	modal %	elastane %
M8-304-1	0.9323	0.8679	69.76	5.06
M8-304-2	0.9877	0.9173	69.13	5.27
M8-304-3	0.9650	0.8965	69.42	5.24
M8-304-4	0.9577	0.8914	69.58	5.08
M8-304-5	0.9555	0.8870	69.11	5.30
M8-304-6	0.9020	0.8394	69.17	5.09
M8-304-7	0.9154	0.8507	69.15	5.21
M8-304-8	0.9896	0.9196	69.31	5.22
M8-304-9	0.9422	0.8779	69.52	4.98
M8-304-10	0.8959	0.8327	69.67	5.20
		average	**69.38**	**5.16**
		confidence limit	**0.17**	**0.08**
		SD	**0.24**	**0.11**
		CV %	**0.35**	**2.04**

80 % PTT - 20 % elastane (sample 305)

JRC code	sample mass g	residue mass g	PTT %	elastane %
M7-305-1	1.0151	0.8561	84.34	15.66
M7-305-2	1.0760	0.9050	84.11	15.89
M7-305-3	1.0979	0.9243	84.19	15.81
M7-305-4	0.9966	0.8362	83.91	16.09
M7-305-5	0.9784	0.8203	83.84	16.16
M7-305-6	0.9820	0.8237	83.88	16.12
M7-305-7	1.0726	0.9004	83.95	16.05
M7-305-8	1.0551	0.8898	84.33	15.67
M7-305-9	1.0906	0.9176	84.14	15.86
M7-305-10	1.0245	0.8642	84.35	15.65
		average	**84.10**	**15.90**
		confidence limit	**0.14**	**0.14**
		SD	**0.20**	**0.20**
		CV %	**0.24**	**1.26**

42 % PTT - 58 % polyamide (sample 306)

JRC code	sample mass g	residue mass g	PTT %	polyamide %
M4-306-1	0.8684	0.3747	42.57	57.43
M4-306-2	0.8727	0.3808	43.06	56.94
M4-306-3	0.9451	0.4118	43.00	57.00
M4-306-4	0.9023	0.3961	43.33	56.67
M4-306-5	0.9113	0.3992	43.23	56.77
M4-306-6	0.9122	0.4018	43.48	56.52
		average	**43.11**	**56.89**
		confidence limit	**0.33**	**0.33**
		SD	**0.32**	**0.32**
		CV %	**0.73**	**0.56**

76 % PTT - 17 % PET - 7 % polyamide (sample 307)

JRC code	sample mass g	residue mass g	PTT+PET %	polyamide %
M4-307-1	0.9315	0.8601	93.00	7.00
M4-307-2	0.9683	0.8933	92.91	7.09
M4-307-3	0.9287	0.8574	92.98	7.02
M4-307-4	0.9334	0.8626	93.08	6.92
M4-307-5	0.9370	0.8649	92.96	7.04
M4-307-6	0.9152	0.8447	92.96	7.04
		average	**92.98**	**7.02**
		confidence limit	**0.06**	**0.06**
		SD	**0.06**	**0.06**
		CV %	**0.06**	**0.80**

30 % PTT - 70 % cotton (sample 314=321)

JRC code	sample mass g	residue mass g	PTT %	cotton %
M7-314-1	0.9679	0.2979	29.38	70.62
M7-314-2	0.9225	0.2835	29.33	70.67
M7-314-3	0.9186	0.2812	29.21	70.79
M7-314-4	0.9838	0.3012	29.22	70.78
M7-314-5	0.8804	0.2700	29.27	70.73
M7-314-6	0.9583	0.2942	29.30	70.70
M7-314-7	0.9503	0.2932	29.45	70.55
M7-314-8	0.9633	0.2946	29.19	70.81
M7-314-9	1.0736	0.3304	29.37	70.63
M7-314-10	0.9965	0.3059	29.30	70.70
		average	29.30	70.70
		confidence limit	0.06	0.06
		SD	0.08	0.08
		CV %	0.28	0.12

76 % PTT - 24 % wool (sample 322)

JRC code	sample mass g	residue mass g	PTT %	wool %
M2-322-1	0.9681	0.7733	77.31	22.69
M2-322-2	0.9839	0.7853	77.24	22.76
M2-322-3	0.9739	0.7784	77.36	22.64
M2-322-4	0.9834	0.7860	77.36	22.64
M2-322-5	0.9960	0.7978	77.55	22.45
M2-322-6	1.0129	0.8127	77.70	22.30
M2-322-7	1.0076	0.8050	77.33	22.67
M2-322-8	0.9948	0.7954	77.40	22.60
M2-322-9	1.0410	0.8340	77.57	22.43
M2-322-10	0.9721	0.7743	77.06	22.94
		average	77.39	22.61
		confidence limit	0.13	0.13
		SD	0.18	0.18
		CV %	0.23	0.80

40 % PTT - 60 % cotton (sample 323)

JRC code	sample mass g	residue mass g	PTT %	cotton %
M7-323-1	1.0267	0.4504	42.23	57.77
M7-323-2	1.0289	0.4487	41.98	58.02
M7-323-3	1.0719	0.4710	42.31	57.69
M7-323-4	1.0280	0.4503	42.17	57.83
M7-323-5	1.0139	0.4445	42.21	57.79
M7-323-6	1.0534	0.4579	41.84	58.16
M7-323-7	1.0555	0.4632	42.25	57.75
M7-323-8	1.0341	0.4495	41.84	58.16
M7-323-9	0.9881	0.4278	41.67	58.33
M7-323-10	1.0004	0.4369	42.04	57.96
		average	42.05	57.95
		confidence limit	0.15	0.15
		SD	0.22	0.22
		CV %	0.51	0.37

58 % promodal - 37 % PTT - 5 % elastane (sample 324)

Variant 4

JRC code	sample mass g	PTT+modal mass g	PTT mass g	PTT %	modal %	elastane %
M8+7-324-1	0.9299	0.8849	0.3759	39.18	57.92	2.90
M8+7-324-2	0.9561	0.9088	0.3882	39.37	57.63	3.00
M8+7-324-3	0.9260	0.8801	0.3753	39.29	57.70	3.01
M8+7-324-4	0.9799	0.9325	0.3970	39.27	57.83	2.89
M8+7-324-5	0.9677	0.9222	0.3910	39.16	58.08	2.77
M8+7-324-6	0.9345	0.8928	0.3716	38.50	58.95	2.55
M8+7-324-7	0.9759	0.9282	0.3948	39.22	57.84	2.94
M8+7-324-8	0.9212	0.8778	0.3737	39.32	57.91	2.77
M8+7-324-9	0.9490	0.9038	0.3855	39.38	57.80	2.82
M8+7-324-10	0.9740	0.9277	0.3946	39.27	57.92	2.81
			average	**39.20**	**57.96**	**2.85**
			confidence limit	**0.18**	**0.26**	**0.10**
			SD	**0.26**	**0.37**	**0.14**
			CV %	**0.65**	**0.64**	**4.76**

Variant 2

JRC code	sample mass g	PTT mass g	PTT %
M7-324-1	10135	0.4219	39.17
M7-324-2	0.9543	0.3953	38.98
M7-324-3	0.9801	0.4088	39.26
M7-324-4	0.9766	0.4085	39.37
M7-324-5	0.9783	0.4081	39.25
M7-324-6	0.9584	0.3996	39.22
M7-324-7	0.9745	0.4053	39.14
M7-324-8	0.9862	0.4084	38.96
M7-324-9	0.9650	0.4001	39.01
M7-324-10	0.9624	0.4012	39.23
		average	**39.16**
		confidence limit	**0.10**
		SD	**0.14**
		CV %	**0.35**

JRC code	sample mass g	PTT + modal mass g	modal %	elastane %
M8-324-1	0.9299	0.8849	57.93	2.90
M8-324-2	0.9561	0.9088	58.02	3.00
M8-324-3	0.9260	0.8801	57.73	3.01
M8-324-4	0.9799	0.9325	57.74	2.89
M8-324-5	0.9677	0.9222	57.98	2.77
M8-324-6	0.9345	0.8928	58.24	2.54
M8-324-7	0.9759	0.9282	57.92	2.94
M8-324-8	0.9212	0.8778	58.26	2.78
M8-324-9	0.9490	0.9038	58.17	2.83
M8-324-10	0.9740	0.9277	57.96	2.82
		average	**57.99**	**2.85**
		confidence limit	**0.13**	**0.10**
		SD	**0.19**	**0.14**
		CV %	**0.32**	**4.88**

Manual separation

66 % PTT - 34 % PET (sample 298)

JRC code	PTT mass g	PET mass g	sample mass g	PTT %	PET %
298-1	0.5386	0.2753	0.8139	66.18	33.82
298-2	0.6698	0.3376	1.0074	66.49	33.51
298-3	0.5022	0.2537	0.7559	66.44	33.56
298-4	0.6275	0.318	0.9455	66.37	33.63
298-5	0.5545	0.2822	0.8367	66.27	33.73
298-6	0.5218	0.2659	0.7877	66.24	33.76
			average	**66.33**	**33.67**
			confidence limit	**0.13**	**0.13**
			SD	**0.12**	**0.12**
			CV %	**0.18**	**0.36**

65 % PTT - 35 % PET (sample 302)

JRC code	PTT mass g	PET mass g	sample mass g	PTT %	PET %
302-1	0.7701	0.2659	1.0360	74.33	25.67
302-2	0.7590	0.2656	1.0246	74.08	25.92
302-3	0.7519	0.2629	1.0148	74.09	25.91
302-4	0.7147	0.2500	0.9647	74.09	25.91
302-5	0.7167	0.2495	0.9662	74.18	25.82
302-6	0.7366	0.2584	0.9950	74.03	25.97
302-7	0.7351	0.2577	0.9928	74.04	25.96
302-8	0.7885	0.2743	1.0628	74.19	25.81
302-9	0.7574	0.2639	1.0213	74.16	25.84
302-10	0.7606	0.2647	1.0253	74.18	25.82
302-11	0.6424	0.2257	0.8681	74.00	26.00
302-12	0.6876	0.2389	0.9265	74.21	25.79
302-13	0.7352	0.2597	0.9949	73.90	26.10
302-14	0.6387	0.2250	0.8637	73.95	26.05
302-15	0.6082	0.2137	0.8219	74.00	26.00
302-16	0.6200	0.2163	0.8363	74.14	25.86
302-17	0.6141	0.2135	0.8276	74.20	25.80
302-18	0.6164	0.2156	0.8320	74.09	25.91
302-19	0.6418	0.2291	0.8709	73.69	26.31
302-20	0.6150	0.2130	0.8280	74.28	25.72
			average	**74.09**	**25.91**
			confidence limit	**0.07**	**0.07**
			SD	**0.14**	**0.14**
			CV %	**0.19**	**0.55**

50 % PTT - 50 % PET (sample 303)

JRC code	PTT mass g	PET mass g	sample mass g	PTT %	PET %
303-1	0.2332	0.5628	0.7960	29.30	70.70
303-2	0.2238	0.5631	0.7869	28.44	71.56
303-3	0.1857	0.4562	0.6419	28.93	71.07
303-4	0.2044	0.5244	0.7288	28.05	71.95
303-5	0.2440	0.6553	0.8993	27.13	72.87
303-6	0.2487	0.6423	0.8910	27.91	72.09
303-7	0.2420	0.6048	0.8468	28.58	71.42
303-8	0.2484	0.6059	0.8543	29.08	70.92
303-9	0.1784	0.4443	0.6227	28.65	71.35
303-10	0.2001	0.5208	0.7209	27.76	72.24
303-11	0.2103	0.5428	0.7531	27.92	72.08
303-12	0.2274	0.5625	0.7899	28.79	71.21
303-13	0.1909	0.4601	0.6510	29.32	70.68
303-14	0.2044	0.5317	0.7361	27.77	72.23
303-15	0.2195	0.5335	0.7530	29.15	70.85
303-16	0.2139	0.5050	0.7189	29.75	70.25
303-17	0.2042	0.5208	0.7250	28.17	71.83
303-18	0.2029	0.5118	0.7147	28.39	71.61
303-19	0.1597	0.3980	0.5577	28.64	71.36
303-20	0.2031	0.5130	0.7161	28.36	71.64
			average	**28.50**	**71.50**
			confidence limit	**0.30**	**0.30**
			SD	**0.65**	**0.65**
			CV %	**2.27**	**0.90**

42 % PTT - 58 % polyamide (sample 306)

JRC code	PTT mass g	polyamide mass g	sample mass g	PTT %	polyamide %
306-1	0.3981	0.5139	0.9120	42.65	57.35
306-2	0.3702	0.4811	0.8513	42.48	57.52
306-3	0.4029	0.5218	0.9247	42.57	57.43
306-4	0.3679	0.4918	0.8597	41.79	58.21
306-5	0.3664	0.4880	0.8544	41.88	58.12
306-6	0.3642	0.4809	0.8451	42.09	57.91
			average	**42.24**	**57.76**
			confidence limit	**0.39**	**0.39**
			SD	**0.37**	**0.37**
			CV %	**0.87**	**0.64**

76 % PTT - 17 % PET - 7 % polyamide (sample 307)

JRC code	PTT mass g	PET+PA mass g	sample mass g	PTT %	PET+PA %
307-1	0.7242	0.2223	0.9465	76.51	23.49
307-2	0.7299	0.2239	0.9538	76.53	23.47
307-3	0.7093	0.2201	0.9294	76.32	23.68
307-4	0.8066	0.2479	1.0545	76.49	23.51
307-5	0.7030	0.2168	0.9198	76.43	23.57
307-6	0.7170	0.2201	0.9371	76.51	23.49
			average	**76.47**	**23.53**
			confidence limit	**0.08**	**0.08**
			SD	**0.08**	**0.08**
			CV %	**0.10**	**0.34**

75 % PTT - 25 % PET (sample 309)

JRC code	PTT mass g	PET mass g	sample mass g	PTT %	PET %
309-1	0.7832	0.2460	1.0292	76.10	23.90
309-2	0.7819	0.2455	1.0274	76.10	23.90
309-3	0.7108	0.2242	0.9350	76.02	23.98
309-4	0.8155	0.2578	1.0733	75.98	24.02
309-5	0.7602	0.2387	0.9989	76.10	23.90
309-6	0.8061	0.2549	1.0610	75.98	24.02
309-7	0.7882	0.2463	1.0345	76.19	23.81
309-8	0.7752	0.2433	1.0185	76.11	23.89
309-9	0.7824	0.2466	1.0290	76.03	23.97
309-10	0.8359	0.2644	1.1003	75.97	24.03
309-11	0.6952	0.2200	0.9152	75.96	24.04
309-12	0.7010	0.2219	0.9229	75.96	24.04
309-13	0.7149	0.2255	0.9404	76.02	23.98
309-14	0.7873	0.2485	1.0358	76.01	23.99
309-15	0.7318	0.2310	0.9628	76.01	23.99
309-16	0.6708	0.2115	0.8823	76.03	23.97
309-17	0.7008	0.2207	0.9215	76.05	23.95
309-18	0.7363	0.2333	0.9696	75.94	24.06
309-19	0.7150	0.2255	0.9405	76.02	23.98
309-20	0.7091	0.2219	0.9310	76.17	23.83
			average	**76.04**	**23.96**
			confidence limit	**0.03**	**0.03**
			SD	**0.07**	**0.07**
			CV %	**0.09**	**0.29**

60 % PTT - 40 % PET (sample 311)

JRC code	PTT mass g	PET mass g	sample mass g	PTT %	PET %
311-1	0.8003	0.3364	1.1367	70.41	29.59
311-2	0.6239	0.2614	0.8853	70.47	29.53
311-3	0.7523	0.3160	1.0683	70.42	29.58
311-4	0.6359	0.2674	0.9033	70.40	29.60
311-5	0.6915	0.2940	0.9855	70.17	29.83
311-6	0.6399	0.2718	0.9117	70.19	29.81
311-7	0.7405	0.3129	1.0534	70.30	29.70
311-8	0.6658	0.2830	0.9488	70.17	29.83
311-9	0.6705	0.2820	0.9525	70.39	29.61
311-10	0.7363	0.3108	1.0471	70.32	29.68
311-11	0.7913	0.3351	1.1264	70.25	29.75
311-12	0.6597	0.2806	0.9403	70.16	29.84
311-13	0.6842	0.2847	0.9689	70.62	29.38
311-14	0.7049	0.2917	0.9966	70.73	29.27
311-15	0.7422	0.3163	1.0585	70.12	29.88
311-16	0.7097	0.3026	1.0123	70.11	29.89
311-17	0.6961	0.2953	0.9914	70.21	29.79
311-18	0.6268	0.2657	0.8925	70.23	29.77
311-19	0.6553	0.2784	0.9337	70.18	29.82
311-20	0.8256	0.3445	1.1701	70.56	29.44
			average	**70.32**	**29.68**
			confidence limit	**0.08**	**0.08**
			SD	**0.18**	**0.18**
			CV %	**0.25**	**0.59**

60 % PTT - 40 % PET (sample 312)

JRC code	PTT mass g	PET mass g	sample mass g	PTT %	PET %
312-1	0.5976	0.3512	0.9488	62.98	37.02
312-2	0.6108	0.3550	0.9658	63.24	36.76
312-3	0.6195	0.3600	0.9795	63.25	36.75
312-4	0.5443	0.3229	0.8672	62.77	37.23
312-5	0.5857	0.3460	0.9317	62.86	37.14
312-6	0.6122	0.3654	0.9776	62.62	37.38
312-7	0.6597	0.3833	1.0430	63.25	36.75
312-8	0.6304	0.3790	1.0094	62.45	37.55
312-9	0.7139	0.4192	1.1331	63.00	37.00
312-10	0.5565	0.3255	0.8820	63.10	36.90
312-11	0.6411	0.3859	1.0270	62.42	37.58
312-12	0.5559	0.3268	0.8827	62.98	37.02
312-13	0.5500	0.3230	0.8730	63.00	37.00
312-14	0.5320	0.3149	0.8469	62.82	37.18
312-15	0.7127	0.4186	1.1313	63.00	37.00
312-16	0.5728	0.3404	0.9132	62.72	37.28
312-17	0.7909	0.4688	1.2597	62.78	37.22
312-18	0.6436	0.3765	1.0201	63.09	36.91
312-19	0.6477	0.3844	1.0321	62.76	37.24
312-20	0.5608	0.3289	0.8897	63.03	36.97
			average	**62.91**	**37.09**
			confidence limit	**0.11**	**0.11**
			SD	**0.24**	**0.24**
			CV %	**0.38**	**0.65**

76 % PTT - 24 % wool (sample 322)

JRC code	PTT mass g	wool mass g	sample mass g	PTT %	wool %
322-1	0.7948	0.1985	0.9933	77.46	22.54
322-2	0.8154	0.2013	1.0167	77.66	22.34
322-3	0.7712	0.1949	0.9661	77.25	22.75
322-4	0.8329	0.2101	1.0430	77.29	22.71
322-5	0.8574	0.2202	1.0776	76.97	23.03
322-6	0.8217	0.2078	1.0295	77.24	22.76
322-7	0.8417	0.2083	1.0500	77.62	22.38
322-8	0.7656	0.1902	0.9558	77.55	22.45
322-9	0.8195	0.2040	1.0235	77.52	22.48
322-10	0.7456	0.1884	0.9340	77.26	22.74
			average	**77.38**	**22.62**
			confidence limit	**0.15**	**0.15**
			SD	**0.22**	**0.22**
			CV %	**0.28**	**0.96**

Manual separation - DuPont's results

65 % PTT - 35 % PET (sample 302)

JRC code	PTT %	PET %
302-1	74.22	25.78
302-2	74.22	25.78
302-3	74.34	25.66
average	74.26	25.74
confidence limit	0.17	0.17
SD	0.07	0.07
CV %	0.09	0.27

75 % PTT - 25 % PET (sample 309)

JRC code	PTT %	PET %
309-1	76.11	23.89
309-2	76.12	23.88
309-3	76.09	23.91
average	76.11	23.89
confidence limit	0.04	0.04
SD	0.02	0.02
CV %	0.02	0.06

60 % PTT - 40 % PET (sample 311)

JRC code	PTT %	PET %
311-1	70.23	29.77
311-2	70.30	29.70
311-3	70.49	29.51
average	70.34	29.66
confidence limit	0.33	0.33
SD	0.13	0.13
CV %	0.19	0.45

60 % PTT - 40 % PET (sample 312)

JRC code	PTT %	PET %
312-1	62.90	37.10
312-2	62.88	37.12
312-3	62.90	37.10
average	62.89	37.11
confidence limit	0.03	0.03
SD	0.01	0.01
CV %	0.02	0.03

Annex V

DSC Analysis

Quantification *via* calibration curve built with PTT (sample 296) and PET (sample 015)

Quantification based on PTT peak integration

PTT 296 %	PET 015 %	PTT enthalpy J/g
0.00	100.00	0.00
16.34	83.66	8.93
23.34	76.66	13.26
39.67	60.33	22.62
49.85	50.15	29.87
55.08	44.92	32.32
57.34	42.66	35.32
79.22	20.78	47.40
93.85	6.15	58.10
100.00	0.00	61.78

JRC code	PTT enthalpy J/g	PTT %	PET %
298-1	38.25	62.97	37.03
298-2	40.95	67.42	32.58
298-3	40.72	67.04	32.96
average	**39.97**	**65.81**	**34.19**
confidence limit	**3.72**	**6.12**	**6.12**
SD	**1.50**	**2.46**	**2.46**
CV %	**3.74**	**3.74**	**7.21**

JRC code	PTT enthalpy J/g	PTT %	PET %
302-1	46.05	75.81	24.19
302-2	46.27	76.18	23.82
302-3	46.29	76.21	23.79
average	**46.20**	**76.07**	**23.93**
confidence limit	**0.33**	**0.54**	**0.54**
SD	**0.13**	**0.22**	**0.22**
CV %	**0.29**	**0.29**	**0.92**

JRC code	PTT enthalpy J/g	PTT %	PET %
303-1	17.69	29.12	70.88
303-2	18.18	29.93	70.07
303-3	17.94	29.54	70.46
average	**17.94**	**29.53**	**70.47**
confidence limit	**0.61**	**1.00**	**1.00**
SD	**0.25**	**0.40**	**0.40**
CV %	**1.37**	**1.37**	**0.57**

JRC code	PTT enthalpy J/g	PTT %	PET %
304-1	16.21	26.69	73.31
304-2	16.27	26.79	73.21
304-3	16.36	26.93	73.07
average	**16.28**	**26.80**	**73.20**
confidence limit	**0.19**	**0.31**	**0.31**
SD	**0.08**	**0.12**	**0.12**
CV %	**0.46**	**0.46**	**0.17**

JRC code	PTT enthalpy J/g	PTT %	PET %
305-1	48.71	80.19	19.81
305-2	48.78	80.31	19.69
305-3	49.43	81.38	18.62
average	**48.97**	**80.63**	**19.37**
confidence limit	**0.99**	**1.62**	**1.62**
SD	**0.40**	**0.65**	**0.65**
CV %	**0.81**	**0.81**	**3.37**

JRC code	PTT enthalpy J/g	PTT %	PET %
308-1	62.47	102.85	-2.85
308-2	62.20	102.40	-2.40
308-3	62.34	102.63	-2.63
average	**62.34**	**102.63**	**-2.63**
confidence limit	**0.34**	**0.55**	**0.55**
SD	**0.14**	**0.22**	**0.22**
CV %	**0.22**	**0.22**	**-8.46**

JRC code	PTT enthalpy J/g	PTT %	PET %
309-1	47.54	78.27	21.73
309-2	48.01	79.04	20.96
309-3	47.78	78.66	21.34
average	**47.78**	**78.66**	**21.34**
confidence limit	**0.58**	**0.96**	**0.96**
SD	**0.24**	**0.39**	**0.39**
CV %	**0.49**	**0.49**	**1.81**

JRC code	PTT enthalpy J/g	PTT %	PET %
310-1	42.54	70.04	29.96
310-2	42.38	69.77	30.23
310-3	42.63	70.18	29.82
average	**42.52**	**70.00**	**30.00**
confidence limit	**0.31**	**0.52**	**0.52**
SD	**0.13**	**0.21**	**0.21**
CV %	**0.30**	**0.30**	**0.69**

JRC code	PTT enthalpy J/g	PTT %	PET %
311-1	45.11	74.27	25.73
311-2	44.67	73.54	26.46
311-3	44.60	73.43	26.57
average	**44.79**	**73.75**	**26.25**
confidence limit	**0.69**	**1.13**	**1.13**
SD	**0.28**	**0.46**	**0.46**
CV %	**0.62**	**0.62**	**1.73**

JRC code	PTT enthalpy J/g	PTT %	PET %
312-1	40.12	66.05	33.95
312-2	40.41	66.53	33.47
312-3	40.68	66.97	33.03
average	**40.40**	**66.52**	**33.48**
confidence limit	**0.70**	**1.15**	**1.15**
SD	**0.28**	**0.46**	**0.46**
CV %	**0.69**	**0.69**	**1.38**

JRC code	PTT enthalpy J/g	PTT %	PET %
313-1	27.90	45.93	54.07
313-2	27.68	45.57	54.43
313-3	28.19	46.41	53.59
average	**27.92**	**45.97**	**54.03**
confidence limit	**0.64**	**1.05**	**1.05**
SD	**0.26**	**0.42**	**0.42**
CV %	**0.92**	**0.92**	**0.78**

JRC code	PTT enthalpy J/g	PTT %	PET %
314-1	18.93	31.17	68.83
314-2	18.82	30.98	69.02
314-3	18.73	30.84	69.16
average	**18.83**	**31.00**	**69.00**
confidence limit	**0.25**	**0.41**	**0.41**
SD	**0.10**	**0.16**	**0.16**
CV %	**0.53**	**0.53**	**0.24**

JRC code	PTT enthalpy J/g	PTT %	PET %
315-1	36.05	59.35	40.65
315-2	35.78	58.91	41.09
315-3	35.48	58.41	41.59
average	**35.77**	**58.89**	**41.11**
confidence limit	**0.71**	**1.17**	**1.17**
SD	**0.29**	**0.47**	**0.47**
CV %	**0.80**	**0.80**	**1.14**

Enthalpy of fusion for 100% PTT samples

JRC code	PTT enthalpy J/g
293-1	64.35
293-2	64.28
293-3	64.49
average	**64.37**
confidence limit	**0.27**
SD	**0.11**
CV %	**0.17**

JRC code	PTT enthalpy J/g
294-1	64.12
294-2	64.23
294-3	64.29
average	**64.21**
confidence limit	**0.21**
SD	**0.09**
CV %	**0.13**

JRC code	PTT enthalpy J/g
295-1	64.36
295-2	64.42
295-3	64.31
average	**64.36**
confidence limit	**0.14**
SD	**0.06**
CV %	**0.09**

JRC code	PTT enthalpy J/g
296-1	61.39
296-2	61.21
296-3	61.15
296-4	62.27
296-5	61.50
296-6	62.33
296-7	62.24
296-8	62.11
average	**62.23**
confidence limit	**0.09**
SD	**0.11**
CV %	**0.18**

JRC code	PTT enthalpy J/g
297-1	61.36
297-2	61.54
297-3	61.40
average	**61.43**
confidence limit	**0.23**
SD	**0.09**
CV %	**0.15**

JRC code	PTT enthalpy J/g
299-1	61.95
299-2	61.95
299-3	61.81
average	**61.90**
confidence limit	**0.20**
SD	**0.08**
CV %	**0.13**

JRC code	PTT enthalpy J/g
300-1	59.75
300-2	59.43
300-3	59.59
average	**59.59**
confidence limit	**0.40**
SD	**0.16**
CV %	**0.27**

JRC code	PTT enthalpy J/g
317-1	61.98
317-2	61.88
317-3	61.96
average	**61.94**
confidence limit	**0.13**
SD	**0.05**
CV %	**0.09**

Enthalpy of fusion for 100% PTT samples before and after heat-treatment

JRC code	PTT enthalpy J/g
293-1	64.35
293-2	64.28
293-3	64.49
average	**64.37**
confidence limit	**0.27**
SD	**0.11**
CV %	**0.17**

JRC code	PTT enthalpy J/g
293-1 heat treated	63.45
293-2 heat treated	63.41
293-3 heat treated	63.37
average	**63.41**
confidence limit	**0.10**
SD	**0.04**
CV %	**0.06**

JRC code	PTT enthalpy J/g
296-1	61.39
296-2	61.21
296-3	61.15
296-4	62.27
296-5	61.50
296-6	62.33
296-7	62.24
296-8	62.11
average	**61.78**
confidence limit	**0.43**
SD	**0.51**
CV %	**0.82**

JRC code	PTT enthalpy J/g
296-1 heat treated	58.06
296-2 heat treated	60.82
296-3 heat treated	60.87
average	**59.92**
confidence limit	**3.99**
SD	**1.61**
CV %	**2.68**

JRC code	PTT enthalpy J/g
317-1	61.98
317-2	61.88
317-3	61.96
average	**61.94**
confidence limit	**0.13**
SD	**0.05**
CV %	**0.09**

JRC code	PTT enthalpy J/g
317-1 heat treated	61.96
317-2 heat treated	61.73
317-3 heat treated	61.86
average	**61.85**
confidence limit	**0.29**
SD	**0.12**
CV %	**0.19**

Quantification of heat treated samples *via* calibration curve built with various heat treated PTTs and PETs

Quantification based on PTT peak integration

PTT 296-HT %	PET 316-HT %	PTT enthalpy J/g
0.00	100.00	0.00
15.42	84.58	7.40
26.88	73.13	14.41
38.72	61.28	21.62
56.90	43.10	33.10
71.38	28.62	41.82
81.69	18.31	48.24
100.00	0.00	60.85

PTT 317-HT %	PET 316-HT %	PTT enthalpy J/g
0.00	100.00	0.00
16.99	83.01	9.59
27.02	72.98	15.79
45.80	54.20	26.96
64.51	35.49	38.46
72.22	27.78	42.00
82.66	17.34	50.48
100.00	0.00	61.85

PTT 293-HT %	PET 316-HT %	PTT enthalpy J/g
0.00	100.00	0.00
18.96	81.04	10.74
31.88	68.12	18.92
48.45	51.55	27.43
63.89	36.11	38.52
78.66	21.34	48.53
100.00	0.00	63.41

		calibration with PTT (296) and PET (316) heat treated		calibration with PTT (317) and PET (316) heat treated		calibration with PTT (293) and PET (316) heat treated	
JRC code	PTT enthalpy J/g	PTT %	PET %	PTT %	PET %	PTT %	PET %
298-1	40.56	68.63	31.37	67.22	32.78	65.95	34.05
298-2	39.43	66.72	33.28	65.35	34.65	64.11	35.89
298-3	40.67	68.82	31.18	67.40	32.60	66.13	33.87
average	**40.22**	**68.05**	**31.95**	**66.66**	**33.34**	**65.40**	**34.60**
confidence limit	**1.71**	**2.88**	**2.88**	**2.83**	**2.83**	**2.77**	**2.77**
SD	**0.69**	**1.16**	**1.16**	**1.14**	**1.14**	**1.12**	**1.12**
CV %	**1.71**	**1.71**	**3.64**	**1.71**	**3.41**	**1.71**	**3.23**
302-1	41.36	69.98	30.02	68.54	31.46	67.25	32.75
302-2	42.15	71.32	28.68	69.85	30.15	68.54	31.46
302-3	40.87	69.15	30.85	67.73	32.27	66.46	33.54
average	**41.46**	**70.15**	**29.85**	**68.71**	**31.29**	**67.41**	**32.59**
confidence limit	**1.60**	**2.71**	**2.71**	**2.66**	**2.66**	**2.61**	**2.61**
SD	**0.65**	**1.09**	**1.09**	**1.07**	**1.07**	**1.05**	**1.05**
CV %	**1.56**	**1.56**	**3.66**	**1.56**	**3.42**	**1.56**	**3.22**
303-1	13.26	22.44	77.56	21.98	78.02	21.56	78.44
303-2	12.34	20.88	79.12	20.45	79.55	20.07	79.93
303-3	11.51	19.48	80.52	19.08	80.92	18.72	81.28
average	**12.37**	**20.93**	**79.07**	**20.50**	**79.50**	**20.11**	**79.89**
confidence limit	**2.17**	**3.68**	**3.68**	**3.60**	**3.60**	**3.54**	**3.54**
SD	**0.88**	**1.48**	**1.48**	**1.45**	**1.45**	**1.42**	**1.42**
CV %	**7.08**	**7.08**	**1.87**	**7.08**	**1.82**	**7.08**	**1.78**
304-1	14.63	24.75	75.25	24.25	75.75	23.79	76.21
304-2	15.94	26.97	73.03	26.42	73.58	25.92	74.08
304-3	14.61	24.72	75.28	24.21	75.79	23.76	76.24
average	**15.06**	**25.48**	**74.52**	**24.96**	**75.04**	**24.49**	**75.51**
confidence limit	**1.89**	**3.20**	**3.20**	**3.14**	**3.14**	**3.08**	**3.08**
SD	**0.76**	**1.29**	**1.29**	**1.26**	**1.26**	**1.24**	**1.24**
CV %	**5.06**	**5.06**	**1.73**	**5.06**	**1.68**	**5.06**	**1.64**
305-1	45.21	76.50	23.50	74.93	25.07	73.51	26.49
305-2	47.50	80.37	19.63	78.72	21.28	77.24	22.76
305-3	45.59	77.14	22.86	75.56	24.44	74.13	25.87
average	**46.10**	**78.00**	**22.00**	**76.40**	**23.60**	**74.96**	**25.04**
confidence limit	**3.05**	**5.16**	**5.16**	**5.05**	**5.05**	**4.96**	**4.96**
SD	**1.23**	**2.08**	**2.08**	**2.03**	**2.03**	**2.00**	**2.00**
CV %	**2.66**	**2.66**	**9.44**	**2.66**	**8.62**	**2.66**	**7.97**

		calibration with PTT (296) and PET (316) heat treated		calibration with PTT (317) and PET (316) heat treated		calibration with PTT (293) and PET (316) heat treated	
JRC code	PTT enthalpy J/g	PTT %	PET %	PTT %	PET %	PTT %	PET %
308-1	61.35	103.81	-3.81	101.67	-1.67	99.76	0.24
308-2	60.15	101.78	-1.78	99.69	0.31	97.80	2.20
308-3	61.08	103.35	-3.35	101.23	-1.23	99.32	0.68
average	**60.86**	**102.98**	**-2.98**	**100.86**	**-0.86**	**98.96**	**1.04**
confidence limit	**1.56**	**2.65**	**2.65**	**2.59**	**2.59**	**2.54**	**2.54**
SD	**0.63**	**1.07**	**1.07**	**1.04**	**1.04**	**1.02**	**1.02**
CV %	**1.03**	**1.03**	**-35.77**	**1.03**	**-121.06**	**1.03**	**98.36**
309-1	44.45	75.21	24.79	73.67	26.33	72.28	27.72
309-2	43.21	73.11	26.89	71.61	28.39	70.26	29.74
309-3	41.34	69.95	30.05	68.51	31.49	67.22	32.78
average	**43.00**	**72.76**	**27.24**	**71.26**	**28.74**	**69.92**	**30.08**
confidence limit	**3.89**	**6.58**	**6.58**	**6.45**	**6.45**	**6.32**	**6.32**
SD	**1.57**	**2.65**	**2.65**	**2.59**	**2.59**	**2.55**	**2.55**
CV %	**3.64**	**3.64**	**9.72**	**3.64**	**9.03**	**3.64**	**8.46**
310-1	37.84	64.03	35.97	62.71	37.29	61.53	38.47
310-2	37.56	63.55	36.45	62.25	37.75	61.07	38.93
310-3	35.63	60.29	39.71	59.05	40.95	57.93	42.07
average	**37.01**	**62.62**	**37.38**	**61.34**	**38.66**	**60.18**	**39.82**
confidence limit	**2.99**	**5.06**	**5.06**	**4.95**	**4.95**	**4.86**	**4.86**
SD	**1.20**	**2.04**	**2.04**	**1.99**	**1.99**	**1.96**	**1.96**
CV %	**3.25**	**3.25**	**5.45**	**3.25**	**5.16**	**3.25**	**4.91**
311-1	41.60	70.39	29.61	68.94	31.06	67.64	32.36
311-2	40.65	68.78	31.22	67.37	32.63	66.10	33.90
311-3	39.88	67.48	32.52	66.09	33.91	64.85	35.15
average	**40.71**	**68.88**	**31.12**	**67.47**	**32.53**	**66.20**	**33.80**
confidence limit	**2.14**	**3.62**	**3.62**	**3.55**	**3.55**	**3.48**	**3.48**
SD	**0.86**	**1.46**	**1.46**	**1.43**	**1.43**	**1.40**	**1.40**
CV %	**2.12**	**2.12**	**4.68**	**2.12**	**4.39**	**2.12**	**4.14**
312-1	34.65	58.63	41.37	57.42	42.58	56.34	43.66
312-2	35.65	60.32	39.68	59.08	40.92	57.97	42.03
312-3	35.12	59.42	40.58	58.20	41.80	57.11	42.89
average	**35.14**	**59.46**	**40.54**	**58.24**	**41.76**	**57.14**	**42.86**
confidence limit	**1.24**	**2.10**	**2.10**	**2.06**	**2.06**	**2.02**	**2.02**
SD	**0.50**	**0.85**	**0.85**	**0.83**	**0.83**	**0.81**	**0.81**
CV %	**1.42**	**1.42**	**2.09**	**1.42**	**1.99**	**1.42**	**1.90**
313-1	25.43	43.03	56.97	42.14	57.86	41.35	58.65
313-2	26.54	44.91	55.09	43.98	56.02	43.15	56.85
313-3	27.05	45.77	54.23	44.83	55.17	43.98	56.02
average	**26.34**	**44.57**	**55.43**	**43.65**	**56.35**	**42.83**	**57.17**
confidence limit	**2.06**	**3.48**	**3.48**	**3.41**	**3.41**	**3.35**	**3.35**
SD	**0.83**	**1.40**	**1.40**	**1.37**	**1.37**	**1.35**	**1.35**
CV %	**3.14**	**3.14**	**2.53**	**3.14**	**2.44**	**3.14**	**2.36**
314-1	18.32	31.00	69.00	30.36	69.64	29.79	70.21
314-2	16.98	28.73	71.27	28.14	71.86	27.61	72.39
314-3	17.44	29.51	70.49	28.90	71.10	28.36	71.64
average	**17.58**	**29.75**	**70.25**	**29.13**	**70.87**	**28.59**	**71.41**
confidence limit	**1.69**	**2.86**	**2.86**	**2.80**	**2.80**	**2.75**	**2.75**
SD	**0.68**	**1.15**	**1.15**	**1.13**	**1.13**	**1.11**	**1.11**
CV %	**3.87**	**3.87**	**1.64**	**3.87**	**1.59**	**3.87**	**1.55**
315-1	32.56	55.09	44.91	53.96	46.04	52.94	47.06
315-2	34.25	57.95	42.05	56.76	43.24	55.69	44.31
315-3	35.07	59.34	40.66	58.12	41.88	57.02	42.98
average	**33.96**	**57.46**	**42.54**	**56.28**	**43.72**	**55.22**	**44.78**
confidence limit	**3.18**	**5.38**	**5.38**	**5.27**	**5.27**	**5.17**	**5.17**
SD	**1.28**	**2.17**	**2.17**	**2.12**	**2.12**	**2.08**	**2.08**
CV %	**3.77**	**3.77**	**5.09**	**3.77**	**4.85**	**3.77**	**4.65**

		calibration with PTT (296) and PET (316) heat treated		calibration with PTT (317) and PET (316) heat treated		calibration with PTT (293) and PET (316) heat treated	
322-1	38.99	65.97	34.03	64.62	35.38	63.40	36.60
322-2	40.23	68.07	31.93	66.67	33.33	65.41	34.59
322-3	40.88	69.17	30.83	67.75	32.25	66.47	33.53
average	**40.03**	**67.74**	**32.26**	**66.35**	**33.65**	**65.09**	**34.91**
confidence limit	**2.39**	**4.04**	**4.04**	**3.95**	**3.95**	**3.88**	**3.88**
SD	**0.96**	**1.62**	**1.62**	**1.59**	**1.59**	**1.56**	**1.56**
CV %	**2.40**	**2.40**	**5.04**	**2.40**	**4.73**	**2.40**	**4.47**
323-1	24.53	41.51	58.49	40.65	59.35	39.89	60.11
323-2	26.55	44.92	55.08	44.00	56.00	43.17	56.83
323-3	24.97	42.25	57.75	41.38	58.62	40.60	59.40
average	**25.35**	**42.89**	**57.11**	**42.01**	**57.99**	**41.22**	**58.78**
confidence limit	**2.64**	**4.46**	**4.46**	**4.37**	**4.37**	**4.29**	**4.29**
SD	**1.06**	**1.80**	**1.80**	**1.76**	**1.76**	**1.73**	**1.73**
CV %	**4.19**	**4.19**	**3.15**	**4.19**	**3.04**	**4.19**	**2.94**
324-1	21.15	35.79	64.21	35.05	64.95	34.39	65.61
324-2	22.51	38.09	61.91	37.31	62.69	36.60	63.40
324-3	23.00	38.92	61.08	38.12	61.88	37.40	62.60
average	**22.22**	**37.60**	**62.40**	**36.82**	**63.18**	**36.13**	**63.87**
confidence limit	**2.38**	**4.03**	**4.03**	**3.95**	**3.95**	**3.87**	**3.87**
SD	**0.96**	**1.62**	**1.62**	**1.59**	**1.59**	**1.56**	**1.56**
CV %	**4.31**	**4.31**	**2.60**	**4.31**	**2.51**	**4.31**	**2.44**

Enthalpy of fusion of heat treated samples 293, 296, 300 in different days

JRC code	Day 1 PTT enthalpy J/g	Day 2 PTT enthalpy J/g	Day 3 PTT enthalpy J/g	Day 4 PTT enthalpy J/g	Day 5 PTT enthalpy J/g
293-1	60.27	60.34	60.42	60.03	59.46
293-2	61.61	60.51	61.95	58.89	59.81
293-3	60.52	60.22	60.14	61.09	59.60
293-4	60.30	60.57	60.72	60.60	60.12
293-5	61.61	58.64	60.79	60.41	58.74
293-6	60.85	59.08	62.20	60.94	59.33
293-7	61.77	60.45	61.29	59.40	59.78
293-8	60.68	59.69	62.16	61.66	59.04
293-9	60.80	59.40	61.96	59.12	60.02
293-10	60.46	60.21	60.51	61.30	59.83
average	**60.89**	**59.91**	**61.21**	**60.34**	**59.57**
confidence limit	**0.41**	**0.48**	**0.57**	**0.68**	**0.31**
SD	**0.57**	**0.67**	**0.79**	**0.96**	**0.44**
CV %	**0.94**	**1.12**	**1.30**	**1.58**	**0.73**

JRC code	Day 1 PTT enthalpy J/g	Day 2 PTT enthalpy J/g	Day 3 PTT enthalpy J/g	Day 4 PTT enthalpy J/g	Day 5 PTT enthalpy J/g
296-1	57.33	56.37	57.48	56.49	56.14
296-2	57.02	56.93	58.44	56.64	56.65
296-3	56.87	56.67	57.28	56.43	55.1
296-4	56.44	57.16	64.88	55.52	55.84
296-5	56.09	54.33	64.24	56.11	55.93
296-6	56.84	57.24	56.38	52.89	55.92
296-7	56.05	57.33	56.70	56.22	56.44
296-8	56.38	56.50	56.37	56.43	55.92
296-9	56.24	57.24	57.02	55.53	56.67
296-10	56.12	57.62	56.63	57.18	56.78
average	**56.54**	**56.74**	**58.54**	**55.94**	**56.14**
confidence limit	**0.32**	**0.67**	**2.31**	**0.85**	**0.37**
SD	**0.45**	**0.93**	**3.23**	**1.18**	**0.51**
CV %	**0.79**	**1.65**	**5.52**	**2.11**	**0.91**

JRC code	Day 1 PTT enthalpy J/g	Day 2 PTT enthalpy J/g	Day 3 PTT enthalpy J/g	Day 4 PTT enthalpy J/g	Day 5 PTT enthalpy J/g
300-1	55.51	56.34	56.74	57.13	55.73
300-2	57.26	56.31	56.02	56.16	56.30
300-3	57.56	56.83	54.78	56.82	54.38
300-4	55.97	55.83	56.03	57.18	55.49
300-5	56.13	56.45	57.03	57.01	57.58
300-6	56.74	56.69	56.10	56.32	55.38
300-7	56.65	56.82	55.87	55.58	55.27
300-8	56.88	56.68	55.75	56.38	55.24
300-9	56.73	56.53	56.55	56.48	56.03
300-10	56.95	56.50	55.93	57.01	56.06
average	**56.64**	**56.50**	**56.08**	**56.61**	**55.75**
confidence limit	**0.44**	**0.21**	**0.44**	**0.37**	**0.60**
SD	**0.61**	**0.30**	**0.62**	**0.51**	**0.84**
CV %	**1.08**	**0.53**	**1.10**	**0.91**	**1.51**

Enthalpy of fusion of samples 295, 296, 300 according to different cooling rates between the first and second heating cycles

JRC code	2.5 °C/min PTT enthalpy J/g 1st cycle	2.5 °C/min PTT enthalpy J/g 2nd cycle	5 °C/min PTT enthalpy J/g 1st cycle	5 °C/min PTT enthalpy J/g 2nd cycle	20 °C/min PTT enthalpy J/g 1st cycle	20 °C/min PTT enthalpy J/g 2nd cycle	30 °C/min PTT enthalpy J/g 1st cycle	30 °C/min PTT enthalpy J/g 2nd cycle
295-1	74.12	58.03	68.90	64.42	73.59	75.37	70.67	78.96
295-2	75.45	59.02	69.40	65.98	71.58	72.87	68.58	77.98
295-3	74.95	58.13	71.32	66.24	71.98	73.65	68.12	76.52
average	**74.84**	**58.39**	**69.87**	**65.55**	**72.38**	**73.96**	**69.12**	**77.82**
confidence limit	**1.67**	**1.35**	**3.17**	**2.45**	**2.64**	**3.18**	**3.38**	**3.05**
SD	**0.67**	**0.55**	**1.28**	**0.98**	**1.06**	**1.28**	**1.36**	**1.23**
CV %	**0.90**	**0.93**	**1.83**	**1.50**	**1.47**	**1.73**	**1.97**	**1.58**

JRC code	2.5 °C/min PTT enthalpy J/g 1st cycle	2.5 °C/min PTT enthalpy J/g 2nd cycle	5 °C/min PTT enthalpy J/g 1st cycle	5 °C/min PTT enthalpy J/g 2nd cycle	20 °C/min PTT enthalpy J/g 1st cycle	20 °C/min PTT enthalpy J/g 2nd cycle	30 °C/min PTT enthalpy J/g 1st cycle	30 °C/min PTT enthalpy J/g 2nd cycle
296-1	68.62	58.69	68.10	74.49	69.07	71.50	64.89	70.11
296-2	67.34	56.43	69.89	75.17	67.94	69.69	66.55	72.58
296-3	68.07	57.15	70.20	76.60	67.02	69.11	66.32	71.98
average	**68.01**	**57.42**	**69.40**	**75.42**	**68.01**	**70.10**	**65.92**	**71.56**
confidence limit	**1.60**	**2.87**	**2.82**	**2.68**	**2.55**	**3.10**	**2.23**	**3.20**
SD	**0.64**	**1.15**	**1.13**	**1.08**	**1.03**	**1.25**	**0.90**	**1.29**
CV %	**0.94**	**2.01**	**1.63**	**1.43**	**1.51**	**1.78**	**1.36**	**1.80**

JRC code	2.5 °C/min PTT enthalpy J/g 1st cycle	2.5 °C/min PTT enthalpy J/g 2nd cycle	5 °C/min PTT enthalpy J/g 1st cycle	5 °C/min PTT enthalpy J/g 2nd cycle	20 °C/min PTT enthalpy J/g 1st cycle	20 °C/min PTT enthalpy J/g 2nd cycle	30 °C/min PTT enthalpy J/g 1st cycle	30 °C/min PTT enthalpy J/g 2nd cycle
300-1	68.71	58.70	69.16	75.87	69.43	72.23	67.79	75.02
300-2	67.56	57.36	70.11	77.24	68.78	70.63	66.17	72.98
300-3	66.08	56.15	71.07	76.94	70.65	74.14	66.05	72.92
average	**67.45**	**57.40**	**70.11**	**76.68**	**69.62**	**72.33**	**66.67**	**73.64**
confidence limit	**3.28**	**3.17**	**2.37**	**1.79**	**2.36**	**4.37**	**2.41**	**2.97**
SD	**1.32**	**1.28**	**0.96**	**0.72**	**0.95**	**1.76**	**0.97**	**1.20**
CV %	**1.95**	**2.22**	**1.36**	**0.94**	**1.36**	**2.43**	**1.46**	**1.62**

Enthalpy of fusion of samples 295, 296, 300 according to different annealing temperatures

	oven 130 ºC		oven 150 ºC		oven 200 ºC	
JRC code	PTT enthalpy J/g 1st cycle	PTT enthalpy J/g 2nd cycle	PTT enthalpy J/g 1st cycle	PTT enthalpy J/g 2nd cycle	PTT enthalpy J/g 1st cycle	PTT enthalpy J/g 2nd cycle
295-1	74.57	88.56	72.32	82.76	82.13	74.46
295-2	72.43	85.45	71.21	79.44	80.88	72.89
295-3	75.06	86.30	68.21	78.47	80.02	72.59
average	**74.02**	**86.77**	**70.58**	**80.22**	**81.01**	**73.31**
confidence limit	**3.47**	**3.99**	**5.28**	**5.59**	**2.64**	**2.49**
SD	**1.40**	**1.61**	**2.13**	**2.25**	**1.06**	**1.00**
CV %	**1.89**	**1.85**	**3.01**	**2.80**	**1.31**	**1.37**

	oven 130 ºC		oven 150 ºC		oven 200 ºC	
JRC code	PTT enthalpy J/g 1st cycle	PTT enthalpy J/g 2nd cycle	PTT enthalpy J/g 1st cycle	PTT enthalpy J/g 2nd cycle	PTT enthalpy J/g 1st cycle	PTT enthalpy J/g 2nd cycle
296-1	70.20	84.81	69.06	78.95	83.46	76.92
296-2	69.30	83.34	69.12	76.61	82.11	75.03
296-3	68.94	83.43	66.67	77.35	80.29	74.47
average	**69.48**	**83.86**	**68.28**	**77.64**	**81.95**	**75.47**
confidence limit	**1.61**	**2.05**	**3.47**	**2.97**	**3.95**	**3.19**
SD	**0.65**	**0.82**	**1.40**	**1.20**	**1.59**	**1.28**
CV %	**0.93**	**0.98**	**2.05**	**1.54**	**1.94**	**1.70**

	oven 130 ºC		oven 150 ºC		oven 200 ºC	
JRC code	PTT enthalpy J/g 1st cycle	PTT enthalpy J/g 2nd cycle	PTT enthalpy J/g 1st cycle	PTT enthalpy J/g 2nd cycle	PTT enthalpy J/g 1st cycle	PTT enthalpy J/g 2nd cycle
300-1	70.71	83.44	68.67	81.47	79.44	72.98
300-2	70.20	82.56	69.19	82.65	77.45	70.77
300-3	68.95	80.64	67.32	81.64	77.89	70.73
average	**69.95**	**82.21**	**68.39**	**81.92**	**78.26**	**71.49**
confidence limit	**2.25**	**3.56**	**2.40**	**1.58**	**2.60**	**3.20**
SD	**0.91**	**1.43**	**0.97**	**0.64**	**1.05**	**1.29**
CV %	**1.29**	**1.74**	**1.41**	**0.78**	**1.34**	**1.80**

Influence of different heat treatments and quenching times on PTT and PET enthalpies of fusion

Muffle at 300 °C for 10 min/ 1 min of quenching time

JRC code	PTT enthalpy J/g
295-1	71.37
295-2	69.5
295-3	66.83
average	69.23
confidence limit	2.83
SD	2.28
CV %	3.30

JRC code	PTT enthalpy J/g
296-1	66.64
296-2	66.00
296-3	62.96
average	65.20
confidence limit	2.44
SD	1.97
CV %	3.02

JRC code	PTT enthalpy J/g
300-1	65.44
300-2	63.65
300-3	66.87
average	65.32
confidence limit	2.00
SD	1.61
CV %	2.47

JRC code	PTT enthalpy J/g
316-1	49.36
316-2	49.45
316-3	54.66
average	51.16
confidence limit	4.83
SD	3.03
CV %	5.93

JRC code	PTT enthalpy J/g
298-1	32.75
298-2	31.05
298-3	30.88
average	31.56
confidence limit	1.28
SD	1.03
CV %	3.28

JRC code	PTT enthalpy J/g
302-1	38.62
302-2	37.96
302-3	37.63
average	38.07
confidence limit	0.63
SD	0.50
CV %	1.32

JRC code	PTT enthalpy J/g
309-1	40.24
309-2	40.05
309-3	40.78
average	40.36
confidence limit	0.47
SD	0.38
CV %	0.94

JRC code	PTT enthalpy J/g
311-1	36.17
311-2	33.95
311-3	35.66
average	35.26
confidence limit	1.85
SD	1.16
CV %	3.30

JRC code	PTT enthalpy J/g
312-1	29.82
312-2	30.85
312-3	30.52
average	30.40
confidence limit	0.65
SD	0.53
CV %	1.73

JRC code	PTT enthalpy J/g
311-1	33.10
311-2	32.50
311-3	31.27
311-4	32.49
311-5	30.30
average	31.93
confidence limit	1.40
SD	1.13
CV %	3.54

JRC code	PTT enthalpy J/g
312-1	26.19
312-2	25.65
312-3	24.81
312-4	27.92
average	26.14
confidence limit	2.09
SD	1.31
CV %	5.03

JRC code	PET enthalpy J/g
316-1	41.60
316-2	41.44
316-3	39.62
316-4	39.89
316-5	43.73
average	41.26
confidence limit	2.04
SD	1.64
CV %	3.99

JRC code	PTT enthalpy J/g
317-1	54.70
317-2	56.74
317-3	52.58
317-4	54.92
317-5	55.66
average	54.92
confidence limit	1.90
SD	1.53
CV %	2.79

Muffle at 265 °C for 3 min/ 1 min of quenching time

JRC code	PTT enthalpy J/g
312-1	31.82
312-2	29.50
312-3	29.38
312-4	31.81
312-5	31.10
average	**30.72**
confidence limit	**1.50**
SD	**1.21**
CV %	**3.93**

JRC code	PET enthalpy J/g
316-1	47.56
316-2	42.13
316-3	54.46
316-4	50.08
316-5	50.10
average	**48.87**
confidence limit	**5.60**
SD	**4.51**
CV %	**9.23**

JRC code	PTT enthalpy J/g
317-1	52.24
317-2	50.50
317-3	55.20
317-4	52.30
317-5	52.30
average	**52.51**
confidence limit	**2.10**
SD	**1.69**
CV %	**3.22**

Oven at 245 °C for 10 min/ 1 min of quenching time

JRC code	PTT enthalpy J/g
296-1	53.66
296-2	52.94
296-3	54.33
296-4	53.14
296-5	54.48
average	**53.71**
confidence limit	**0.86**
SD	**0.69**
CV %	**1.28**

JRC code	PTT enthalpy J/g
312-1	31.49
312-2	31.66
312-3	31.00
312-4	32.28
312-5	32.41
average	**31.77**
confidence limit	**0.72**
SD	**0.58**
CV %	**1.83**

Oven at 245 °C for 10 min/ 3 min of quenching time

JRC code	PTT enthalpy J/g
312-1	31.57
312-2	29.31
312-3	31.72
312-4	30.22
312-5	30.03
average	**30.57**
confidence limit	**1.29**
SD	**1.04**
CV %	**3.40**

Oven at 245 °C for 10 min/ 5 min of quenching time

JRC code	PTT enthalpy J/g
296-1	53.82
296-2	53.57
296-3	52.74
296-4	53.60
296-5	54.17
average	**53.58**
confidence limit	**0.65**
SD	**0.53**
CV %	**0.98**

JRC code	PTT enthalpy J/g
317-1	55.49
317-2	54.81
317-3	54.28
317-4	53.77
317-5	53.78
average	**54.43**
confidence limit	**0.91**
SD	**0.73**
CV %	**1.35**

JRC code	PTT enthalpy J/g
311-1	35.11
311-2	36.23
311-3	36.29
311-4	36.10
311-5	36.52
average	**36.05**
confidence limit	**0.68**
SD	**0.55**
CV %	**1.52**

JRC code	PTT enthalpy J/g
312-1	29.86
312-2	28.22
312-3	29.89
312-4	29.28
312-5	30.73
average	**29.60**
confidence limit	**1.15**
SD	**0.93**
CV %	**3.13**

Oven at 245 °C for 10 min/ 10 min of quenching time

JRC code	PTT enthalpy J/g
295-1	55.06
295-2	53.95
295-3	55.46
295-4	55.70
295-5	54.00
average	**54.83**
confidence limit	**1.01**
SD	**0.82**
CV %	**1.49**

JRC code	PTT enthalpy J/g
296-1	49.51
296-2	49.95
296-3	50.46
296-4	50.12
296-5	50.36
average	**50.08**
confidence limit	**0.47**
SD	**0.38**
CV %	**0.75**

JRC code	PTT enthalpy J/g
317-1	55.49
317-2	54.81
317-3	54.28
317-4	53.77
317-5	53.78
average	**54.43**
confidence limit	**0.91**
SD	**0.73**
CV %	**1.35**

Oven at 235 °C for 10 min/ 10 min of quenching time

JRC code	PTT enthalpy J/g
295-1	54.41
295-2	53.87
295-3	54.35
295-4	55.01
295-5	53.87
average	**54.30**
confidence limit	**0.59**
SD	**0.47**
CV %	**0.87**

JRC code	PTT enthalpy J/g
296-1	50.09
296-2	50.45
296-3	51.17
296-4	51.87
296-5	51.36
average	**50.99**
confidence limit	**0.89**
SD	**0.72**
CV %	**1.40**

JRC code	PTT enthalpy J/g
317-1	52.32
317-2	50.35
317-3	52.33
317-4	51.79
317-5	51.81
average	**51.72**
confidence limit	**1.01**
SD	**0.81**
CV %	**1.57**

JRC code	PTT enthalpy J/g
311-1	33.73
311-2	33.36
311-3	34.20
311-4	34.01
311-5	34.89
average	**34.04**
confidence limit	**0.71**
SD	**0.57**
CV %	**1.68**

JRC code	PTT enthalpy J/g
312-1	31.67
312-2	31.50
312-3	33.68
312-4	33.15
312-5	33.98
average	**32.80**
confidence limit	**1.42**
SD	**1.15**
CV %	**3.50**

JRC code	PET enthalpy J/g
316-1	54.94
316-2	54.90
316-3	54.87
316-4	55.93
316-5	55.43
average	**55.21**
confidence limit	**0.57**
SD	**0.46**
CV %	**0.84**

Quantification of heat treated samples at 300 °C for 10 min and quenched for 1 min *via* calibration curve built with various PTTs and PETs treated with the same conditions

Quantification based on PTT peak integration

PTT 300 %	PET 316 %	PTT enthalpy J/g
0.00	100.00	0.00
28.22	71.78	11.01
39.94	60.06	19.41
55.36	44.64	28.83
75.40	24.60	43.23
86.92	13.08	56.91
100.00	0.00	65.32

PTT 296 %	PET 316 %	PTT enthalpy J/g
0.00	100.00	0.00
30.77	69.23	13.86
41.18	58.82	19.34
51.62	48.38	24.10
74.21	25.79	41.15
87.37	12.63	53.52
100.00	0.00	65.20

PTT 295 %	PET 316 %	PTT enthalpy J/g
0.00	100.00	0.00
22.96	77.04	7.11
40.08	59.92	16.74
52.03	47.97	27.03
61.54	38.46	29.91
78.13	21.87	46.18
100.00	0.00	69.23

		calibration with PTT (300) and PET (316)		calibration with PTT (296) and PET (316)		calibration with PTT (295) and PET (316)	
JRC code	PTT enthalpy J/g	PTT %	PET %	PTT %	PET %	PTT %	PET %
298-1	32.75	60.58	39.42	62.67	37.33	62.91	37.09
298-2	31.05	58.26	41.74	60.31	39.69	60.77	39.23
298-3	30.88	58.03	41.97	60.07	39.93	60.55	39.45
average	**31.56**	**58.95**	**41.05**	**61.02**	**38.98**	**61.41**	**38.59**
confidence limit	**2.57**	**3.51**	**3.51**	**3.56**	**3.56**	**3.24**	**3.24**
SD	**1.03**	**1.41**	**1.41**	**1.43**	**1.43**	**1.30**	**1.30**
CV %	**3.28**	**2.39**	**3.44**	**2.35**	**3.68**	**2.12**	**3.38**
302-1	38.62	68.24	31.76	70.44	29.56	69.94	30.06
302-2	37.96	67.41	32.59	69.59	30.41	69.17	30.83
302-3	37.63	66.99	33.01	69.17	30.83	68.79	31.21
average	**38.07**	**67.55**	**32.45**	**69.73**	**30.27**	**69.30**	**30.70**
confidence limit	**1.25**	**1.59**	**1.59**	**1.61**	**1.61**	**1.45**	**1.45**
SD	**0.50**	**0.64**	**0.64**	**0.65**	**0.65**	**0.58**	**0.58**
CV %	**1.32**	**0.95**	**1.97**	**0.93**	**2.14**	**0.84**	**1.90**
309-1	40.24	70.28	29.72	72.50	27.50	71.79	28.21
309-2	40.05	70.04	29.96	72.26	27.74	71.57	28.43
309-3	40.78	70.94	29.06	73.18	26.82	72.40	27.60
average	**40.36**	**70.42**	**29.58**	**72.64**	**27.36**	**71.92**	**28.08**
confidence limit	**0.94**	**1.17**	**1.17**	**1.18**	**1.18**	**1.06**	**1.06**
SD	**0.38**	**0.47**	**0.47**	**0.48**	**0.48**	**0.43**	**0.43**
CV %	**0.94**	**0.67**	**1.59**	**0.66**	**1.74**	**0.59**	**1.52**
311-1	36.17	65.11	34.89	67.26	32.74	67.07	32.93
311-2	33.95	62.19	37.81	64.30	35.70	64.39	35.61
311-3	35.66	64.44	35.56	66.59	33.41	66.46	33.54
average	**35.26**	**63.91**	**36.09**	**66.05**	**33.95**	**65.97**	**34.03**
confidence limit	**2.89**	**3.80**	**3.80**	**3.85**	**3.85**	**3.49**	**3.49**
SD	**1.16**	**1.53**	**1.53**	**1.55**	**1.55**	**1.40**	**1.40**
CV %	**3.30**	**2.39**	**4.24**	**2.35**	**4.57**	**2.13**	**4.13**
312-1	29.82	56.55	43.45	58.57	41.43	59.19	40.81
312-2	30.85	57.98	42.02	60.03	39.97	60.51	39.49
312-3	30.52	57.53	42.47	59.57	40.43	60.09	39.91
average	**30.40**	**57.35**	**42.65**	**59.39**	**40.61**	**59.93**	**40.07**
confidence limit	**1.31**	**1.82**	**1.82**	**1.85**	**1.85**	**1.68**	**1.68**
SD	**0.53**	**0.73**	**0.73**	**0.74**	**0.74**	**0.68**	**0.68**
CV %	**1.73**	**1.28**	**1.72**	**1.25**	**1.83**	**1.13**	**1.69**

Quantification based on PTT peak integration

PTT 317 %	PET 316 %	PTT enthalpy J/g
0.00	100.00	0.00
19.34	80.66	5.78
34.32	65.68	11.78
51.25	48.75	22.67
60.83	39.17	29.21
73.78	26.22	35.51
100.00	0.00	54.92

PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
20.69	79.31	6.21
31.38	68.62	10.64
45.32	54.68	19.87
58.54	41.46	29.10
70.82	29.18	36.85

		calibration with PTT (317) and PET (316)		calibration with PTT (312) and PET (312)	
JRC code	PTT enthalpy J/g	PTT %	PET %	PTT %	PET %
311-1	33.10	69.84	30.16	65.17	34.83
311-2	32.50	68.92	31.08	64.40	35.60
311-3	31.27	66.99	33.01	62.80	37.20
311-4	32.49	68.90	31.10	64.39	35.61
311-5	30.3	65.45	34.55	61.51	38.49
average	**31.93**	**68.02**	**31.98**	**63.65**	**36.35**
confidence limit	**1.40**	**2.20**	**2.20**	**1.83**	**1.83**
SD	**1.13**	**1.77**	**1.77**	**1.47**	**1.47**
CV %	**3.54**	**2.60**	**5.54**	**2.32**	**4.06**
312-1	26.19	58.71	41.29	55.86	44.14
312-2	25.65	57.80	42.20	55.09	44.91
312-3	24.81	56.36	43.64	53.88	46.12
312-4	27.92	61.59	38.41	58.29	41.71
average	**26.14**	**58.62**	**41.38**	**55.78**	**44.22**
confidence limit	**2.09**	**3.51**	**3.51**	**2.96**	**2.96**
SD	**1.31**	**2.21**	**2.21**	**1.86**	**1.86**
CV %	**5.03**	**3.77**	**5.34**	**3.34**	**4.21**

Quantification of heat treated samples at 245 °C for 10 min and quenched for 1 min *via* calibration curve built with various PTTs and PETs treated with the same conditions

Quantification based on PTT peak integration

PTT 317 %	PET 316 %	PTT enthalpy J/g
0.00	100.00	0.00
37.70	62.30	17.80
52.98	47.02	27.09
70.68	29.32	36.28
78.61	21.39	40.09
100.00	0.00	54.43

		calibration with PTT (317) and PET (316)	
JRC code	**PTT enthalpy J/g**	**PTT %**	**PET %**
311-1	35.11	68.48	31.52
311-2	36.23	70.40	29.60
311-3	36.29	70.51	29.49
311-4	36.1	70.18	29.82
311-5	36.52	70.90	29.10
average	**36.05**	**70.09**	**29.91**
confidence limit	**0.68**	**1.16**	**1.16**
SD	**0.55**	**0.94**	**0.94**
CV %	**1.52**	**1.34**	**3.14**
312-1	29.86	59.30	40.70
312-2	28.22	56.38	43.62
312-3	29.89	59.36	40.64
312-4	29.28	58.27	41.73
312-5	30.73	60.85	39.15
average	**29.60**	**58.83**	**41.17**
confidence limit	**1.15**	**2.05**	**2.05**
SD	**0.93**	**1.65**	**1.65**
CV %	**3.13**	**2.81**	**4.01**

Quantification of heat treated samples at 245 °C for 10 min and quenched for 5 min *via* calibration curve built with various PTTs and PETs treated with the same conditions

Quantification based on PTT peak integration

PTT 317 %	PET 316 %	PTT enthalpy J/g
0.00	100.00	0.00
20.10	79.90	7.82
37.97	62.03	16.29
71.29	28.71	33.42
80.18	19.82	37.79
100.00	0.00	53.39

PTT 296 %	PET 316 %	PTT enthalpy J/g
0.00	100.00	0.00
40.94	59.06	18.33
48.11	51.89	21.77
57.86	42.14	26.56
72.38	27.62	36.28
78.94	21.06	41.20
100.00	0.00	52.21

PTT 295 %	PET 316 %	PTT enthalpy J/g
0.00	100.00	0.00
31.22	68.78	12.91
42.98	57.02	19.77
53.78	46.22	26.94
71.75	28.25	37.53
80.24	19.76	41.58
100.00	0.00	54.49

		calibration with PTT (317) and PET (316)		calibration with PTT (296) and PET (316)		calibration with PTT (295) and PET (316)	
JRC code	**PTT enthalpy J/g**	**PTT %**	**PET %**	**PTT %**	**PET %**	**PTT %**	**PET %**
298-1	28.97	63.57	36.43	60.78	39.22	58.81	41.19
298-2	28.85	63.37	36.63	60.57	39.43	58.60	41.40
298-3	30.10	65.52	34.48	62.81	37.19	60.76	39.24
298-4	30.17	65.64	34.36	62.93	37.07	60.88	39.12
298-5	29.81	65.02	34.98	62.29	37.71	60.26	39.74
average	**29.58**	**64.63**	**35.37**	**61.88**	**38.12**	**59.86**	**40.14**
confidence limit	**0.78**	**1.34**	**1.34**	**1.40**	**1.40**	**1.35**	**1.35**
SD	**0.63**	**1.08**	**1.08**	**1.12**	**1.12**	**1.09**	**1.09**
CV %	**2.12**	**1.68**	**3.06**	**1.82**	**2.95**	**1.82**	**2.71**
302-1	32.69	69.90	30.10	67.38	32.62	65.18	34.82
302-2	31.47	67.85	32.15	65.24	34.76	63.11	36.89
302-3	33.47	71.19	28.81	68.73	31.27	66.49	33.51
302-4	31.91	68.59	31.41	66.01	33.99	63.86	36.14
302-5	35.56	74.60	25.40	72.33	27.67	69.97	30.03
average	**33.02**	**70.43**	**29.57**	**67.94**	**32.06**	**65.72**	**34.28**
confidence limit	**2.00**	**3.30**	**3.30**	**3.47**	**3.47**	**3.35**	**3.35**
SD	**1.61**	**2.66**	**2.66**	**2.80**	**2.80**	**2.70**	**2.70**
CV %	**4.88**	**3.78**	**9.00**	**4.12**	**8.72**	**4.11**	**7.88**
309-1	39.56	80.95	19.05	79.06	20.94	76.47	23.53
309-2	36.64	76.34	23.66	74.17	25.83	71.74	28.26
309-3	38.69	79.59	20.41	77.61	22.39	75.07	24.93
309-4	37.32	77.43	22.57	75.32	24.68	72.85	27.15
309-5	39.32	80.58	19.42	78.66	21.34	76.08	23.92
average	**38.31**	**78.98**	**21.02**	**76.96**	**23.04**	**74.44**	**25.56**
confidence limit	**1.58**	**2.50**	**2.50**	**2.65**	**2.65**	**2.56**	**2.56**
SD	**1.27**	**2.01**	**2.01**	**2.14**	**2.14**	**2.06**	**2.06**
CV %	**3.33**	**2.55**	**9.56**	**2.77**	**9.27**	**2.77**	**8.07**
311-1	34.51	72.90	27.10	70.53	29.47	68.23	31.77
311-2	33.83	71.78	28.22	69.36	30.64	67.09	32.91
311-3	33.78	71.70	28.30	69.27	30.73	67.01	32.99
311-4	33.31	70.92	29.08	68.46	31.54	66.22	33.78
311-5	32.54	69.65	30.35	68.85	31.15	66.61	33.39
average	**33.59**	**71.39**	**28.61**	**69.29**	**30.71**	**67.03**	**32.97**
confidence limit	**0.90**	**1.49**	**1.49**	**0.97**	**0.97**	**0.94**	**0.94**
SD	**0.73**	**1.20**	**1.20**	**0.78**	**0.78**	**0.75**	**0.75**
CV %	**2.17**	**1.68**	**4.20**	**1.12**	**2.54**	**1.12**	**2.28**
312-1	29.30	64.15	35.85	61.38	38.62	59.38	40.62
312-2	30.58	66.34	33.66	63.66	36.34	61.59	38.41
312-3	29.87	65.13	34.87	62.40	37.60	60.37	39.63
312-4	30.66	66.48	33.52	63.80	36.20	61.73	38.27
312-5	31.12	67.26	32.74	64.62	35.38	62.51	37.49
average	**30.31**	**65.87**	**34.13**	**63.17**	**36.83**	**61.11**	**38.89**
confidence limit	**0.89**	**1.53**	**1.53**	**1.59**	**1.59**	**1.54**	**1.54**
SD	**0.72**	**1.23**	**1.23**	**1.28**	**1.28**	**1.24**	**1.24**
CV %	**2.37**	**1.87**	**3.60**	**2.03**	**3.48**	**2.02**	**3.18**

Quantification based on PET peak integration

PTT 317 %	PET 316 %	PET enthalpy J/g
100.00	0.00	0.00
80.18	19.82	7.37
71.29	28.71	10.50
37.97	62.03	24.92
20.10	79.90	31.75

PTT 296 %	PET 316 %	PET enthalpy J/g
100.00	0.00	0.00
78.94	21.06	8.10
72.38	27.62	10.89
57.86	42.14	15.92
48.11	51.89	21.14
40.94	59.06	24.05

PTT 295 %	PET 316 %	PET enthalpy J/g
100.00	0.00	0.00
80.24	19.76	8.12
71.75	28.25	11.94
53.78	46.22	18.77
42.98	57.02	22.32
31.22	68.78	29.24

		calibration with PTT (317) and PET (316)		calibration with PTT (296) and PET (316)		calibration with PTT (295) and PET (316)	
JRC code	PET enthalpy J/g	PTT %	PET %	PTT %	PET %	PTT %	PET %
309-1	12.38	32.68	67.32	32.16	67.84	30.70	69.30
309-2	11.37	30.09	69.91	29.67	70.33	28.25	71.75
309-3	12.88	33.95	66.05	33.38	66.62	31.91	68.09
309-4	10.80	28.63	71.37	28.25	71.75	26.86	73.14
309-5	12.23	32.29	67.71	31.79	68.21	30.34	69.66
average	**11.93**	**31.53**	**68.47**	**31.05**	**68.95**	**29.61**	**70.39**
confidence limit	**1.04**	**2.65**	**2.65**	**2.55**	**2.55**	**2.52**	**2.52**
SD	**0.83**	**2.14**	**2.14**	**2.06**	**2.06**	**2.03**	**2.03**
CV %	**6.99**	**6.77**	**3.12**	**6.63**	**2.98**	**6.85**	**2.88**
311-1	10.61	28.14	71.86	27.78	72.22	26.40	73.60
311-2	10.41	27.62	72.38	27.28	72.72	25.91	74.09
311-3	10.09	26.80	73.20	26.48	73.52	25.13	74.87
311-4	10.55	27.98	72.02	27.63	72.37	26.25	73.75
311-5	9.96	26.46	73.54	26.15	73.85	24.81	75.19
average	**10.32**	**27.40**	**72.60**	**27.06**	**72.94**	**25.70**	**74.30**
confidence limit	**0.36**	**0.92**	**0.92**	**0.89**	**0.89**	**0.87**	**0.87**
SD	**0.29**	**0.74**	**0.74**	**0.71**	**0.71**	**0.70**	**0.70**
CV %	**2.77**	**2.69**	**1.02**	**2.64**	**0.98**	**2.72**	**0.94**
312-1	10.73	28.45	71.55	28.08	71.92	26.69	73.31
312-2	11.58	30.63	69.37	30.19	69.81	28.76	71.24
312-3	12.43	32.80	67.20	32.28	67.72	30.82	69.18
312-4	12.80	33.75	66.25	33.18	66.82	31.72	68.28
average	**11.89**	**31.41**	**68.59**	**30.93**	**69.07**	**29.50**	**70.50**
confidence limit	**1.47**	**3.76**	**3.76**	**3.63**	**3.63**	**3.57**	**3.57**
SD	**0.92**	**2.37**	**2.37**	**2.28**	**2.28**	**2.25**	**2.25**
CV %	**7.77**	**7.53**	**3.45**	**7.37**	**3.30**	**7.61**	**3.18**

Quantification of heat treated samples at 235 °C for 10 min and quenched for 10 min *via* calibration curve built with various PTTs and PETs treated with the same conditions

Quantification based on PTT peak integration

PTT 317 %	PET 316 %	PTT enthalpy J/g	PTT 296 %	PET 316 %	PTT enthalpy J/g	PTT 295 %	PET 316 %	PTT enthalpy J/g
0.00	100.00	0.00	0.00	100.00	0.00	0.00	100.00	0.00
25.44	74.56	12.03	36.70	63.30	17.83	24.40	75.60	11.52
37.89	62.11	18.87	23.36	76.64	10.99	35.50	64.50	17.57
48.96	51.04	24.85	50.57	49.43	25.38	48.86	51.14	24.27
62.68	37.32	31.64	66.29	33.71	33.46	62.72	37.28	32.00
75.54	24.46	38.58	84.72	15.28	44.27	74.30	25.70	37.96
100.00	0.00	51.72	100.00	0.00	50.99	100.00	0.00	54.30

		calibration with PTT (317) and PET (316)		calibration with PTT (296) and PET (316)		calibration with PTT (295) and PET (316)	
JRC code	**PTT enthalpy J/g**	**PTT %**	**PET %**	**PTT %**	**PET %**	**PTT %**	**PET %**
298-1	31.30	61.32	38.68	61.41	38.59	60.07	39.93
298-2	30.92	60.58	39.42	60.66	39.34	59.34	40.66
298-3	31.39	61.50	38.50	61.59	38.41	60.24	39.76
298-4	32.99	64.64	35.36	64.72	35.28	63.31	36.69
298-5	33.02	64.69	35.31	64.78	35.22	63.37	36.63
average	**31.92**	**62.55**	**37.45**	**62.63**	**37.37**	**61.26**	**38.74**
confidence limit	**1.24**	**2.44**	**2.44**	**2.44**	**2.44**	**2.39**	**2.39**
SD	**1.00**	**1.96**	**1.96**	**1.97**	**1.97**	**1.92**	**1.92**
CV %	**3.14**	**3.14**	**5.24**	**3.14**	**5.26**	**3.14**	**4.97**
302-1	39.08	76.57	23.43	76.67	23.33	73.94	26.06
302-2	38.53	75.49	24.51	75.59	24.41	74.30	25.70
302-3	38.72	75.86	24.14	75.97	24.03	74.11	25.89
302-4	38.62	75.67	24.33	75.77	24.23	73.52	26.48
302-5	38.31	75.06	24.94	75.16	24.84	74.17	25.83
average	**38.65**	**75.73**	**24.27**	**75.83**	**24.17**	**74.01**	**25.99**
confidence limit	**0.35**	**0.69**	**0.69**	**0.69**	**0.69**	**0.38**	**0.38**
SD	**0.28**	**0.55**	**0.55**	**0.56**	**0.56**	**0.30**	**0.30**
CV %	**0.73**	**0.73**	**2.29**	**0.73**	**2.30**	**0.41**	**1.17**
309-1	37.74	73.94	26.06	74.04	25.96	74.30	25.70
309-2	38.72	75.86	24.14	75.97	24.03	74.27	25.73
309-3	38.70	75.82	24.18	75.93	24.07	73.67	26.33
309-4	38.39	75.22	24.78	75.32	24.68	73.06	26.94
309-5	38.07	74.59	25.41	74.69	25.31	73.54	26.46
average	**38.32**	**75.09**	**24.91**	**75.19**	**24.81**	**73.77**	**26.23**
confidence limit	**0.52**	**1.02**	**1.02**	**1.02**	**1.02**	**0.65**	**0.65**
SD	**0.42**	**0.82**	**0.82**	**0.83**	**0.83**	**0.52**	**0.52**
CV %	**1.10**	**1.10**	**3.31**	**1.10**	**3.33**	**0.71**	**2.00**
311-1	33.73	66.09	33.91	66.18	33.82	64.73	35.27
311-2	33.36	65.36	34.64	65.45	34.55	64.02	35.98
311-3	34.20	67.01	32.99	67.10	32.90	65.63	34.37
311-4	34.01	66.63	33.37	66.73	33.27	65.27	34.73
311-5	34.89	68.36	31.64	68.45	31.55	66.95	33.05
average	**34.04**	**66.69**	**33.31**	**66.78**	**33.22**	**65.32**	**34.68**
confidence limit	**0.71**	**1.39**	**1.39**	**1.39**	**1.39**	**1.36**	**1.36**
SD	**0.57**	**1.12**	**1.12**	**1.12**	**1.12**	**1.10**	**1.10**
CV %	**1.68**	**1.68**	**3.36**	**1.68**	**3.38**	**1.68**	**3.16**
312-1	31.67	62.05	37.95	62.13	37.87	60.78	39.22
312-2	31.50	61.72	38.28	61.80	38.20	60.45	39.55
312-3	33.68	65.99	34.01	66.08	33.92	64.63	35.37
312-4	33.15	64.95	35.05	65.04	34.96	63.62	36.38
312-5	33.98	66.58	33.42	66.67	33.33	65.21	34.79
average	**32.80**	**64.26**	**35.74**	**64.34**	**35.66**	**62.94**	**37.06**
confidence limit	**1.42**	**2.79**	**2.79**	**2.79**	**2.79**	**2.73**	**2.73**
SD	**1.15**	**2.25**	**2.25**	**2.25**	**2.25**	**2.20**	**2.20**
CV %	**3.50**	**3.50**	**6.28**	**3.50**	**6.31**	**3.50**	**5.94**

Quantification based on PET peak integration

PTT 317 %	PET 316 %	PET enthalpy J/g
100.00	0.00	0.00
75.54	24.46	13.65
62.68	37.32	20.84
48.96	51.04	28.49
37.89	62.11	34.79
25.44	74.56	41.97
0.00	100.00	55.21

PTT 296 %	PET 316 %	PET enthalpy J/g
100.00	0.00	0.00
84.72	15.28	8.38
66.29	33.71	18.53
50.57	49.43	27.75
36.70	63.30	35.49
23.36	76.64	43.25
0.00	100.00	55.21

PTT 295 %	PET 316 %	PET enthalpy J/g
100.00	0.00	0.00
74.30	25.70	13.67
48.86	51.14	28.78
35.50	64.50	36.26
24.40	75.60	42.44
0.00	100.00	55.21

		calibration with PTT (317) and PET (316)		calibration with PTT (296) and PET (316)		calibration with PTT (295) and PET (316)	
JRC code	PET enthalpy J/g	PTT %	PET %	PTT %	PET %	PTT %	PET %
298-1	16.86	30.26	69.74	30.25	69.75	30.28	69.72
298-2	16.95	30.43	69.57	30.41	69.59	30.44	69.56
298-3	17.36	31.16	68.84	31.14	68.86	31.18	68.82
298-4	16.54	29.69	70.31	29.67	70.33	29.71	70.29
298-5	17.70	31.77	68.23	31.75	68.25	31.79	68.21
average	**17.08**	**30.66**	**69.34**	**30.65**	**69.35**	**30.68**	**69.32**
confidence limit	**0.56**	**1.01**	**1.01**	**1.01**	**1.01**	**1.01**	**1.01**
SD	**0.45**	**0.81**	**0.81**	**0.81**	**0.81**	**0.81**	**0.81**
CV %	**2.65**	**2.65**	**1.17**	**2.65**	**1.17**	**2.65**	**1.17**
302-1	9.72	17.45	82.55	17.45	82.55	17.46	82.54
302-2	9.03	16.21	83.79	16.20	83.80	16.21	83.79
302-3	9.78	17.55	82.45	17.54	82.46	17.56	82.44
302-4	9.92	17.80	82.20	17.79	82.21	17.81	82.19
302-5	9.02	16.19	83.81	16.18	83.82	16.20	83.80
average	**9.49**	**17.04**	**82.96**	**17.03**	**82.97**	**17.05**	**82.95**
confidence limit	**0.54**	**0.97**	**0.97**	**0.97**	**0.97**	**0.97**	**0.97**
SD	**0.43**	**0.78**	**0.78**	**0.78**	**0.78**	**0.78**	**0.78**
CV %	**4.58**	**4.58**	**0.94**	**4.58**	**0.94**	**4.58**	**0.94**
309-1	13.31	23.89	76.11	23.88	76.12	23.90	76.10
309-2	13.30	23.87	76.13	23.86	76.14	23.89	76.11
309-3	13.06	23.44	76.56	23.43	76.57	23.46	76.54
309-4	13.99	25.11	74.89	25.10	74.90	25.13	74.87
309-5	13.41	24.07	75.93	24.06	75.94	24.08	75.92
average	**13.41**	**24.08**	**75.92**	**24.07**	**75.93**	**24.09**	**75.91**
confidence limit	**0.43**	**0.77**	**0.77**	**0.77**	**0.77**	**0.77**	**0.77**
SD	**0.35**	**0.62**	**0.62**	**0.62**	**0.62**	**0.62**	**0.62**
CV %	**2.58**	**2.58**	**0.82**	**2.58**	**0.82**	**2.58**	**0.82**
311-1	14.71	26.40	73.60	26.39	73.61	26.42	73.58
311-2	14.77	26.51	73.49	26.50	73.50	26.53	73.47
311-3	15.71	28.20	71.80	28.18	71.82	28.21	71.79
311-4	15.11	27.12	72.88	27.11	72.89	27.14	72.86
311-5	16.10	28.90	71.10	28.88	71.12	28.92	71.08
average	**15.28**	**27.43**	**72.57**	**27.41**	**72.59**	**27.44**	**72.56**
confidence limit	**0.75**	**1.35**	**1.35**	**1.35**	**1.35**	**1.35**	**1.35**
SD	**0.61**	**1.09**	**1.09**	**1.09**	**1.09**	**1.09**	**1.09**
CV %	**3.97**	**3.97**	**1.50**	**3.97**	**1.50**	**3.97**	**1.50**
312-1	18.06	32.42	67.58	32.40	67.60	32.44	67.56
312-2	16.81	30.17	69.83	30.16	69.84	30.19	69.81
312-3	15.48	27.79	72.21	27.77	72.23	27.80	72.20
312-4	17.77	31.90	68.10	31.88	68.12	31.91	68.09
312-5	16.51	29.64	70.36	29.62	70.38	29.65	70.35
average	**16.93**	**30.38**	**69.62**	**30.37**	**69.63**	**30.40**	**69.60**
confidence limit	**1.28**	**2.30**	**2.30**	**2.30**	**2.30**	**2.31**	**2.31**
SD	**1.03**	**1.86**	**1.86**	**1.85**	**1.85**	**1.86**	**1.86**
CV %	**6.11**	**6.11**	**2.67**	**6.11**	**2.66**	**6.11**	**2.67**

Quantification of heat treated samples at 235 °C for 20 min and quenched for 10 min *via* calibration curve built with various PTTs and PETs treated with the same conditions

Quantification based on PTT peak integration

PTT 317 %	PET 316 %	PTT enthalpy J/g
0.00	100.00	0.00
32.46	67.54	17.34
41.23	58.77	22.03
57.60	42.40	29.84
71.16	28.84	38.07
74.41	25.59	39.37

PTT 296 %	PET 316 %	PTT enthalpy J/g
0.00	100.00	0.00
30.33	69.67	14.14
44.71	55.29	23.24
61.86	38.14	32.34
83.20	16.80	44.62

PTT 295 %	PET 316 %	PTT enthalpy J/g
0.00	100.00	0.00
31.42	68.58	15.03
42.21	57.79	21.54
48.21	51.79	24.89
60.62	39.38	31.86
70.69	29.31	37.35

		calibration with PTT (317) and PET (316)		calibration with PTT (296) and PET (316)		calibration with PTT (295) and PET (316)	
JRC code	PTT enthalpy J/g	PTT %	PET %	PTT %	PET %	PTT %	PET %
302-1	38.66	73.01	26.99	73.58	26.42	74.40	25.60
302-2	38.96	73.58	26.42	74.15	25.85	74.98	25.02
302-3	38.69	73.07	26.93	73.64	26.36	74.46	25.54
302-4	38.53	72.77	27.23	73.33	26.67	74.15	25.85
302-5	37.79	71.37	28.63	71.93	28.07	72.73	27.27
302-6	38.01	71.78	28.22	72.34	27.66	73.15	26.85
average	**38.44**	**72.60**	**27.40**	**73.16**	**26.84**	**73.98**	**26.02**
confidence limit	**0.47**	**0.88**	**0.88**	**0.89**	**0.89**	**0.90**	**0.90**
SD	**0.45**	**0.84**	**0.84**	**0.85**	**0.85**	**0.86**	**0.86**
CV %	**1.16**	**1.16**	**3.08**	**1.16**	**3.17**	**1.16**	**3.30**
309-1	39.47	74.54	25.46	75.12	24.88	75.96	24.04
309-2	39.35	74.32	25.68	74.90	25.10	75.73	24.27
309-3	39.40	74.41	25.59	74.99	25.01	75.83	24.17
309-4	38.62	72.94	27.06	73.51	26.49	74.33	25.67
309-5	37.96	71.69	28.31	72.25	27.75	73.06	26.94
309-6	39.28	74.18	25.82	74.76	25.24	75.60	24.40
average	**39.01**	**73.68**	**26.32**	**74.25**	**25.75**	**75.08**	**24.92**
confidence limit	**0.63**	**1.19**	**1.19**	**1.20**	**1.20**	**1.21**	**1.21**
SD	**0.60**	**1.14**	**1.14**	**1.14**	**1.14**	**1.16**	**1.16**
CV %	**1.54**	**1.54**	**4.31**	**1.54**	**4.44**	**1.54**	**4.64**
311-1	36.16	68.29	31.71	68.82	31.18	69.59	30.41
311-2	36.18	68.33	31.67	68.86	31.14	69.63	30.37
311-3	36.56	69.05	30.95	69.59	30.41	70.36	29.64
311-4	36.93	69.75	30.25	70.29	29.71	71.07	28.93
311-5	36.95	69.78	30.22	70.33	29.67	71.11	28.89
311-6	36.35	68.65	31.35	69.19	30.81	69.96	30.04
average	**36.52**	**68.97**	**31.03**	**69.51**	**30.49**	**70.29**	**29.71**
confidence limit	**0.37**	**0.70**	**0.70**	**0.71**	**0.71**	**0.72**	**0.72**
SD	**0.35**	**0.67**	**0.67**	**0.67**	**0.67**	**0.68**	**0.68**
CV %	**0.97**	**0.97**	**2.16**	**0.97**	**2.21**	**0.97**	**2.30**
312-1	32.64	61.64	38.36	62.12	37.88	62.82	37.18
312-2	32.39	61.17	38.83	61.65	38.35	62.34	37.66
312-3	31.97	60.38	39.62	60.85	39.15	61.53	38.47
312-4	32.40	61.19	38.81	61.67	38.33	62.36	37.64
312-5	31.48	59.45	40.55	59.92	40.08	60.59	39.41
312-6	31.52	59.53	40.47	59.99	40.01	60.66	39.34
average	**32.07**	**60.56**	**39.44**	**61.03**	**38.97**	**61.71**	**38.29**
confidence limit	**0.51**	**0.97**	**0.97**	**0.98**	**0.98**	**0.99**	**0.99**
SD	**0.49**	**0.92**	**0.92**	**0.93**	**0.93**	**0.94**	**0.94**
CV %	**1.53**	**1.53**	**2.34**	**1.53**	**2.39**	**1.53**	**2.46**

Quantification based on PET peak integration

PTT 317 %	PET 316 %	PET enthalpy J/g
100.00	0.00	0.00
74.41	25.59	14.57
71.16	28.84	17.08
57.60	42.40	25.04
41.23	58.77	33.91
32.46	67.54	39.53

PTT 296 %	PET 316 %	PET enthalpy J/g
100.00	0.00	0.00
83.20	16.80	9.68
61.86	38.14	22.24
44.71	55.29	32.61
30.33	69.67	40.03

PTT 295 %	PET 316 %	PET enthalpy J/g
100.00	0.00	0.00
70.69	29.31	17.16
60.62	39.38	23.06
48.21	51.79	29.78
42.21	57.79	33.43
31.42	68.58	39.54

		calibration with PTT (317) and PET (316)		calibration with PTT (296) and PET (316)		calibration with PTT (295) and PET (316)	
JRC code	PET enthalpy J/g	PTT %	PET %	PTT %	PET %	PTT %	PET %
302-1	11.71	20.08	79.92	20.17	79.83	20.25	79.75
302-2	11.94	20.47	79.53	20.56	79.44	20.64	79.36
302-3	11.83	20.28	79.72	20.37	79.63	20.45	79.55
302-4	11.62	19.92	80.08	20.01	79.99	20.09	79.91
302-5	11.41	19.56	80.44	19.65	80.35	19.73	80.27
average	**11.70**	**20.07**	**79.93**	**20.15**	**79.85**	**20.23**	**79.77**
confidence limit	**0.25**	**0.43**	**0.43**	**0.43**	**0.43**	**0.44**	**0.44**
SD	**0.20**	**0.35**	**0.35**	**0.35**	**0.35**	**0.35**	**0.35**
CV %	**1.74**	**1.74**	**0.44**	**1.74**	**0.44**	**1.74**	**0.44**
309-1	14.52	24.90	75.10	25.00	75.00	25.10	74.90
309-2	14.20	24.35	75.65	24.45	75.55	24.55	75.45
309-3	13.98	23.97	76.03	24.07	75.93	24.17	75.83
309-4	13.84	23.73	76.27	23.83	76.17	23.93	76.07
309-5	14.15	24.26	75.74	24.37	75.63	24.46	75.54
average	**14.14**	**24.24**	**75.76**	**24.35**	**75.65**	**24.44**	**75.56**
confidence limit	**0.32**	**0.46**	**0.46**	**0.46**	**0.46**	**0.47**	**0.47**
SD	**0.26**	**0.44**	**0.44**	**0.44**	**0.44**	**0.44**	**0.44**
CV %	**1.82**	**1.82**	**0.58**	**1.82**	**0.58**	**1.82**	**0.59**
311-1	16.75	28.72	71.28	28.84	71.16	28.96	71.04
311-2	16.95	29.06	70.94	29.19	70.81	29.30	70.70
311-3	17.08	29.29	70.71	29.41	70.59	29.53	70.47
311-4	17.06	29.25	70.75	29.38	70.62	29.50	70.50
311-5	17.27	29.61	70.39	29.74	70.26	29.86	70.14
311-6	16.78	28.77	71.23	28.90	71.10	29.01	70.99
average	**16.98**	**29.12**	**70.88**	**29.24**	**70.76**	**29.36**	**70.64**
confidence limit	**0.21**	**0.35**	**0.35**	**0.36**	**0.36**	**0.36**	**0.36**
SD	**0.20**	**0.34**	**0.34**	**0.34**	**0.34**	**0.34**	**0.34**
CV %	**1.16**	**1.16**	**0.48**	**1.16**	**0.48**	**1.16**	**0.48**
312-1	21.10	36.18	63.82	36.34	63.66	36.48	63.52
312-2	20.99	35.99	64.01	36.15	63.85	36.29	63.71
312-3	20.66	35.43	64.57	35.58	64.42	35.72	64.28
312-4	20.82	35.70	64.30	35.85	64.15	36.00	64.00
312-5	20.21	34.65	65.35	34.80	65.20	34.94	65.06
312-6	20.82	35.70	64.30	35.85	64.15	36.00	64.00
average	**20.77**	**35.61**	**64.39**	**35.76**	**64.24**	**35.90**	**64.10**
confidence limit	**0.33**	**0.56**	**0.56**	**0.56**	**0.56**	**0.57**	**0.57**
SD	**0.31**	**0.54**	**0.54**	**0.54**	**0.54**	**0.54**	**0.54**
CV %	**1.50**	**1.50**	**0.83**	**1.50**	**0.84**	**1.50**	**0.84**

Quantification of samples *via* calibration curve built with PTTs and PETs manually extracted from the samples to be quantified

3 days study

LINEAR INTEGRATION

Quantification based on PTT peak integration

DAY 1

PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
21.45	78.55	12.12
36.75	63.25	21.26
51.87	48.13	31.00
68.98	31.02	40.72
84.77	15.23	53.12
100.00	0.00	64.31

DAY 2

PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
18.32	81.68	8.92
30.27	69.73	16.11
54.98	45.02	30.74
80.27	19.73	47.21
100.00	0.00	61.42

DAY 3

PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
24.84	75.16	13.11
29.71	70.29	15.76
44.47	55.53	24.96
54.52	45.48	31.62
61.68	38.32	34.05
75.74	24.26	45.58
86.49	13.51	48.80
100.00	0.00	63.33

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-302-1	64.02	302-1	46.20	74.54	71.86
	PTT-302-2	64.47	302-2	47.01	75.85	73.12
	PTT-302-3	64.43	302-3	45.90	74.06	71.40
	average	64.31		46.37	74.81	72.13
	confidence limit	0.62		1.43	2.30	2.22
	SD	0.25		0.57	0.93	0.89
	CV %	0.39		1.24	1.24	1.24
Day 2	PTT-302-1	62.33	302-1	44.51	76.01	72.47
	PTT-302-2	60.29	302-2	45.27	77.11	73.71
	PTT-302-3	62.66	302-3	44.64	76.20	72.69
	PTT-302-4	60.38	302-4	45.22	77.04	73.63
	average	61.42		44.91	76.59	73.13
	confidence limit	2.00		0.62	0.91	1.01
	SD	1.25		0.39	0.57	0.64
	CV %	2.04		0.87	0.74	0.87
Day 3	PTT-302-1	63.89	302-1	45.30	77.22	71.53
	PTT-302-2	63.04	302-2	45.13	76.97	71.26
	PTT-302-3	62.75	302-3	46.07	78.35	72.74
	PTT-302-4	63.65	302-4	45.82	77.98	72.35
	average	63.33		45.58	77.63	71.97
	confidence limit	0.84		0.70	1.03	1.10
	SD	0.53		0.44	0.65	0.69
	CV %	0.83		0.96	0.83	0.96

DAY 1		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
19.95	80.05	10.76
30.53	69.47	16.16
47.34	52.66	26.41
71.51	28.49	41.11
83.03	16.97	49.73
100.00	0.00	59.14

DAY 2		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
22.36	77.64	12.05
44.55	55.45	24.89
58.05	41.95	35.49
72.66	27.34	43.49
79.95	20.05	47.67
100.00	0.00	62.8

DAY 3		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
23.48	76.52	13.25
30.41	69.59	17.81
40.64	59.36	23.80
48.49	51.51	28.59
58.99	41.01	36.20
71.17	28.83	43.33
79.31	20.69	48.39
100.00	0.00	61.84

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-309-1	59.71	309-1	45.85	78.50	77.53
	PTT-309-2	58.85	309-2	44.21	75.69	74.75
	PTT-309-3	58.85	309-3	42.59	72.92	72.02
	average	59.14		44.22	75.70	74.77
	confidence limit	1.23		2.59	6.93	6.85
	SD	0.50		1.63	2.79	2.76
	CV %	0.84		3.69	3.69	3.69
Day 2	PTT-309-1	62.40	309-1	46.58	76.69	74.17
	PTT-309-2	65.46	309-2	46.29	76.26	73.71
	PTT-309-3	62.33	309-3	47.73	78.37	76.00
	PTT-309-4	61.02	309-4	46.60	76.72	74.20
	average	62.80		46.80	77.01	74.52
	confidence limit	2.99		1.01	1.48	1.61
	SD	1.88		0.64	0.93	1.01
	CV %	3.00		1.36	1.20	1.36
Day 3	PTT-309-1	61.81	309-1	46.86	77.01	75.77
	PTT-309-2	61.32	309-2	46.92	77.11	75.87
	PTT-309-3	63.03	309-3	46.77	76.86	75.63
	PTT-309-4	61.21	309-4	46.94	77.14	75.90
	average	61.84		46.87	77.03	75.79
	confidence limit	1.33		0.12	0.20	0.20
	SD	0.83		0.08	0.13	0.12
	CV %	1.35		0.16	0.16	0.16

DAY 1		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
24.32	75.68	13.23
39.25	60.75	21.11
47.44	52.56	27.09
64.81	35.19	39.91
78.21	21.79	47.97
100.00	0.00	60.07

DAY 2		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
20.45	79.55	12.10
38.11	61.89	22.70
61.15	38.85	36.95
73.83	26.17	46.59
100.00	0.00	61.17

DAY 3		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
26.52	73.48	15.53
34.99	65.01	21.32
46.77	53.23	27.89
59.14	40.86	36.69
70.72	29.28	45.08
77.24	22.76	48.63
100.00	0.00	62.88

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	**PTT-311-1**	61.17	**311-1**	43.64	72.93	72.65
	PTT-311-2	60.11	**311-2**	40.72	68.05	67.79
	PTT-311-3	58.92	**311-3**	43.45	72.61	72.34
	average	**60.07**		**42.60**	**71.20**	**70.93**
	confidence limit	**2.80**		**4.06**	**6.78**	**6.76**
	SD	**1.13**		**1.63**	**2.73**	**2.72**
	CV %	**1.87**		**3.83**	**3.83**	**3.83**
Day 2	**PTT-311-1**	60.04	**311-1**	44.47	72.27	72.34
	PTT-311-2	61.98	**311-2**	44.94	73.04	73.11
	PTT-311-3	61.06	**311-3**	44.41	72.18	72.25
	PTT-311-4	62.80	**311-4**	44.81	72.83	72.90
	average	**61.47**		**44.66**	**72.58**	**72.65**
	confidence limit	**1.89**		**0.41**	**0.67**	**0.67**
	SD	**1.19**		**0.26**	**0.42**	**0.42**
	CV %	**1.93**		**0.58**	**0.58**	**0.58**
Day 3	**PTT-311-1**	62.74	**311-1**	45.11	72.16	71.74
	PTT-311-2	61.66	**311-2**	44.92	71.86	71.44
	PTT-311-3	63.35	**311-3**	44.71	71.52	71.10
	PTT-311-4	63.63	**311-4**	44.99	71.97	71.55
	average	**62.85**		**44.93**	**71.88**	**71.46**
	confidence limit	**1.39**		**0.27**	**0.43**	**0.42**
	SD	**0.87**		**0.17**	**0.27**	**0.27**
	CV %	**1.39**		**0.37**	**0.37**	**0.37**

DAY 1 PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
23.53	76.47	11.49
35.52	64.48	19.84
53.15	46.85	31.48
70.49	29.51	41.87
78.69	21.31	49.49
100.00	0.00	59.57

DAY 2 PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
21.29	78.71	12.02
34.38	65.62	19.36
51.45	48.55	30.29
62.42	37.58	36.68
73.51	26.49	44.48
100.00	0.00	59.73

DAY 3 PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
22.67	77.33	12.62
32.70	67.30	18.33
45.22	54.78	26.19
54.33	45.67	30.72
73.38	26.62	44.23
79.39	20.61	47.85
100.00	0.00	59.50

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-312-1	58.99	312-1	36.67	61.23	61.56
	PTT-312-2	60.15	312-2	37.67	62.90	63.24
	PTT-312-3	59.58	312-3	38.38	64.08	64.43
	average	59.57		37.57	62.74	63.07
	confidence limit	1.44		2.13	3.56	3.58
	SD	0.58		0.86	1.43	1.44
	CV %	0.97		2.29	2.29	2.29
Day 2	PTT-312-1	59.75	312-1	39.42	66.34	66.00
	PTT-312-2	60.27	312-2	38.77	65.25	64.91
	PTT-312-3	59.17	312-3	36.81	61.95	61.63
	PTT-312-4		312-4	39.79	66.96	66.62
	average	59.73		38.70	65.13	64.79
	confidence limit	1.37		2.11	3.55	3.54
	SD	0.55		1.33	2.23	2.22
	CV %	0.92		3.43	3.43	3.43
Day 3	PTT-312-1	59.72	312-1	37.97	64.14	63.82
	PTT-312-2	59.38	312-2	37.58	63.48	63.16
	PTT-312-3	60.52	312-3	38.34	64.76	64.44
	PTT-312-4	58.38	312-4	37.82	63.89	63.56
	average	59.50		37.93	64.07	63.74
	confidence limit	1.41		0.51	0.86	0.85
	SD	0.89		0.32	0.54	0.54
	CV %	1.49		0.84	0.84	0.84

PARALLEL INTEGRATION

Quantification based on PTT peak integration

DAY 1

PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
21.45	78.55	10.47
36.75	63.25	19.38
51.87	48.13	28.88
68.98	31.02	39.06
84.77	15.23	51.10
100.00	0.00	59.33

DAY 2

PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
18.32	81.68	7.55
30.27	69.73	14.58
54.98	45.02	28.86
80.27	19.73	45.07
100.00	0.00	59.01

DAY 3

PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
24.84	75.16	11.55
29.71	70.29	14.14
44.47	55.53	23.21
54.52	45.48	29.58
61.68	38.32	32.18
75.74	24.26	43.26
86.49	13.51	46.61
100.00	0.00	60.21

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	**PTT-302-1**	58.51	**302-1**	43.30	74.39	72.98
	PTT-302-2	60.69	**302-2**	44.25	76.02	74.58
	PTT-302-3	58.80	**302-3**	43.28	74.35	72.94
	average	**59.33**		**43.61**	**74.92**	**73.50**
	confidence limit	**2.94**		**1.38**	**2.37**	**2.32**
	SD	**1.18**		**0.55**	**0.95**	**0.93**
	CV %	**2.00**		**1.27**	**1.27**	**1.27**
Day 2	**PTT-302-1**	59.81	**302-1**	42.30	76.64	71.69
	PTT-302-2	58.06	**302-2**	42.93	77.57	72.76
	PTT-302-3	60.07	**302-3**	42.44	76.85	71.93
	PTT-302-4	58.08	**302-4**	42.98	77.65	72.84
	average	**59.01**		**42.66**	**77.18**	**72.30**
	confidence limit	**1.73**		**0.55**	**0.81**	**0.93**
	SD	**1.08**		**0.34**	**0.51**	**0.58**
	CV %	**1.84**		**0.80**	**0.66**	**0.80**
Day 3	**PTT-302-1**	60.83	**302-1**	43.02	76.93	71.46
	PTT-302-2	59.42	**302-2**	42.85	76.68	71.17
	PTT-302-3	60.05	**302-3**	43.71	77.95	72.60
	PTT-302-4	60.52	**302-4**	43.45	77.57	72.17
	average	**60.21**		**43.26**	**77.28**	**71.85**
	confidence limit	**0.98**		**0.63**	**0.93**	**1.04**
	SD	**0.61**		**0.39**	**0.58**	**0.65**
	CV %	**1.02**		**0.91**	**0.75**	**0.91**

DAY 1		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
19.95	80.05	9.78
30.53	69.47	15.48
47.34	52.66	24.89
71.51	28.49	39.49
83.03	16.97	46.74
100.00	0.00	59.70

DAY 2		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
22.36	77.64	10.85
44.55	55.45	23.16
58.05	41.95	33.07
72.66	27.34	41.22
79.95	20.05	45.02
100.00	0.00	58.59

DAY 3		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
23.48	76.52	12.09
30.41	69.59	16.36
40.64	59.36	22.40
48.49	51.51	26.62
58.99	41.01	34.22
71.17	28.83	40.80
79.31	20.69	45.91
100.00	0.00	58.79

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-309-1	58.54	309-1	42.65	75.13	71.44
	PTT-309-2	61.04	309-2	42.21	74.35	70.70
	PTT-309-3	59.53	309-3	41.79	73.61	70.00
	average	59.70		42.22	74.36	70.71
	confidence limit	3.13		0.68	1.88	1.79
	SD	1.26		0.43	0.76	0.72
	CV %	2.11		1.02	1.02	1.02
Day 2	PTT-309-1	57.69	309-1	44.06	77.94	75.20
	PTT-309-2	61.05	309-2	44.26	78.25	75.55
	PTT-309-3	58.22	309-3	45.50	80.19	77.66
	PTT-309-4	57.39	309-4	44.40	78.47	75.78
	average	58.59		44.56	78.71	76.05
	confidence limit	2.67		1.03	1.61	1.75
	SD	1.68		0.65	1.01	1.10
	CV %	2.86		1.45	1.29	1.45
Day 3	PTT-309-1	58.41	309-1	44.53	77.47	75.74
	PTT-309-2	58.41	309-2	44.64	77.66	75.93
	PTT-309-3	59.99	309-3	44.50	77.42	75.69
	PTT-309-4	58.35	309-4	44.61	77.61	75.88
	average	58.79		44.57	77.54	75.81
	confidence limit	1.27		0.10	0.18	0.18
	SD	0.80		0.07	0.11	0.11
	CV %	1.36		0.15	0.15	0.15

DAY 1		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
24.32	75.68	12.89
39.25	60.75	20.76
47.44	52.56	27.41
64.81	35.19	38.40
78.21	21.79	47.80
100.00	0.00	59.87

DAY 2		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
20.45	79.55	11.01
38.11	61.89	21.33
61.15	38.85	35.13
73.83	26.17	44.27
100.00	0.00	58.70

DAY 3		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
26.52	73.48	14.33
34.99	65.01	19.90
46.77	53.23	26.22
59.14	40.86	34.43
70.72	29.28	42.68
77.24	22.76	45.97
100.00	0.00	59.79

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	**PTT-311-1**	61.43	**311-1**	43.68	73.68	72.95
	PTT-311-2	59.85	**311-2**	40.53	68.37	67.69
	PTT-311-3	58.34	**311-3**	43.88	74.02	73.29
	average	**59.87**		**42.70**	**72.03**	**71.31**
	confidence limit	**3.84**		**4.67**	**7.87**	**7.80**
	SD	**1.55**		**1.88**	**3.17**	**3.14**
	CV %	**2.58**		**4.40**	**4.40**	**4.40**
Day 2	**PTT-311-1**	57.44	**311-1**	42.43	72.51	72.29
	PTT-311-2	59.23	**311-2**	42.87	73.26	73.04
	PTT-311-3	58.32	**311-3**	42.35	72.37	72.15
	PTT-311-4	59.79	**311-4**	42.81	73.15	72.94
	average	**58.70**		**42.62**	**72.82**	**72.60**
	confidence limit	**1.64**		**0.42**	**0.72**	**0.71**
	SD	**1.03**		**0.26**	**0.45**	**0.45**
	CV %	**1.76**		**0.62**	**0.62**	**0.62**
Day 3	**PTT-311-1**	59.75	**311-1**	42.88	72.57	71.71
	PTT-311-2	58.77	**311-2**	42.94	72.67	71.82
	PTT-311-3	59.96	**311-3**	42.87	72.55	71.70
	PTT-311-4	60.69	**311-4**	42.78	72.40	71.55
	average	**59.79**		**42.87**	**72.55**	**71.69**
	confidence limit	**1.26**		**0.11**	**0.18**	**0.18**
	SD	**0.79**		**0.07**	**0.11**	**0.11**
	CV %	**1.32**		**0.15**	**0.15**	**0.15**

DAY 1

PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
23.53	76.47	11.49
35.52	64.48	19.45
53.15	46.85	31.18
70.49	29.51	42.01
78.69	21.31	49.20
100.00	0.00	60.07

DAY 2

PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
21.29	78.71	10.93
34.38	65.62	18.09
51.45	48.55	28.13
62.42	37.58	34.53
73.51	26.49	42.49
100.00	0.00	57.02

DAY 3

PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
22.67	77.33	11.30
32.70	67.30	16.94
45.22	54.78	24.37
54.33	45.67	29.02
73.38	26.62	41.03
79.39	20.61	45.28
100.00	0.00	56.85

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-312-1	59.90	312-1	38.34	64.00	63.82
	PTT-312-2	60.74	312-2	37.61	62.78	62.61
	PTT-312-3	59.58	312-3	39.25	65.51	65.34
	average	60.07		38.40	64.10	63.92
	confidence limit	1.49		2.04	3.41	3.40
	SD	0.60		0.82	1.37	1.37
	CV %	1.00		2.14	2.14	2.14
Day 2	PTT-312-1	57.08	312-1	37.31	66.23	65.43
	PTT-312-2	57.74	312-2	36.66	65.08	64.29
	PTT-312-3	56.25	312-3	34.75	61.69	60.94
	PTT-312-4		312-4	37.89	67.26	66.45
	average	57.02		36.65	65.07	64.28
	confidence limit	1.19		2.17	3.85	3.81
	SD	0.75		1.36	2.42	2.39
	CV %	1.31		3.72	3.72	3.72
Day 3	PTT-312-1	56.94	312-1	35.82	64.17	63.01
	PTT-312-2	56.65	312-2	35.79	64.12	62.96
	PTT-312-3	57.81	312-3	36.26	64.96	63.78
	PTT-312-4	55.99	312-4	35.67	63.90	62.75
	average	56.85		35.89	64.29	63.13
	confidence limit	1.20		0.41	0.74	0.72
	SD	0.75		0.26	0.46	0.45
	CV %	1.33		0.72	0.72	0.72

LINEAR FIXED RANGE INTEGRATION

Quantification based on PTT peak integration

DAY 1

PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
21.45	78.55	12.16
36.75	63.25	21.05
51.87	48.13	30.37
68.98	31.02	40.01
84.77	15.23	49.71
100.00	0.00	60.21

DAY 2

PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
18.32	81.68	7.78
30.27	69.73	15.79
54.98	45.02	30.71
80.27	19.73	46.81
100.00	0.00	57.14

DAY 3

PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
24.84	75.16	11.59
29.71	70.29	14.73
44.47	55.53	24.97
54.52	45.48	31.69
61.68	38.32	34.12
75.74	24.26	45.28
86.49	13.51	48.39
100.00	0.00	61.85

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	**PTT-302-1**	59.20	**302-1**	44.27	75.02	73.52
	PTT-302-2	60.94	**302-2**	44.99	76.24	74.72
	PTT-302-3	60.50	**302-3**	44.03	74.61	73.12
	average	**60.21**		**44.43**	**75.29**	**73.79**
	confidence limit	**2.25**		**1.24**	**2.10**	**2.06**
	SD	**0.90**		**0.50**	**0.85**	**0.83**
	CV %	**1.50**		**1.12**	**1.12**	**1.12**
Day 2	**PTT-302-1**	60.10	**302-1**	44.35	78.00	77.62
	PTT-302-2	48.93	**302-2**	44.98	79.11	78.72
	PTT-302-3	60.66	**302-3**	44.47	78.21	77.83
	PTT-302-4	58.86	**302-4**	44.98	79.11	78.72
	average	**57.14**		**44.70**	**78.61**	**78.22**
	confidence limit	**8.79**		**0.53**	**0.93**	**0.93**
	SD	**5.52**		**0.33**	**0.59**	**0.58**
	CV %	**9.67**		**0.74**	**0.74**	**0.74**
Day 3	**PTT-302-1**	62.17	**302-1**	47.07	80.60	76.11
	PTT-302-2	61.47	**302-2**	45.13	77.73	72.97
	PTT-302-3	61.72	**302-3**	46.06	79.11	74.47
	PTT-302-4	62.03	**302-4**	45.80	78.73	74.05
	average	**61.85**		**46.02**	**79.04**	**74.40**
	confidence limit	**0.50**		**1.28**	**1.89**	**2.07**
	SD	**0.31**		**0.81**	**1.19**	**1.30**
	CV %	**0.51**		**1.75**	**1.50**	**1.75**

DAY 1		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
19.95	80.05	10.89
30.53	69.47	16.59
47.34	52.66	26.51
71.51	28.49	39.75
83.03	16.97	48.03
100.00	0.00	60.72

DAY 2		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
22.36	77.64	13.16
44.55	55.45	26.39
58.05	41.95	35.66
72.66	27.34	43.92
100.00	0.00	60.48

DAY 3		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
23.48	76.52	14.48
30.41	69.59	18.98
40.64	59.36	24.88
48.49	51.51	29.66
58.99	41.01	36.15
71.17	28.83	43.23
79.31	20.69	48.39
100.00	0.00	60.69

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-309-1	59.45	309-1	43.56	74.87	71.74
	PTT-309-2	61.50	309-2	43.70	75.11	71.97
	PTT-309-3	61.22	309-3	42.15	72.45	69.41
	average	60.72		43.14	74.14	71.04
	confidence limit	2.76		1.36	3.66	3.51
	SD	1.11		0.86	1.47	1.41
	CV %	1.83		1.99	1.99	1.99
Day 2	PTT-309-1	59.15	309-1	45.91	75.92	75.92
	PTT-309-2	62.21	309-2	46.30	76.57	76.56
	PTT-309-3	60.66	309-3	46.47	76.85	76.84
	PTT-309-4	59.88	309-4	46.96	77.66	77.65
	average	60.48		46.41	76.75	76.74
	confidence limit	2.09		0.69	1.15	1.15
	SD	1.31		0.44	0.72	0.72
	CV %	2.17		0.94	0.94	0.94
Day 3	PTT-309-1	59.98	309-1	45.67	74.91	75.26
	PTT-309-2	61.82	309-2	47.46	77.84	78.21
	PTT-309-3	60.70	309-3	47.17	77.37	77.73
	PTT-309-4	60.24	309-4	45.48	74.59	74.94
	average	60.69		46.45	76.18	76.53
	confidence limit	1.29		1.61	2.65	2.66
	SD	0.81		1.01	1.66	1.67
	CV %	1.34		2.18	2.18	2.18

DAY 1		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
24.32	75.68	14.04
39.25	60.75	23.08
47.44	52.56	28.97
64.81	35.19	40.20
78.21	21.79	50.09
100.00	0.00	61.86

DAY 2		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
20.45	79.55	12.19
38.11	61.89	22.74
61.15	38.85	36.96
73.83	26.17	45.72
100.00	0.00	60.05

DAY 3		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
26.52	73.48	15.57
34.99	65.01	21.39
46.77	53.23	27.84
59.14	40.86	36.47
70.72	29.28	43.71
77.24	22.76	47.72
88.31	11.69	51.78
100.00	0.00	61.76

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-311-1	63.23	311-1	45.29	72.97	73.21
	PTT-311-2	62.32	311-2	41.75	67.26	67.49
	PTT-311-3	60.03	311-3	44.89	72.32	72.57
	average	61.86		43.98	70.85	71.09
	confidence limit	4.10		4.82	7.76	7.79
	SD	1.65		1.94	3.12	3.13
	CV %	2.67		4.41	4.41	4.41
Day 2	PTT-311-1	58.60	311-1	44.14	72.87	73.50
	PTT-311-2	61.31	311-2	44.46	73.40	74.04
	PTT-311-3	60.44	311-3	43.91	72.49	73.12
	PTT-311-4	59.86	311-4	44.35	73.22	73.85
	average	60.05		44.22	73.00	73.63
	confidence limit	1.81		0.39	0.64	0.64
	SD	1.14		0.24	0.40	0.40
	CV %	1.89		0.55	0.55	0.55
Day 3	PTT-311-1	62.46	311-1	44.52	73.13	72.09
	PTT-311-2	60.56	311-2	44.58	73.23	72.19
	PTT-311-3	61.78	311-3	44.61	73.28	72.23
	PTT-311-4	62.23	311-4	44.55	73.18	72.14
	average	61.76		44.57	73.20	72.16
	confidence limit	1.35		0.06	0.10	0.10
	SD	0.85		0.04	0.06	0.06
	CV %	1.37		0.09	0.09	0.09

DAY 1		
PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
23.53	76.47	12.92
35.52	64.48	20.98
53.15	46.85	33.18
70.49	29.51	44.34
78.69	21.31	50.55
100.00	0.00	61.31

DAY 2		
PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
21.29	78.71	12.08
34.38	65.62	19.36
51.45	48.55	30.08
62.42	37.58	36.04
73.51	26.49	43.84
100.00	0.00	57.88

DAY 3		
PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
22.67	77.33	12.62
32.70	67.30	18.40
45.22	54.78	26.05
54.33	45.67	30.35
73.38	26.62	44.53
79.39	20.61	46.93
100.00	0.00	57.87

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-312-1	61.13	312-1	40.14	64.54	65.47
	PTT-312-2	62.39	312-2	39.71	63.85	64.77
	PTT-312-3	60.41	312-3	40.39	64.95	65.88
	average	61.31		40.08	64.45	65.37
	confidence limit	2.49		0.85	1.37	1.39
	SD	1.00		0.34	0.55	0.56
	CV %	1.63		0.86	0.86	0.86
Day 2	PTT-312-1	57.54	312-1	39.00	66.99	67.38
	PTT-312-2	59.05	312-2	38.34	65.85	66.24
	PTT-312-3	57.04	312-3	36.49	62.68	63.05
	PTT-312-4		312-4	39.75	68.28	68.68
	average	57.88		38.40	65.95	66.34
	confidence limit	2.60		2.22	3.81	3.83
	SD	1.05		1.39	2.40	2.41
	CV %	1.81		3.63	3.63	3.63
Day 3	PTT-312-1	58.51	312-1	37.46	64.20	64.74
	PTT-312-2	57.42	312-2	37.46	64.20	64.74
	PTT-312-3	59.06	312-3	37.47	64.22	64.75
	PTT-312-4	56.47	312-4	37.27	63.87	64.41
	average	57.87		37.42	64.12	64.66
	confidence limit	1.83		0.15	0.26	0.27
	SD	1.15		0.10	0.17	0.17
	CV %	1.99		0.26	0.26	0.26

Quantification based on PTT peak integration- DuPont's results

DAY 1			DAY 2			DAY 3		
PTT 302 %	PET 302 %	PTT enthalpy J/g	PTT 302 %	PET 302 %	PTT enthalpy J/g	PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00	0.00	100.00	0.00	0.00	100.00	0.00
9.08	90.92	4.33	27.66	72.34	12.65	20.00	80.00	5.53
15.82	84.18	1.55	37.21	62.79	18.84	33.87	66.13	15.42
30.90	69.10	11.60	53.85	46.15	30.29	51.26	48.74	28.22
54.21	45.79	29.30	67.19	32.81	37.51	69.02	30.98	40.57
70.48	29.52	41.10	83.85	16.15	48.81	87.71	12.29	52.48
80.43	19.57	49.50	100.00	0.00	61.05	100.00	0.00	62.45
100.00	0.00	63.04						

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-302-1	63.04	302-1	46.58	79.47	73.89
			302-2	47.26	80.29	74.97
			302-3	45.32	77.94	71.89
	average	63.04		46.39	79.23	73.58
	confidence limit			2.45	2.97	3.88
	SD			0.98	1.20	1.56
	CV %			2.12	1.51	2.12
Day 2	PTT-302-1	59.86	302-1	46.33	79.72	75.89
	PTT-302-2	60.71	302-2	43.91	76.28	71.92
	PTT-302-3	61.62	302-3	42.84	74.74	70.17
	PTT-302-4	62.01	302-4	45.18	78.09	74.00
	average	61.05		44.57	77.21	73.00
	confidence limit	1.53		2.41	3.44	3.95
	SD	0.96		1.52	2.16	2.48
	CV %	1.58		3.40	2.80	3.40
Day 3	PTT-302-1	62.69	302-1	46.83	79.41	74.98
	PTT-302-2	62.62	302-2	46.65	79.18	74.70
	PTT-302-3	62.74	302-3	46.34	78.79	74.20
	PTT-302-4	61.76	302-4	46.43	78.90	74.34
	average	62.45		46.56	79.07	74.56
	confidence limit	0.74		0.35	0.45	0.56
	SD	0.46		0.22	0.28	0.35
	CV %	0.74		0.47	0.35	0.47

DAY 1		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
12.73	87.27	4.24
24.32	75.68	11.67
36.54	63.46	20.75
49.02	50.98	29.30
76.32	23.68	46.84
85.59	14.41	55.77
100	0	64.44

DAY 2		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
13.45	86.55	6.29
38.87	61.13	22.89
48.36	51.64	27.72
68.00	32.00	41.52
81.82	18.18	49.18
100	0.00	61.31

DAY 3		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
23.12	76.88	11.41
35.65	64.35	19.41
54.00	46.00	30.14
67.03	32.97	37.18
100.00	0.00	60.30

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	**PTT-309-1**	64.44	**309-1**	49.37	79.11	76.61
			309-2	49.30	79.02	76.51
			309-3	48.81	78.36	75.74
	average	**64.44**		**49.16**	**78.83**	**76.29**
	confidence limit			**0.49**	**1.01**	**1.18**
	SD			**0.31**	**0.41**	**0.47**
	CV %			**0.62**	**0.52**	**0.62**
Day 2	**PTT-309-1**	60.74	**309-1**	47.94	79.46	78.19
	PTT-309-2	61.77	**309-2**	47.12	78.10	76.86
	PTT-309-3	61.05	**309-3**	47.38	78.53	77.28
	PTT-309-4	61.68	**309-4**	47.83	79.28	78.01
	average	**61.31**		**47.57**	**78.85**	**77.59**
	confidence limit	**0.79**		**0.61**	**1.01**	**1.00**
	SD	**0.50**		**0.38**	**0.64**	**0.63**
	CV %	**0.81**		**0.81**	**0.81**	**0.81**
Day 3	**PTT-309-1**	58.89	**309-1**	46.65	80.72	77.37
	PTT-309-2	61.93	**309-2**	46.57	80.60	77.24
	PTT-309-3	60.40	**309-3**	45.68	79.27	75.76
	PTT-309-4	59.96	**309-4**	46.32	80.23	76.82
	average	**60.30**		**46.31**	**80.21**	**76.80**
	confidence limit	**2.01**		**0.70**	**1.05**	**1.16**
	SD	**1.26**		**0.44**	**0.66**	**0.73**
	CV %	**2.09**		**0.95**	**0.82**	**0.95**

DAY 1		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
12.77	87.23	5.11
25.03	74.97	13.89
36.99	63.01	20.84
59.13	40.87	36.24
76.76	23.24	50.24
88.59	11.41	59.43
100	0.00	64.88

DAY 2		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
16.97	83.03	9.10
26.60	73.40	15.73
51.79	48.21	31.64
64.29	35.71	39.58
78.47	21.53	49.65
100.00	0.00	62.52

DAY 3		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
16.00	84.00	7.68
27.41	72.59	15.03
53.16	46.84	31.26
76.24	23.76	43.20
87.32	12.68	52.91
100.00	0.00	59.30

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	**PTT-311-1**	64.88	**311-1**	45.33	71.77	69.87
			311-2	46.40	73.23	71.52
			311-3	45.69	72.26	70.42
	average	**64.88**		**45.81**	**72.42**	**70.60**
	confidence limit			**1.35**	**1.84**	**2.08**
	SD			**0.54**	**0.74**	**0.84**
	CV %			**1.19**	**1.02**	**1.19**
Day 2	**PTT-311-1**	62.72	**311-1**	44.97	72.32	71.93
	PTT-311-2	62.08	**311-2**	44.52	71.60	71.21
	PTT-311-3	62.67	**311-3**	43.89	70.59	70.21
	PTT-311-4	62.59	**311-4**	44.71	71.90	71.52
	average	**62.52**		**44.52**	**71.60**	**71.22**
	confidence limit	**0.47**		**0.73**	**1.18**	**1.17**
	SD	**0.29**		**0.46**	**0.74**	**0.74**
	CV %	**0.47**		**1.03**	**1.03**	**1.03**
Day 3	**PTT-311-1**	58.50	**311-1**	41.69	70.88	70.30
	PTT-311-2	59.09	**311-2**	40.90	69.53	68.97
	PTT-311-3	59.13	**311-3**	41.65	70.81	70.23
	PTT-311-4	60.49	**311-4**	44.84	76.23	75.61
	average	**59.30**		**42.27**	**71.86**	**71.28**
	confidence limit	**1.34**		**2.79**	**4.74**	**4.70**
	SD	**0.84**		**1.75**	**2.98**	**2.95**
	CV %	**1.42**		**4.14**	**4.14**	**4.14**

DAY 1

PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
10.71	89.29	4.64
25.70	74.30	14.28
42.24	57.76	25.60
60.78	39.22	37.08
76.71	23.29	48.61
87.97	12.03	56.06
100.00	0.00	63.42

DAY 2

PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
16.30	83.70	8.46
28.19	71.81	15.90
52.79	47.21	29.52
77.34	22.66	46.98
87.19	12.81	52.22
100.00	0.00	61.64

DAY 3

PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
20.24	79.76	10.83
40.57	59.43	23.13
50.51	49.49	30.45
66.67	33.33	39.23
86.41	13.59	53.81
100.00	0.00	61.28

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-312-1	63.42	312-1	40.79	64.98	64.32
			312-2	40.35	64.28	63.62
			312-3	40.55	64.60	63.94
	average	63.42		40.56	64.62	63.96
	confidence limit			0.55	0.87	0.86
	SD			0.22	0.35	0.35
	CV %			0.54	0.54	0.54
Day 2	PTT-312-1	61.68	312-1	38.25	63.61	62.05
	PTT-312-2	61.61	312-2	38.71	64.38	62.80
	PTT-312-3	61.85	312-3	38.62	64.23	62.65
	PTT-312-4	61.43	312-4	39.00	64.86	63.27
	average	61.64		38.65	64.27	62.69
	confidence limit	0.28		0.49	0.82	0.80
	SD	0.17		0.31	0.51	0.50
	CV %	0.28		0.80	0.80	0.80
Day 3	PTT-312-1	60.99	312-1	38.61	63.64	63.01
	PTT-312-2	61.09	312-2	39.09	64.43	63.79
	PTT-312-3	61.43	312-3	37.86	62.40	61.78
	PTT-312-4	61.60	312-4	38.94	64.18	63.55
	average	61.28		38.63	63.66	63.03
	confidence limit	0.45		0.87	1.44	1.42
	SD	0.29		0.55	0.90	0.89
	CV %	0.47		1.42	1.42	1.42

SIGMOIDAL FIXED RANGE INTEGRATION

Quantification based on PTT peak integration

DAY 1

PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
21.45	78.55	12.90
36.75	63.25	21.86
51.87	48.13	30.80
68.98	31.02	40.30
84.77	15.23	49.41
100.00	0.00	59.96

DAY 2

PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
18.32	81.68	10.82
30.27	69.73	18.56
54.98	45.02	33.07
80.27	19.73	48.75
100.00	0.00	60.46

DAY 3

PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
24.84	75.16	14.74
29.71	70.29	17.85
44.47	55.53	27.08
54.52	45.48	34.04
61.68	38.32	36.24
75.74	24.26	46.81
86.49	13.51	49.70
100.00	0.00	62.97

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-302-1	58.98	302-1	44.27	74.84	73.83
	PTT-302-2	60.86	302-2	45.21	76.43	75.40
	PTT-302-3	60.05	302-3	44.42	75.10	74.08
	average	59.96		44.63	75.46	74.43
	confidence limit	2.34		1.25	2.12	2.09
	SD	0.94		0.51	0.85	0.84
	CV %	1.57		1.13	1.13	1.13
Day 2	PTT-302-1	60.80	302-1	46.19	76.33	76.40
	PTT-302-2	59.84	302-2	46.64	77.08	77.15
	PTT-302-3	61.39	302-3	46.38	76.65	76.72
	PTT-302-4	59.80	302-4	46.86	77.44	77.51
	average	60.46		46.52	76.88	76.94
	confidence limit	1.23		0.47	0.77	0.77
	SD	0.77		0.29	0.49	0.49
	CV %	1.28		0.63	0.63	0.63
Day 3	PTT-302-1	63.07	302-1	45.22	74.88	71.82
	PTT-302-2	62.90	302-2	47.96	79.30	76.17
	PTT-302-3	62.78	302-3	47.92	79.24	76.11
	PTT-302-4	63.11	302-4	47.76	78.98	75.85
	average	62.97		47.22	78.10	74.99
	confidence limit	0.24		2.12	3.42	3.37
	SD	0.15		1.33	2.15	2.12
	CV %	0.24		2.82	2.75	2.82

DAY 1

PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
19.95	80.05	11.89
30.53	69.47	17.69
47.34	52.66	27.61
71.51	28.49	39.26
83.03	16.97	48.73
100.00	0.00	61.28

DAY 2

PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
22.36	77.64	13.16
44.55	55.45	26.39
58.05	41.95	35.66
72.66	27.34	43.92
100.00	0.00	60.48

DAY 3

PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
23.48	76.52	14.48
30.41	69.59	18.98
40.64	59.36	24.88
48.49	51.51	29.66
58.99	41.01	36.15
71.17	28.83	43.23
79.31	20.69	48.39
100.00	0.00	60.69

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-309-1	60.12	309-1	43.87	74.48	71.59
	PTT-309-2	61.79	309-2	43.71	74.21	71.33
	PTT-309-3	61.92	309-3	42.24	71.71	68.93
	average	61.28		43.27	73.47	70.62
	confidence limit	2.49		1.43	3.79	3.64
	SD	1.00		0.90	1.53	1.47
	CV %	1.64		2.08	2.08	2.08
Day 2	PTT-309-1	59.15	309-1	45.91	75.92	75.92
	PTT-309-2	62.21	309-2	46.30	76.57	76.56
	PTT-309-3	60.66	309-3	46.47	76.85	76.84
	PTT-309-4	59.88	309-4	46.96	77.66	77.65
	average	60.48		46.41	76.75	76.74
	confidence limit	2.09		0.69	1.15	1.15
	SD	1.31		0.44	0.72	0.72
	CV %	2.17		0.94	0.94	0.94
Day 3	PTT-309-1	59.98	309-1	45.67	74.91	75.26
	PTT-309-2	61.82	309-2	47.46	77.84	78.21
	PTT-309-3	60.70	309-3	47.17	77.37	77.73
	PTT-309-4	60.24	309-4	45.48	74.59	74.94
	average	60.69		46.45	76.18	76.53
	confidence limit	1.29		1.61	2.65	2.66
	SD	0.81		1.01	1.66	1.67
	CV %	1.34		2.18	2.18	2.18

DAY 1		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
24.32	75.68	15.25
39.25	60.75	25.36
47.44	52.56	30.18
64.81	35.19	41.73
78.21	21.79	51.11
100.00	0.00	63.08

DAY 2		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
20.45	79.55	13.41
38.11	61.89	23.83
61.15	38.85	38.19
73.83	26.17	46.57
100.00	0.00	61.16

DAY 3		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
26.52	73.48	16.83
34.99	65.01	22.55
46.77	53.23	29.25
59.14	40.86	37.82
70.72	29.28	45.71
77.24	22.76	48.57
88.31	11.69	52.62
100.00	0.00	62.82

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-311-1	64.38	311-1	46.21	72.20	73.26
	PTT-311-2	63.89	311-2	42.55	66.48	67.45
	PTT-311-3	60.97	311-3	45.65	71.33	72.37
	average	63.08		44.80	70.01	71.03
	confidence limit	4.58		4.90	7.65	7.76
	SD	1.84		1.97	3.08	3.13
	CV %	2.92		4.40	4.40	4.40
Day 2	PTT-311-1	59.31	311-1	45.29	72.97	74.06
	PTT-311-2	62.27	311-2	45.43	73.19	74.29
	PTT-311-3	61.81	311-3	44.94	72.40	73.49
	PTT-311-4	61.23	311-4	45.22	72.85	73.94
	average	61.16		45.22	72.85	73.94
	confidence limit	2.07		0.33	0.53	0.54
	SD	1.30		0.21	0.33	0.34
	CV %	2.13		0.46	0.46	0.46
Day 3	PTT-311-1	63.73	311-1	45.54	72.81	72.49
	PTT-311-2	61.57	311-2	45.70	73.06	72.74
	PTT-311-3	62.88	311-3	45.77	73.17	72.86
	PTT-311-4	63.11	311-4	45.68	73.03	72.71
	average	62.82		45.67	73.02	72.70
	confidence limit	1.45		0.15	0.25	0.24
	SD	0.91		0.10	0.15	0.15
	CV %	1.45		0.21	0.21	0.21

DAY 1		
PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
23.53	76.47	14.14
35.52	64.48	22.41
53.15	46.85	34.50
70.49	29.51	45.95
78.69	21.31	51.21
100.00	0.00	62.30

DAY 2		
PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
21.29	78.71	13.09
34.38	65.62	20.35
51.45	48.55	31.11
62.42	37.58	36.77
73.51	26.49	44.56
100.00	0.00	58.40

DAY 3		
PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
22.67	77.33	13.62
32.70	67.30	19.62
45.22	54.78	27.05
54.33	45.67	31.04
73.38	26.62	45.28
79.39	20.61	47.72
100.00	0.00	58.42

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	**PTT-312-1**	62.47	**312-1**	41.11	64.44	65.99
	PTT-312-2	63.47	**312-2**	41.05	64.34	65.89
	PTT-312-3	60.95	**312-3**	41.00	64.26	65.81
	average	**62.30**		**41.05**	**64.35**	**65.90**
	confidence limit	**3.15**		**0.14**	**0.21**	**0.22**
	SD	**1.27**		**0.06**	**0.09**	**0.09**
	CV %	**2.04**		**0.13**	**0.13**	**0.13**
Day 2	**PTT-312-1**	57.99	**312-1**	40.05	67.52	68.57
	PTT-312-2	59.81	**312-2**	39.54	66.66	67.70
	PTT-312-3	57.41	**312-3**	37.62	63.42	64.41
	PTT-312-4		**312-4**	40.84	68.85	69.93
	average	**58.40**		**39.51**	**66.61**	**67.65**
	confidence limit	**1.99**		**2.18**	**3.68**	**3.73**
	SD	**1.25**		**1.37**	**2.31**	**2.35**
	CV %	**2.14**		**3.47**	**3.47**	**3.47**
Day 3	**PTT-312-1**	59.31	**312-1**	38.45	64.64	65.82
	PTT-312-2	57.82	**312-2**	38.48	64.69	65.87
	PTT-312-3	59.72	**312-3**	38.60	64.90	66.07
	PTT-312-4	56.83	**312-4**	38.29	64.37	65.54
	average	**58.42**		**38.46**	**64.65**	**65.83**
	confidence limit	**2.13**		**0.20**	**0.34**	**0.35**
	SD	**1.34**		**0.13**	**0.21**	**0.22**
	CV %	**2.29**		**0.33**	**0.33**	**0.33**

Quantification based on PTT peak integration- DuPont's results

DAY 1 PTT 302 %	DAY 1 PET 302 %	DAY 1 PTT enthalpy J/g	DAY 2 PTT 302 %	DAY 2 PET 302 %	DAY 2 PTT enthalpy J/g	DAY 3 PTT 302 %	DAY 3 PET 302 %	DAY 3 PTT enthalpy J/g
0.00	100.00	0.00	0.00	100.00	0.00	0.00	100.00	0.00
9.08	90.92	6.17	27.66	72.34	16.53	20.00	80.00	11.77
15.82	84.18	8.18	37.21	62.79	22.58	33.87	66.13	20.96
30.90	69.10	19.92	53.85	46.15	33.04	51.26	48.74	33.79
54.21	45.79	35.63	67.19	32.81	40.73	69.02	30.98	43.95
70.48	29.52	46.22	83.85	16.15	50.59	87.71	12.29	54.48
80.43	19.57	53.45	100.00	0.00	61.97	100.00	0.00	63.64
100.00	0.00	65.42						

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	**PTT-302-1**	65.42	**302-1**	51.19	78.05	78.25
			302-2	51.80	78.98	79.18
			302-3	50.79	77.44	77.64
	average	**65.42**		**51.26**	**78.15**	**78.36**
	confidence limit			**1.26**	**1.93**	**1.93**
	SD			**0.51**	**0.78**	**0.78**
	CV %			**0.99**	**0.99**	**0.99**
Day 2	**PTT-302-1**	60.19	**302-1**	48.80	79.86	78.75
	PTT-302-2	61.73	**302-2**	46.52	76.13	75.07
	PTT-302-3	62.69	**302-3**	46.16	75.54	74.49
	PTT-302-4	63.26	**302-4**	48.13	78.76	77.67
	average	**61.97**		**47.40**	**77.57**	**76.50**
	confidence limit	**2.14**		**2.01**	**3.30**	**3.25**
	SD	**1.34**		**1.27**	**2.07**	**2.04**
	CV %	**2.17**		**2.67**	**2.67**	**2.67**
Day 3	**PTT-302-1**	64.02	**302-1**	50.33	79.54	79.09
	PTT-302-2	63.52	**302-2**	50.31	79.50	79.06
	PTT-302-3	64.09	**302-3**	49.91	78.87	78.43
	PTT-302-4	62.92	**302-4**	50.15	79.25	78.81
	average	**63.64**		**50.18**	**79.29**	**78.85**
	confidence limit	**0.86**		**0.31**	**0.49**	**0.49**
	SD	**0.54**		**0.19**	**0.31**	**0.31**
	CV %	**0.85**		**0.39**	**0.39**	**0.39**

DAY 1		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
12.73	87.27	7.62
24.32	75.68	15.78
36.54	63.46	24.80
49.02	50.98	33.00
76.32	23.68	50.29
85.59	14.41	58.93
100	0.00	67.51

DAY 2		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
13.45	86.55	9.05
38.87	61.13	25.52
48.36	51.64	30.62
68.00	32.00	43.54
81.82	18.18	51.28
100	0.00	62.79

DAY 3		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
23.12	76.88	15.21
35.65	64.35	22.17
54.00	46.00	33.21
67.03	32.97	39.99
100.00	0.00	62.25

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	**PTT-309-1**	67.51	**309-1**	53.01	78.63	78.52
			309-2	53.08	78.73	78.63
			309-3	52.50	77.87	77.77
	average	**67.51**		**52.86**	**78.41**	**78.30**
	confidence limit			**0.50**	**1.17**	**1.16**
	SD			**0.32**	**0.47**	**0.47**
	CV %			**0.60**	**0.60**	**0.60**
Day 2	**PTT-309-1**	61.77	**309-1**	50.16	79.32	79.89
	PTT-309-2	63.69	**309-2**	49.45	78.19	78.76
	PTT-309-3	62.41	**309-3**	49.79	78.73	79.30
	PTT-309-4	63.28	**309-4**	50.24	79.44	80.02
	average	**62.79**		**49.91**	**78.92**	**79.49**
	confidence limit	**1.37**		**0.58**	**0.92**	**0.92**
	SD	**0.86**		**0.36**	**0.58**	**0.58**
	CV %	**1.37**		**0.73**	**0.73**	**0.73**
Day 3	**PTT-309-1**	63.02	**309-1**	50.89	82.59	81.76
	PTT-309-2	62.90	**309-2**	50.54	82.02	81.20
	PTT-309-3	63.58	**309-3**	49.72	80.69	79.88
	PTT-309-4	59.48	**309-4**	50.14	81.37	80.55
	average	**62.25**		**50.32**	**81.67**	**80.85**
	confidence limit	**2.97**		**0.80**	**1.30**	**1.29**
	SD	**1.87**		**0.51**	**0.82**	**0.81**
	CV %	**3.00**		**1.00**	**1.00**	**1.00**

DAY 1		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
12.77	87.23	9.50
25.03	74.97	18.70
36.99	63.01	25.97
59.13	40.87	40.61
76.76	23.24	54.50
88.59	11.41	64.06
100	0.00	69.22

DAY 2		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
16.97	83.03	11.59
26.60	73.40	17.57
51.79	48.21	33.66
64.29	35.71	41.47
78.47	21.53	51.05
100.00	0.00	63.67

DAY 3		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
16.00	84.00	10.58
27.41	72.59	17.46
53.16	46.84	33.39
76.24	23.76	46.07
87.32	12.68	54.50
100.00	0.00	60.85

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-311-1	69.22	311-1	50.14	71.09	72.44
			311-2	50.96	72.25	73.62
			311-3	50.76	71.97	73.33
	average	69.22		50.62	71.77	73.13
	confidence limit			1.06	1.51	1.53
	SD			0.43	0.61	0.62
	CV %			0.84	0.84	0.84
Day 2	PTT-311-1	63.97	311-1	47.08	73.06	73.94
	PTT-311-2	63.13	311-2	46.79	72.61	73.49
	PTT-311-3	63.71	311-3	46.10	71.54	72.40
	PTT-311-4	63.87	311-4	46.69	72.45	73.33
	average	63.67		46.67	72.42	73.29
	confidence limit	0.60		0.65	1.02	1.03
	SD	0.38		0.41	0.64	0.65
	CV %	0.59		0.88	0.88	0.88
Day 3	PTT-311-1	60.05	311-1	44.29	71.98	72.79
	PTT-311-2	60.14	311-2	43.44	70.60	71.39
	PTT-311-3	60.89	311-3	44.41	72.18	72.98
	PTT-311-4	62.32	311-4	47.15	76.63	77.49
	average	60.85		44.82	72.85	73.66
	confidence limit	1.67		2.56	4.17	4.21
	SD	1.05		1.61	2.62	2.65
	CV %	1.73		3.59	3.59	3.59

DAY 1 PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
10.71	89.29	7.20
25.70	74.30	17.30
42.24	57.76	28.20
60.78	39.22	39.59
76.71	23.29	50.65
87.97	12.03	57.67
100.00	0.00	65.40

DAY 2 PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
16.30	83.70	9.84
28.19	71.81	17.28
52.79	47.21	30.25
77.34	22.66	47.65
87.19	12.81	52.50
100.00	0.00	62.52

DAY 3 PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
20.24	79.76	12.69
40.57	59.43	24.51
50.51	49.49	32.16
66.67	33.33	40.27
86.41	13.59	54.70
100.00	0.00	62.16

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-312-1	65.40	312-1	43.76	66.65	66.91
	PTT-312-2		312-2	43.28	65.92	66.18
	PTT-312-3		312-3	43.49	66.24	66.50
	average	65.40		43.51	66.27	66.53
	confidence limit			0.60	0.91	0.91
	SD			0.24	0.37	0.37
	CV %			0.55	0.55	0.55
Day 2	PTT-312-1	62.78	312-1	39.77	65.09	63.61
	PTT-312-2	62.45	312-2	40.07	65.58	64.09
	PTT-312-3	62.52	312-3	40.58	66.42	64.90
	PTT-312-4	62.34	312-4	40.90	66.94	65.42
	average	62.52		40.33	66.01	64.50
	confidence limit	0.30		0.81	1.32	1.29
	SD	0.19		0.51	0.83	0.81
	CV %	0.30		1.26	1.26	1.26
Day 3	PTT-312-1	62.13	312-1	40.33	64.81	64.89
	PTT-312-2	61.55	312-2	40.57	65.19	65.27
	PTT-312-3	62.38	312-3	40.31	64.78	64.85
	PTT-312-4	62.56	312-4	40.53	65.13	65.21
	average	62.16		40.44	64.98	65.06
	confidence limit	0.70		0.21	0.34	0.34
	SD	0.44		0.13	0.22	0.22
	CV %	0.71		0.33	0.33	0.33

PERPENDICULAR DROP FIXED RANGE INTEGRATION

Quantification based on PTT peak integration

DAY 1		
PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
21.45	78.55	15.31
36.75	63.25	24.59
51.87	48.13	32.46
68.98	31.02	41.40
84.77	15.23	49.91
100.00	0.00	60.21

DAY 2		
PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
18.32	81.68	13.78
30.27	69.73	21.31
54.98	45.02	34.41
80.27	19.73	48.65
100.00	0.00	57.14

DAY 3		
PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
29.71	70.29	20.51
44.47	55.53	28.86
54.52	45.48	35.82
61.68	38.32	37.74
75.74	24.26	47.66
86.49	13.51	49.76
100.00	0.00	61.85

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-302-1	59.20	302-1	45.08	74.40	74.87
	PTT-302-2	60.94	302-2	45.77	75.54	76.01
	PTT-302-3	60.50	302-3	44.99	74.25	74.72
	average	60.21		45.28	74.73	75.20
	confidence limit	2.25		1.06	1.75	1.76
	SD	0.90		0.43	0.70	0.71
	CV %	1.50		0.94	0.94	0.94
Day 2	PTT-302-1	60.10	302-1	46.61	76.78	81.58
	PTT-302-2	48.93	302-2	47.02	77.64	82.29
	PTT-302-3	60.66	302-3	46.65	76.86	81.65
	PTT-302-4	58.86	302-4	47.10	77.81	82.43
	average	57.14		46.85	77.27	81.99
	confidence limit	8.79		0.40	0.84	0.70
	SD	5.52		0.25	0.53	0.44
	CV %	9.67		0.54	0.68	0.54
Day 3	PTT-302-1	62.17	302-1	47.62	76.68	77.00
	PTT-302-2	61.47	302-2	47.74	76.89	77.19
	PTT-302-3	61.72	302-3	48.53	78.31	78.47
	PTT-302-4	62.03	302-4	48.39	78.05	78.24
	average	61.85		48.07	77.48	77.72
	confidence limit	0.50		0.73	1.30	1.17
	SD	0.31		0.46	0.82	0.74
	CV %	0.51		0.95	1.06	0.95

DAY 1 PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
19.95	80.05	12.16
30.53	69.47	18.11
47.34	52.66	27.73
71.51	28.49	38.90
83.03	16.97	48.51
100.00	0.00	60.72

DAY 2 PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
22.36	77.64	13.32
44.55	55.45	26.47
58.05	41.95	35.54
72.66	27.34	43.59
100.00	0.00	60.09

DAY 3 PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
23.48	76.52	14.25
30.41	69.59	18.66
48.49	51.51	29.10
58.99	41.01	35.47
71.17	28.83	42.78
79.31	20.69	47.90
100.00	0.00	61.85

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-309-1	59.45	309-1	46.25	78.92	76.17
	PTT-309-2	61.50	309-2	45.71	78.00	75.28
	PTT-309-3	61.22	309-3	44.79	76.43	73.76
	average	60.72		45.58	77.79	75.07
	confidence limit	2.76		1.17	3.13	3.02
	SD	1.11		0.74	1.26	1.22
	CV %	1.83		1.62	1.62	1.62
Day 2	PTT-309-1	58.77	309-1	45.43	75.50	75.60
	PTT-309-2	62.02	309-2	45.87	76.23	76.34
	PTT-309-3	60.00	309-3	45.98	76.42	76.52
	PTT-309-4	59.57	309-4	46.48	77.25	77.35
	average	60.09		45.94	76.35	76.45
	confidence limit	2.20		0.69	1.14	1.14
	SD	1.38		0.43	0.72	0.72
	CV %	2.30		0.94	0.94	0.94
Day 3	PTT-309-1	62.17	309-1	45.97	75.58	74.33
	PTT-309-2	61.47	309-2	46.85	77.03	75.75
	PTT-309-3	61.72	309-3	46.55	76.54	75.27
	PTT-309-4	62.03	309-4	45.84	75.37	74.12
	average	61.85		46.30	76.13	74.87
	confidence limit	0.50		0.76	1.25	1.23
	SD	0.31		0.48	0.79	0.77
	CV %	0.51		1.03	1.03	1.03

DAY 1		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
24.32	75.68	15.76
39.25	60.75	26.23
47.44	52.56	30.34
64.81	35.19	42.00
78.21	21.79	50.64
100.00	0.00	61.86

DAY 2		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
20.45	79.55	13.54
38.11	61.89	23.30
61.15	38.85	38.05
73.83	26.17	46.18
100.00	0.00	60.05

DAY 3		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
46.77	53.23	28.98
59.14	40.86	37.53
70.72	29.28	45.18
77.24	22.76	48.14
88.31	11.69	52.13
100.00	0.00	61.76

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	**PTT-311-1**	63.23	**311-1**	46.59	73.20	75.32
	PTT-311-2	62.32	**311-2**	42.96	67.49	69.45
	PTT-311-3	60.03	**311-3**	45.89	72.10	74.18
	average	**61.86**		**45.15**	**70.93**	**72.98**
	confidence limit	**4.10**		**4.78**	**7.52**	**7.73**
	SD	**1.65**		**1.93**	**3.03**	**3.11**
	CV %	**2.67**		**4.27**	**4.27**	**4.27**
Day 2	**PTT-311-1**	58.60	**311-1**	44.74	73.01	74.50
	PTT-311-2	61.31	**311-2**	45.06	73.53	75.03
	PTT-311-3	60.44	**311-3**	44.51	72.63	74.12
	PTT-311-4	59.86	**311-4**	44.91	73.29	74.78
	average	**60.05**		**44.81**	**73.12**	**74.61**
	confidence limit	**1.81**		**0.38**	**0.61**	**0.63**
	SD	**1.14**		**0.24**	**0.39**	**0.39**
	CV %	**1.89**		**0.53**	**0.53**	**0.53**
Day 3	**PTT-311-1**	62.46	**311-1**	45.03	72.63	72.91
	PTT-311-2	60.56	**311-2**	45.05	72.67	72.95
	PTT-311-3	61.78	**311-3**	45.63	73.66	73.89
	PTT-311-4	62.23	**311-4**	45.51	73.45	73.69
	average	**61.76**		**45.31**	**73.10**	**73.36**
	confidence limit	**1.35**		**0.49**	**0.85**	**0.80**
	SD	**0.85**		**0.31**	**0.53**	**0.50**
	CV %	**1.37**		**0.68**	**0.73**	**0.68**

DAY 1			DAY 2			DAY 3		
PTT 312 %	PET 312 %	PTT enthalpy J/g	PTT 312 %	PET 312 %	PTT enthalpy J/g	PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00	0.00	100.00	0.00	0.00	100.00	0.00
23.53	76.47	14.58	21.29	78.71	13.67	22.67	77.33	14.12
35.52	64.48	22.81	34.38	65.62	20.85	45.22	54.78	27.37
53.15	46.85	34.56	51.45	48.55	31.54	54.33	45.67	31.38
70.49	29.51	45.84	62.42	37.58	37.02	73.38	26.62	45.68
78.69	21.31	51.54	73.51	26.49	44.90	79.39	20.61	48.16
100.00	0.00	61.31	100.00	0.00	57.88	100.00	0.00	57.87

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-312-1	61.13	312-1	41.09	64.62	67.02
	PTT-312-2	62.39	312-2	40.84	64.22	66.61
	PTT-312-3	60.41	312-3	41.32	64.98	67.40
	average	61.31		41.08	64.61	67.01
	confidence limit	2.49		0.60	0.94	0.97
	SD	1.00		0.24	0.38	0.39
	CV %	1.63		0.58	0.58	0.58
Day 2	PTT-312-1	57.54	312-1	40.17	67.52	69.41
	PTT-312-2	59.05	312-2	39.60	66.57	68.42
	PTT-312-3	57.04	312-3	37.34	62.77	64.52
	PTT-312-4		312-4	40.91	68.77	70.68
	average	57.88		39.51	66.41	68.26
	confidence limit	2.60		2.45	4.12	4.23
	SD	1.05		1.54	2.59	2.66
	CV %	1.81		3.90	3.90	3.90
Day 3	PTT-312-1	58.51	312-1	38.53	63.62	66.59
	PTT-312-2	57.42	312-2	38.49	63.55	66.52
	PTT-312-3	59.06	312-3	38.66	63.84	66.81
	PTT-312-4	56.47	312-4	38.35	63.31	66.27
	average	57.87		38.51	63.58	66.55
	confidence limit	1.83		0.20	0.35	0.35
	SD	1.15		0.13	0.22	0.22
	CV %	1.99		0.33	0.35	0.33

Quantification based on PTT peak integration- DuPont's results

DAY 1

PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
9.08	90.92	9.34
15.82	84.18	13.21
30.90	69.10	22.89
54.21	45.79	36.44
70.48	29.52	46.06
80.43	19.57	52.80
100.00	0.00	63.29

DAY 2

PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
27.66	72.34	21.54
37.21	62.79	26.90
53.85	46.15	36.41
67.19	32.81	43.34
83.85	16.15	52.57
100.00	0.00	61.05

DAY 3

PTT 302 %	PET 302 %	PTT enthalpy J/g
0.00	100.00	0.00
20.00	80.00	16.73
33.87	66.13	25.03
51.26	48.74	36.13
69.02	30.98	45.64
87.71	12.29	55.67
100.00	0.00	62.45

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-302-1	63.29	302-1	49.80	75.70	78.69
			302-2	49.63	75.39	78.42
			302-3	49.45	75.06	78.13
	average	63.29		49.63	75.38	78.41
	confidence limit			0.43	0.80	0.69
	SD			0.18	0.32	0.28
	CV %			0.35	0.43	0.35
Day 2	PTT-302-1	59.86	302-1	49.90	77.41	81.74
	PTT-302-2	60.71	302-2	45.45	68.97	74.45
	PTT-302-3	61.62	302-3	47.14	72.13	77.22
	PTT-302-4	62.01	302-4	48.71	75.11	79.79
	average	61.05		47.80	73.41	78.30
	confidence limit	1.53		3.07	5.83	5.04
	SD	0.96		1.93	3.66	3.16
	CV %	1.58		4.04	4.99	4.04
Day 3	PTT-302-1	62.69	302-1	50.56	76.43	80.96
	PTT-302-2	62.62	302-2	50.24	75.82	80.45
	PTT-302-3	62.74	302-3	50.09	75.53	80.20
	PTT-302-4	61.76	302-4	50.16	75.67	80.32
	average	62.45		50.26	75.86	80.48
	confidence limit	0.74		0.33	0.63	0.53
	SD	0.46		0.21	0.40	0.33
	CV %	0.74		0.41	0.52	0.41

DAY 1		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
12.73	87.27	7.18
24.32	75.68	14.20
36.54	63.46	22.53
49.02	50.98	33.32
76.32	23.68	47.11
85.59	14.41	56.24
100.00	0.00	63.55

DAY 2		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
13.45	86.55	9.44
38.87	61.13	24.45
48.36	51.64	31.02
68.00	32.00	44.15
81.82	18.18	48.77
100	0.00	61.31

DAY 3		
PTT 309 %	PET 309 %	PTT enthalpy J/g
0.00	100.00	0.00
23.12	76.88	13.40
35.65	64.35	20.72
54.00	46.00	30.70
67.03	32.97	39.02
100.00	0.00	60.30

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	**PTT-309-1**	63.55	**309-1**	48.66	76.17	76.57
			309-2	48.85	76.47	76.87
			309-3	48.39	75.75	76.14
	average	**63.55**		**48.63**	**76.13**	**76.53**
	confidence limit			**0.37**	**0.90**	**0.90**
	SD			**0.23**	**0.36**	**0.36**
	CV %			**0.48**	**0.48**	**0.48**
Day 2	**PTT-309-1**	60.74	**309-1**	48.98	77.01	79.89
	PTT-309-2	61.77	**309-2**	48.14	75.64	78.52
	PTT-309-3	61.05	**309-3**	48.49	76.21	79.09
	PTT-309-4	61.68	**309-4**	49.42	77.73	80.61
	average	**61.31**		**48.76**	**76.65**	**79.53**
	confidence limit	**0.79**		**0.89**	**1.45**	**1.45**
	SD	**0.50**		**0.56**	**0.91**	**0.91**
	CV %	**0.81**		**1.15**	**1.19**	**1.15**
Day 3	**PTT-309-1**	58.89	**309-1**	48.37	81.87	80.22
	PTT-309-2	61.93	**309-2**	48.13	81.47	79.82
	PTT-309-3	60.40	**309-3**	47.27	80.01	78.40
	PTT-309-4	59.96	**309-4**	47.88	81.04	79.41
	average	**60.30**		**47.91**	**81.10**	**79.46**
	confidence limit	**2.01**		**0.75**	**1.27**	**1.25**
	SD	**1.26**		**0.47**	**0.80**	**0.78**
	CV %	**2.09**		**0.99**	**0.99**	**0.99**

DAY 1		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
12.77	87.23	7.90
25.03	74.97	15.71
36.99	63.01	22.44
59.13	40.87	36.70
76.76	23.24	50.01
88.59	11.41	58.67
100	0.00	63.98

DAY 2		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
16.97	83.03	12.51
26.60	73.40	17.12
51.79	48.21	33.64
64.29	35.71	41.70
78.47	21.53	51.23
100.00	0.00	62.52

DAY 3		
PTT 311 %	PET 311 %	PTT enthalpy J/g
0.00	100.00	0.00
16.00	84.00	11.04
27.41	72.59	18.25
53.16	46.84	33.46
76.24	23.76	43.41
87.32	12.68	54.50
100.00	0.00	59.30

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	PTT-311-1	63.68	311-1	45.61	70.82	71.29
			311-2	46.81	72.69	73.16
			311-3	46.11	71.60	72.07
	average	63.68		46.18	71.70	72.17
	confidence limit			1.50	2.33	2.34
	SD			0.60	0.94	0.94
	CV %			1.31	1.31	1.31
Day 2	PTT-311-1	62.72	311-1	46.68	72.84	74.67
	PTT-311-2	62.08	311-2	46.09	71.91	73.73
	PTT-311-3	62.67	311-3	44.87	70.01	71.77
	PTT-311-4	62.59	311-4	46.72	72.90	74.73
	average	62.52		46.09	71.91	73.73
	confidence limit	0.47		1.37	2.14	2.20
	SD	0.29		0.86	1.35	1.38
	CV %	0.47		1.87	1.87	1.87
Day 3	PTT-311-1	58.50	311-1	43.08	70.82	72.64
	PTT-311-2	59.09	311-2	42.21	69.26	71.18
	PTT-311-3	59.13	311-3	43.60	71.75	73.52
	PTT-311-4	60.49	311-4			
	average	59.30		42.96	70.61	72.45
	confidence limit	1.34		1.74	3.12	2.94
	SD	0.84		0.70	1.26	1.18
	CV %	1.42		1.63	1.78	1.63

DAY 1		
PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
10.71	89.29	8.33
25.70	74.30	17.18
42.24	57.76	28.51
60.78	39.22	39.73
76.71	23.29	51.11
87.97	12.03	59.64
100.00	0.00	68.46

DAY 2		
PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
16.30	83.70	11.16
28.19	71.81	18.69
52.79	47.21	31.62
77.34	22.66	48.97
87.19	12.81	53.80
100.00	0.00	61.64

DAY 3		
PTT 312 %	PET 312 %	PTT enthalpy J/g
0.00	100.00	0.00
20.24	79.76	14.00
40.57	59.43	26.55
50.51	49.49	33.31
66.67	33.33	41.81
86.41	13.59	56.16
100.00	0.00	61.28

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PTT % calibration	PTT % single point
Day 1	**PTT-312-1**	68.46	**312-1**	42.57	63.08	62.18
			312-2	42.35	62.75	61.86
			312-3	42.41	62.84	61.95
	average	**68.46**		**42.44**	**62.89**	**62.00**
	confidence limit			**0.28**	**0.42**	**0.41**
	SD			**0.11**	**0.17**	**0.17**
	CV %			**0.27**	**0.27**	**0.27**
Day 2	**PTT-312-1**	61.68	**312-1**	39.77	64.09	64.52
	PTT-312-2	61.61	**312-2**	40.07	64.58	65.00
	PTT-312-3	61.85	**312-3**	40.58	65.40	65.83
	PTT-312-4	61.43	**312-4**	40.90	65.91	66.35
	average	**61.64**		**40.33**	**65.00**	**65.43**
	confidence limit	**0.28**		**0.81**	**1.30**	**1.31**
	SD	**0.17**		**0.51**	**0.82**	**0.82**
	CV %	**0.28**		**1.26**	**1.26**	**1.26**
Day 3	**PTT-312-1**	60.99	**312-1**	40.70	63.10	66.42
	PTT-312-2	61.09	**312-2**	40.77	63.21	66.53
	PTT-312-3	61.43	**312-3**	40.44	62.67	65.99
	PTT-312-4	61.60	**312-4**	40.46	62.70	66.03
	average	**61.28**		**40.59**	**62.92**	**66.24**
	confidence limit	**0.45**		**0.27**	**0.44**	**0.43**
	SD	**0.29**		**0.17**	**0.28**	**0.27**
	CV %	**0.47**		**0.41**	**0.44**	**0.41**

Quantification of samples *via* calibration curve built with PTTs and PETs manually extracted from the samples to be quantified

6 days study

LINEAR INTEGRATION

Quantification based on PTT and PET peak integration

DAY 1

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
26.17	73.83	14.47	30.94
34.96	65.04	19.73	26.08
48.29	51.71	27.89	20.08
59.64	40.36	34.40	15.54
70.06	29.94	43.28	11.39
77.44	22.56	46.26	
100.00	0.00	63.87	0.00

DAY 2

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.95	69.05	16.79	27.48
53.82	46.18	30.94	17.63
57.08	42.92	32.74	14.68
70.87	29.13	40.71	10.3
75.88	24.12	45.83	9.06
79.95	20.05	48.79	7.33
100	0.00	61.98	0.00

DAY 3

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
35.24	64.76	21.86	29.44
52.65	47.35	33.80	20.00
62.90	37.10	41.11	15.37
71.13	28.87	46.33	12.27
73.90	26.10	48.92	10.82
79.66	20.34	52.84	8.33
100.00	0.00	72.26	0.00

DAY 4

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
34.15	65.85	19.71	26.20
45.12	54.88	26.21	20.81
55.30	44.70	32.45	16.95
62.06	37.94	37.58	14.52
72.14	27.86	43.91	10.58
77.58	22.42	47.56	8.72
100.00	0.00	64.74	0.00

DAY 5

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.58	69.42	16.55	27.54
48.19	51.81	27.58	19.21
58.97	41.03	33.58	15.22
80.72	19.28	46.93	6.79
100.00	0.00	63.30	0.00

DAY 6

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
53.62	46.38	28.27	17.08
63.85	36.15	36.33	13.62
72.73	27.27	42.31	9.95
81.84	18.16	49.77	7.09
32.63	67.37	17.65	27.25
100.00	0.00	63.39	0.00

Day	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	**PTT-302-1**	64.47	**302-1**	45.74	9.91	75.25	71.62	73.19
	PTT-302-2	63.02	**302-2**	46.12	10.11	75.80	72.21	72.68
	PTT-302-3	63.81	**302-3**	45.70	9.68	75.20	71.55	73.77
	PTT-302-4	64.17	**302-4**	45.50	9.91	74.91	71.24	73.19
	average	**63.87**		**45.77**	**9.90**	**75.29**	**71.66**	**73.21**
	confidence limit	**1.00**		**0.41**	**0.28**	**0.59**	**0.65**	**0.71**
	SD	**0.63**		**0.26**	**0.18**	**0.37**	**0.41**	**0.44**
	CV %	**0.98**		**0.57**	**1.77**	**0.49**	**0.57**	**0.61**
Day 2	**PTT-302-1**	61.93	**302-1**	44.95	9.66	75.53	72.53	73.63
	PTT-302-2	61.89	**302-2**	45.26	9.71	75.99	73.03	73.51
	PTT-302-3	61.38	**302-3**	44.14	9.70	74.34	71.22	73.53
	PTT-302-4	62.71	**302-4**	45.31	10.10	76.07	73.11	72.50
	average	**61.98**		**44.92**	**9.79**	**75.48**	**72.47**	**73.29**
	confidence limit	**0.87**		**0.86**	**0.33**	**1.27**	**1.39**	**0.84**
	SD	**0.55**		**0.54**	**0.21**	**0.80**	**0.87**	**0.53**
	CV %	**0.89**		**1.20**	**2.11**	**1.06**	**1.20**	**0.72**
Day 3	**PTT-302-1**	72.92	**302-1**	52.27	11.54	77.36	72.34	71.97
	PTT-302-2	71.64	**302-2**	51.21	10.95	76.06	70.87	73.31
	PTT-302-3	71.55	**302-3**	52.50	11.47	77.64	72.66	72.13
	PTT-302-4	72.92	**302-4**	52.26	11.53	77.35	72.32	71.99
	average	**72.26**		**52.06**	**11.37**	**77.10**	**72.05**	**72.35**
	confidence limit	**1.22**		**0.92**	**0.45**	**1.13**	**1.27**	**1.02**
	SD	**0.77**		**0.58**	**0.28**	**0.71**	**0.80**	**0.64**
	CV %	**1.06**		**1.11**	**2.49**	**0.92**	**1.11**	**0.89**
Day 4	**PTT-302-1**	64.03	**302-1**	45.10	9.63	73.45	69.67	75.10
	PTT-302-2	65.09	**302-2**	45.27	9.81	73.69	69.93	74.64
	PTT-302-3	63.96	**302-3**	45.73	9.83	74.34	70.64	74.59
	PTT-302-4	65.86	**302-4**	44.57	9.29	72.69	68.85	75.98
	average	**64.74**		**45.17**	**9.64**	**73.54**	**69.77**	**75.08**
	confidence limit	**1.45**		**0.76**	**0.40**	**1.09**	**1.18**	**1.03**
	SD	**0.91**		**0.48**	**0.25**	**0.68**	**0.74**	**0.65**
	CV %	**1.41**		**1.06**	**2.59**	**0.93**	**1.06**	**0.86**
Day 5	**PTT-302-1**	62.22	**302-1**	44.76	9.95	75.76	70.72	71.93
	PTT-302-2	63.15	**302-2**	45.25	10.06	76.47	71.49	71.64
	PTT-302-3	64.39	**302-3**	45.11	10.21	76.27	71.27	71.25
	PTT-302-4	63.42	**302-4**	44.87	9.38	75.92	70.89	73.43
	average	**63.30**		**45.00**	**9.90**	**76.11**	**71.09**	**72.06**
	confidence limit	**1.42**		**0.35**	**0.58**	**0.51**	**0.56**	**1.51**
	SD	**0.89**		**0.22**	**0.36**	**0.32**	**0.35**	**0.95**
	CV %	**1.41**		**0.50**	**3.66**	**0.42**	**0.50**	**1.32**
Day 6	**PTT-302-1**	63.47	**302-1**	44.16	9.94	74.67	69.66	72.85
	PTT-302-2	63.54	**302-2**	45.00	9.87	75.82	70.99	73.03
	PTT-302-3	63.16	**302-3**	44.70	9.89	75.41	70.52	72.98
			302-4	44.76	9.91	75.49	70.61	72.93
	average	**63.39**		**44.66**	**9.90**	**75.35**	**70.44**	**72.95**
	confidence limit	**0.50**		**0.56**	**0.05**	**0.77**	**0.89**	**0.12**
	SD	**0.20**		**0.35**	**0.03**	**0.48**	**0.56**	**0.08**
	CV %	**0.32**		**0.79**	**0.30**	**0.64**	**0.79**	**0.10**

DAY 1

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
37.83	62.17	23.54	33.96
37.99	62.01	22.81	34.04
48.27	51.73	29.21	28.50
65.38	34.62	40.70	19.29
71.62	28.38	43.08	15.65
83.29	16.71	52.19	9.23
100.00	0.00	62.28	0.00

DAY 2

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
42.99	57.01	23.83	31.44
49.17	50.83		27.6
54.32	45.68	32.90	24.44
60.25	39.75	36.89	21.28
69.57	30.43	42.73	16.56
80.31	19.69	46.65	10.18
100	0.00	61.01	0.00

DAY 3

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
38.38	61.62	25.39	38.91
52.13	47.87	35.69	30.10
53.74	46.26	39.52	33.60
59.83	40.17	41.99	24.75
71.52	28.48	51.31	17.99
79.96	20.04	56.19	11.95
100.00	0.00	70.68	0.00

DAY 4

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
36.19	63.81	21.33	35.53
42.95	57.05	26.67	32.65
59.54	40.46	36.30	21.99
75.00	25.00	46.59	14.05
82.34	17.66	51.65	9.89
100.00	0.00	63.12	0.00

DAY 5

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
36.96	63.04	22.32	36.25
49.54	50.46	31.00	28.32
64.56	35.44	39.75	18.63
74.61	25.39	48.02	14.05
100.00	0.00	64.12	0.00

DAY 6

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
29.81	70.19	16.68	35.49
52.50	47.50	30.66	24.50
63.33	36.67	35.03	17.57
71.75	28.25	42.76	15.37
77.60	22.40	46.10	12.39
100.00	0.00	63.18	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	**PTT-309-1**	62.08	**309-1**	47.08	12.06	76.21	75.59	78.06
	PTT-309-2	62.32	**309-2**	47.48	12.27	76.85	76.24	77.67
	PTT-309-3	62.26	**309-3**	46.97	12.12	76.03	75.42	77.95
	PTT-309-4	62.46	**309-4**	46.72		75.62	75.02	
	average	**62.28**		**47.06**	**12.15**	**76.18**	**75.57**	**77.89**
	confidence limit	**0.25**		**0.50**	**0.27**	**0.82**	**0.81**	**0.49**
	SD	**0.16**		**0.32**	**0.11**	**0.51**	**0.51**	**0.20**
	CV %	**0.25**		**0.67**	**0.89**	**0.67**	**0.67**	**0.25**
Day 2	**PTT-309-1**	60.28	**309-1**	45.41	13.36	75.39	74.43	75.36
	PTT-309-2	61.03	**309-2**	47.43	13.07	78.62	77.74	75.89
	PTT-309-3	61.85	**309-3**	45.79	13.32	76.00	75.06	75.43
	PTT-309-4	60.87	**309-4**	46.12	13.63	76.53	75.60	74.86
	average	**61.01**		**46.19**	**13.35**	**76.63**	**75.71**	**75.38**
	confidence limit	**1.03**		**1.40**	**0.36**	**2.23**	**2.29**	**0.67**
	SD	**0.65**		**0.88**	**0.23**	**1.40**	**1.44**	**0.42**
	CV %	**1.06**		**1.90**	**1.72**	**1.83**	**1.90**	**0.56**
Day 3	**PTT-309-1**	68.96	**309-1**	52.10	15.00	74.13	73.72	76.05
	PTT-309-2	72.43	**309-2**	50.43	14.73	71.76	71.35	76.48
	PTT-309-3	70.80	**309-3**	51.12	15.02	72.74	72.33	76.02
	PTT-309-4	70.52	**309-4**	52.11	14.61	74.15	73.73	76.68
	average	**70.68**		**51.44**	**14.84**	**73.19**	**72.78**	**76.31**
	confidence limit	**2.26**		**1.30**	**0.32**	**1.85**	**1.84**	**0.51**
	SD	**1.42**		**0.82**	**0.20**	**1.16**	**1.16**	**0.32**
	CV %	**2.01**		**1.59**	**1.36**	**1.59**	**1.59**	**0.42**
Day 4	**PTT-309-1**	62.74	**309-1**	47.32	13.90	75.93	74.97	75.18
	PTT-309-2	62.46	**309-2**	47.52	12.97	76.25	75.29	76.84
	PTT-309-3	64.00	**309-3**	46.38	13.85	74.42	73.48	75.27
	PTT-309-4	63.28	**309-4**	46.91	13.13	75.27	74.32	76.56
	average	**63.12**		**47.03**	**13.46**	**75.47**	**74.51**	**75.96**
	confidence limit	**1.08**		**0.80**	**0.77**	**1.29**	**1.27**	**1.37**
	SD	**0.68**		**0.50**	**0.48**	**0.81**	**0.80**	**0.86**
	CV %	**1.07**		**1.07**	**3.57**	**1.07**	**1.07**	**1.13**
Day 5	**PTT-309-1**	64.64	**309-1**	48.32	13.78	76.27	75.36	74.03
	PTT-309-2	63.77	**309-2**	48.66	13.84	76.81	75.89	73.92
	PTT-309-3	64.00	**309-3**	48.78	13.83	77.00	76.07	73.94
	PTT-309-4	64.08	**309-4**	48.95	13.79	77.27	76.34	74.01
	average	**64.12**		**48.68**	**13.81**	**76.84**	**75.91**	**73.97**
	confidence limit	**0.59**		**0.42**	**0.05**	**0.67**	**0.66**	**0.08**
	SD	**0.37**		**0.27**	**0.03**	**0.42**	**0.42**	**0.05**
	CV %	**0.58**		**0.55**	**0.21**	**0.55**	**0.55**	**0.07**
Day 6	**PTT-309-1**	63.98	**309-1**	46.35	13.61	77.51	73.36	74.94
	PTT-309-2	62.48	**309-2**	45.30	13.28	76.01	71.70	75.57
	PTT-309-3	63.08	**309-3**	46.56	13.56	77.81	73.69	75.03
	PTT-309-4		**309-4**	46.22	13.55	77.33	73.16	75.05
	average	**63.18**		**46.11**	**13.50**	**77.16**	**72.98**	**75.15**
	confidence limit	**1.88**		**0.89**	**0.24**	**1.27**	**1.40**	**0.45**
	SD	**0.75**		**0.56**	**0.15**	**0.80**	**0.88**	**0.28**
	CV %	**1.19**		**1.21**	**1.10**	**1.03**	**1.21**	**0.38**

DAY 1

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
25.36	74.64	15.09	39.15
43.50	56.50	28.11	27.67
58.10	41.90	34.86	21.63
69.69	30.31	41.67	15.53
78.21	21.79	47.61	11.45
100.00	0.00	61.36	0.00

DAY 2

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.96	81.38	18.62	35.43
42.86	73.33	26.67	30.54
49.83	69.12	30.88	26.28
54.48	65.31	34.69	23.98
71.26	56.39	43.61	15.05
78.62	49.71	50.29	11.51
100	0.00	0.00	0.00

DAY 3

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
23.36	76.64	16.09	45.39
37.43	62.57	26.41	37.66
43.98	56.02	31.70	33.93
57.88	42.12	41.64	25.33
69.18	30.82	50.36	17.95
76.25	23.75	55.25	14.38
100.00	0.00	73.01	0.00

DAY 4

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
23.54	76.46	13.35	42.03
37.12	62.88	22.29	33.55
48.36	51.64	31.34	27.58
54.59	45.41	34.60	24.27
61.90	38.10	39.02	20.35
79.22	20.78	51.05	11.04
100.00	0.00	64.21	0.00

DAY 5

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
46.15	53.85	29.63	29.68
57.81	42.19	38.62	23.08
64.31	35.69	41.67	19.22
76.60	23.40	48.99	12.54
100.00	0.00	65.72	0.00

DAY 6

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.85	69.15	20.19	37.93
59.06	40.94	38.00	21.81
69.87	30.13	45.72	15.85
70.44	29.56	44.94	15.47
100.00	0.00	64.05	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	**PTT-311-1**	61.14	**311-1**	42.03	13.29	68.92	68.49	73.68
	PTT-311-2	60.32	**311-2**	44.72	15.80	73.34	72.88	68.80
	PTT-311-3	62.12	**311-3**	42.99	15.86	70.50	70.06	68.69
	PTT-311-4	61.87	**311-4**	42.00	15.41	68.88	68.45	69.56
	average	**61.36**		**42.94**	**15.09**	**70.41**	**69.97**	**70.18**
	confidence limit	**1.29**		**2.03**	**1.94**	**3.33**	**3.31**	**3.76**
	SD	**0.81**		**1.28**	**1.22**	**2.09**	**2.08**	**2.37**
	CV %	**1.32**		**2.97**	**8.06**	**2.97**	**2.97**	**3.37**
Day 2	**PTT-311-1**		**311-1**	45.33	16.05	71.49	70.97	69.35
	PTT-311-2	63.83	**311-2**	45.29	16.10	71.42	70.91	69.25
	PTT-311-3	64.20	**311-3**	45.19	16.00	71.27	70.75	69.44
	PTT-311-4	63.58	**311-4**	45.01	15.85	70.98	70.47	69.73
	average	**63.87**		**45.21**	**16.00**	**71.29**	**70.78**	**69.44**
	confidence limit	**0.77**		**0.23**	**0.17**	**0.36**	**0.36**	**0.33**
	SD	**0.31**		**0.14**	**0.11**	**0.23**	**0.22**	**0.21**
	CV %	**0.49**		**0.32**	**0.68**	**0.32**	**0.32**	**0.30**
Day 3	**PTT-311-1**	72.37	**311-1**	49.33	17.43	68.08	67.56	70.86
	PTT-311-2	73.05	**311-2**	50.07	17.54	69.10	68.58	70.67
	PTT-311-3	73.45	**311-3**	50.67	18.14	69.93	69.40	69.67
	PTT-311-4	73.18	**311-4**	49.02	17.81	67.65	67.14	70.22
	average	**73.01**		**49.77**	**17.73**	**68.69**	**68.17**	**70.36**
	confidence limit	**0.73**		**1.18**	**0.50**	**1.63**	**1.62**	**0.84**
	SD	**0.46**		**0.74**	**0.32**	**1.03**	**1.02**	**0.53**
	CV %	**0.63**		**1.49**	**1.79**	**1.49**	**1.49**	**0.75**
Day 4	**PTT-311-1**		**311-1**	45.35	16.08	71.19	70.63	70.19
	PTT-311-2	64.20	**311-2**	45.35	15.93	71.19	70.63	70.47
	PTT-311-3	63.73	**311-3**	44.54	15.70	69.92	69.37	70.90
	PTT-311-4	64.69	**311-4**	45.08	14.85	70.77	70.21	72.47
	average	**64.21**		**45.08**	**15.64**	**70.77**	**70.21**	**71.01**
	confidence limit	**1.19**		**0.61**	**0.87**	**0.95**	**0.95**	**1.62**
	SD	**0.48**		**0.38**	**0.55**	**0.60**	**0.59**	**1.02**
	CV %	**0.75**		**0.85**	**3.51**	**0.85**	**0.85**	**1.43**
Day 5	**PTT-311-1**	66.67	**311-1**	46.99	16.71	72.09	71.50	69.41
	PTT-311-2	65.35	**311-2**	46.37	16.25	71.14	70.55	70.25
	PTT-311-3	65.75	**311-3**	47.12	16.68	72.29	71.70	69.47
	PTT-311-4	65.12	**311-4**	46.81	16.94	71.82	71.22	68.99
	average	**65.72**		**46.82**	**16.65**	**71.84**	**71.24**	**69.53**
	confidence limit	**1.09**		**0.52**	**0.46**	**0.80**	**0.79**	**0.84**
	SD	**0.68**		**0.33**	**0.29**	**0.50**	**0.50**	**0.53**
	CV %	**1.04**		**0.70**	**1.73**	**0.70**	**0.70**	**0.76**
Day 6	**PTT-311-1**	64.88	**311-1**	45.70	15.75	71.00	71.36	70.84
	PTT-311-2	62.91	**311-2**	45.21	14.96	70.23	70.59	72.31
	PTT-311-3	63.80	**311-3**	46.17	15.84	71.73	72.09	70.68
	PTT-311-4	64.59	**311-4**		15.20			71.86
	average	**64.05**		**45.69**	**15.44**	**70.99**	**71.35**	**71.42**
	confidence limit	**1.41**		**1.19**	**0.68**	**1.85**	**1.19**	**1.25**
	SD	**0.88**		**0.48**	**0.43**	**0.75**	**0.75**	**0.79**
	CV %	**1.38**		**1.05**	**2.76**	**1.05**	**1.05**	**1.10**

DAY 1

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
26.62	73.38	15.04	37.96
42.80	57.20	25.70	29.54
53.97	46.03	32.11	24.05
64.72	35.28	38.58	18.00
74.19	25.81	44.82	12.95
78.52	21.48	47.22	10.99
100.00	0.00	61.10	0.00

DAY 2

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
35.56	64.44	21.66	36.14
52.67	47.33	33.78	26.85
62.18	37.82	39.34	20.53
69.91	30.09	46.25	16.72
76.83	23.17	53.51	12.8
81.49	18.51	57.05	10.16
100	0.00	67.61	0.00

DAY 3

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
25.09	74.91	16.98	45.31
32.94	67.06	22.77	40.02
43.17	56.83	28.63	32.78
50.00	50.00	34.01	28.82
66.82	33.18	45.70	18.98
79.43	20.57	56.12	11.66
100.00	0.00	72.57	0.00

DAY 4

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
31.07	68.93	19.03	35.56
49.31	50.69	30.01	26.85
75.99	24.01	49.63	12.31
76.86	23.14	48.64	11.64
84.21	15.79	53.81	7.58
100.00	0.00	62.97	0.00

DAY 5

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
43.32	56.68	25.69	30.46
50.18	49.82	31.46	26.47
61.06	38.94	36.65	20.47
81.41	18.59	52.78	9.69
100.00	0.00	63.77	0.00

DAY 6

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
43.53	56.47	22.06	27.51
55.67	44.33	29.86	22.86
73.89	26.11	42.74	13.05
82.13	17.87	49.14	9.13
100.00	0.00	61.03	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	**PTT-312-1**	60.16	**312-1**	38.49	19.41	63.85	63.00	62.41
	PTT-312-2	62.47	**312-2**	38.73	19.59	64.25	63.39	62.06
	PTT-312-3	60.67	**312-3**	39.09	19.54	64.85	63.98	62.15
	PTT-312-4		**312-4**		20.17			60.93
	average	**61.10**		**38.77**	**19.68**	**64.32**	**63.45**	**61.89**
	confidence limit	**3.01**		**0.75**	**0.54**	**1.24**	**1.23**	**1.04**
	SD	**1.21**		**0.30**	**0.34**	**0.50**	**0.49**	**0.65**
	CV %	**1.99**		**0.78**	**1.71**	**0.78**	**0.78**	**1.05**
Day 2	**PTT-312-1**	67.29	**312-1**	41.79	21.48	63.77	61.81	61.53
	PTT-312-2	68.98	**312-2**	43.75	22.23	66.51	64.71	60.18
	PTT-312-3	65.34	**312-3**	42.48	22.36	64.74	62.83	59.95
	PTT-312-4	68.83	**312-4**	42.26	22.46	64.43	62.51	59.77
	average	**67.61**		**42.57**	**22.13**	**64.86**	**62.96**	**60.36**
	confidence limit	**2.70**		**1.33**	**0.71**	**1.86**	**1.97**	**1.27**
	SD	**1.70**		**0.84**	**0.45**	**1.17**	**1.24**	**0.80**
	CV %	**2.51**		**1.97**	**2.01**	**1.81**	**1.97**	**1.32**
Day 3	**PTT-312-1**	74.43	**312-1**	44.73	21.44	68.37	66.24	63.69
	PTT-312-2	74.03	**312-2**	48.03	21.16	68.31	66.18	64.16
	PTT-312-3	70.98	**312-3**	48.43	20.95	68.88	66.73	64.52
	PTT-312-4	70.85	**312-4**	47.76	20.79	67.93	65.81	64.79
	average	**72.57**		**47.24**	**21.09**	**68.37**	**66.24**	**64.29**
	confidence limit	**3.06**		**2.70**	**0.45**	**0.62**	**0.60**	**0.76**
	SD	**1.92**		**1.69**	**0.28**	**0.39**	**0.38**	**0.48**
	CV %	**2.65**		**3.59**	**1.33**	**0.57**	**0.57**	**0.74**
Day 4	**PTT-312-1**	61.63	**312-1**	39.53	18.88	62.31	62.78	63.55
	PTT-312-2	63.72	**312-2**	40.42	19.27	63.71	64.19	62.79
	PTT-312-3	62.84	**312-3**	41.05	19.11	64.71	65.19	63.10
	PTT-312-4	63.68	**312-4**	41.01	19.51	64.64	65.13	62.33
	average	**62.97**		**40.50**	**19.19**	**63.84**	**64.32**	**62.94**
	confidence limit	**1.56**		**1.13**	**0.42**	**1.78**	**1.79**	**0.82**
	SD	**0.98**		**0.71**	**0.27**	**1.12**	**1.13**	**0.51**
	CV %	**1.56**		**1.75**	**1.38**	**1.75**	**1.75**	**0.81**
Day 5	**PTT-312-1**	64.32	**312-1**	40.18	20.47	64.44	63.00	61.54
	PTT-312-2	64.26	**312-2**	39.25	20.24	63.05	61.55	61.98
	PTT-312-3		**312-3**	40.54	19.77	64.98	63.57	62.86
	PTT-312-4	62.74	**312-4**	39.40	19.89	63.27	61.78	62.63
	average	**63.77**		**39.84**	**20.09**	**63.94**	**62.48**	**62.25**
	confidence limit	**2.22**		**0.98**	**0.51**	**1.47**	**1.54**	**0.96**
	SD	**0.90**		**0.62**	**0.32**	**0.93**	**0.97**	**0.60**
	CV %	**1.40**		**1.55**	**1.60**	**1.45**	**1.55**	**0.97**
Day 6	**PTT-312-1**	60.76	**312-1**	35.60	17.87	64.05	58.34	64.18
	PTT-312-2	61.22	**312-2**	34.52	17.73	62.42	56.57	64.46
	PTT-312-3	61.10	**312-3**	35.15	18.21	63.37	57.60	63.50
	PTT-312-4		**312-4**	36.67	17.51	65.66	60.09	64.90
	average	**61.03**		**35.49**	**17.83**	**63.88**	**58.15**	**64.26**
	confidence limit	**0.59**		**1.44**	**0.47**	**2.17**	**2.36**	**0.94**
	SD	**0.24**		**0.91**	**0.29**	**1.36**	**1.48**	**0.59**
	CV %	**0.39**		**2.55**	**1.65**	**2.13**	**2.55**	**0.92**

PARALLEL INTEGRATION

Quantification based on PTT and PET peak integration

DAY 1

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
26.17	73.83	12.59	28.34
34.96	65.04	17.47	24.26
48.29	51.71	25.64	18.30
59.64	40.36	31.94	14.10
70.06	29.94	40.30	10.66
77.44		43.55	
100.00	0.00	60.44	

DAY 2

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.95	69.05	15.01	25.1
53.82	46.18	28.38	16.09
57.08	42.92	30.36	13.2
70.87	29.13	38.24	9.49
75.88	24.12	43.15	8.42
79.95	20.05	46.06	6.79
100	0.00	59.59	0.00

DAY 3

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
35.24	64.76	19.68	26.73
52.65	47.35	31.16	17.62
62.90	37.10	38.45	13.76
71.13	28.87	43.73	11.13
73.90	26.10	46.29	10.26
79.66	20.34	50.29	7.50
100.00	0.00	68.95	0.00

DAY 4

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
34.15	65.85	17.62	23.68
45.12	54.88	23.95	18.37
55.30	44.70	30.13	14.99
62.06	37.94	35.00	12.94
72.14	27.86	41.87	9.20
77.58	22.42	45.20	7.67
100.00	0.00	62.68	0.00

DAY 5

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.58	69.42	14.98	25.07
48.19	51.81	25.24	16.64
58.97	41.03	31.27	13.43
80.72	19.28	44.87	6.34
100.00	0.00	60.98	0.00

DAY 6

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
32.63	67.37	15.68	25.21
53.62	46.38	25.48	15.26
63.85	36.15	33.63	12.39
72.73	27.27	40.04	9.39
81.84	18.16	46.78	7.05
100.00	0.00	59.85	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	PTT-302-1	60.55	302-1	43.14	9.13	76.28	71.37	73.05
	PTT-302-2	59.76	302-2	43.46	9.62	76.75	71.90	71.70
	PTT-302-3	60.59	302-3	42.68	9.06	75.61	70.61	73.24
	PTT-302-4	60.87	302-4	42.75	9.06	75.71	70.73	73.24
	average	60.44		43.01	9.22	76.09	71.15	72.81
	confidence limit	0.76		0.58	0.43	0.85	0.96	1.18
	SD	0.48		0.36	0.27	0.53	0.60	0.74
	CV %	0.79		0.84	2.93	0.70	0.84	1.02
Day 2	PTT-302-1	59.29	302-1	42.16	8.71	75.01	70.76	74.10
	PTT-302-2	59.57	302-2	42.51	9.11	75.53	71.34	72.96
	PTT-302-3	58.31	302-3	41.68	8.80	74.31	69.95	73.84
	PTT-302-4	61.17	302-4	42.66	9.30	75.75	71.60	72.42
	average	59.59		42.25	8.98	75.15	70.91	73.33
	confidence limit	1.89		0.69	0.44	1.02	1.16	1.23
	SD	1.19		0.44	0.27	0.64	0.73	0.78
	CV %	1.99		1.03	3.05	0.85	1.03	1.06
Day 3	PTT-302-1	69.65	302-1	49.35	10.59	77.60	71.57	71.45
	PTT-302-2	68.18	302-2	48.49	10.13	76.52	70.33	72.62
	PTT-302-3	68.13	302-3	49.65	10.93	77.97	72.01	70.60
	PTT-302-4	69.84	302-4	49.44	10.79	77.71	71.70	70.95
	average	68.95		49.23	10.61	77.45	71.40	71.41
	confidence limit	1.47		0.81	0.56	1.02	1.18	1.40
	SD	0.92		0.51	0.35	0.64	0.74	0.88
	CV %	1.34		1.04	3.29	0.83	1.04	1.23
Day 4	PTT-302-1	61.84	302-1	42.97	8.70	74.18	68.56	73.48
	PTT-302-2	63.18	302-2	43.36	8.66	74.73	69.18	73.60
	PTT-302-3	62.19	302-3	43.39	8.80	74.77	69.23	73.19
	PTT-302-4	63.50	302-4	42.26	8.23	73.17	67.42	74.85
	average	62.68		43.00	8.60	74.21	68.60	73.78
	confidence limit	1.26		0.84	0.40	1.19	1.34	1.17
	SD	0.79		0.53	0.25	0.75	0.84	0.73
	CV %	1.26		1.22	2.93	1.01	1.22	0.99
Day 5	PTT-302-1	59.47	302-1	43.23	9.15	76.49	70.89	70.47
	PTT-302-2	61.28	302-2	42.68	9.34	75.70	69.99	69.92
	PTT-302-3	62.24	302-3	42.76	9.49	75.82	70.12	69.48
	PTT-302-4	60.93	302-4	42.21	8.66	75.03	69.22	71.91
	average	60.98		42.72	9.16	75.76	70.06	70.45
	confidence limit	1.83		0.66	0.57	0.95	1.09	1.68
	SD	1.15		0.42	0.36	0.60	0.68	1.06
	CV %	1.88		0.98	3.94	0.79	0.98	1.50
Day 6	PTT-302-1	60.18	302-1	41.86	9.27	76.26	69.94	72.47
	PTT-302-2	59.09	302-2	41.42	9.30	75.63	69.21	72.39
	PTT-302-3	60.28	302-3	41.57	9.30	75.85	69.46	72.39
	PTT-302-4		302-4	41.60	9.31	75.89	69.51	72.36
	average	59.85		41.61	9.30	75.91	69.53	72.40
	confidence limit	1.64		0.29	0.03	0.41	0.49	0.08
	SD	0.66		0.18	0.02	0.26	0.31	0.05
	CV %	1.10		0.44	0.19	0.34	0.44	0.07

DAY 1

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
37.83	62.17	21.72	32.23
37.99	62.01	20.99	32.77
48.27	51.73	27.05	26.89
65.38	34.62	38.01	18.18
71.62	28.38	41.31	14.29
83.29	16.71	48.97	8.40
100.00	0.00	59.69	0.00

DAY 2

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
42.99	57.01	22.10	29.8
54.32	45.68	30.71	23.02
60.25	39.75	34.52	19.81
69.57	30.43	40.18	15.35
80.31	19.69	44.58	9.31
100.00	0.00	58.38	0

DAY 3

PTT 309 %	PET 309 %	PTT 309 J/g	PET enthalpy J/g
0.00	100.00	0.00	
38.38	61.62	23.40	37.01
52.13	47.87	33.30	27.85
59.83	40.17	39.71	22.07
71.52	28.48	48.21	16.70
79.96	20.04	53.16	11.05
100.00	0.00	66.91	0.00

DAY 4

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
36.19	63.81	20.88	34.05
42.95	57.05	24.97	31.55
59.54	40.46	34.58	21.33
75.00	25.00	44.79	13.15
82.34	17.66	50.21	9.19
100.00	0.00	61.07	0.00

DAY 5

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
36.96	63.04	20.65	35.15
49.54	50.46	29.19	27.18
64.56	35.44	38.52	17.21
74.61	25.39	45.40	13.04
100.00	0.00	61.20	0.00

DAY 6

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
29.81	70.19	15.24	34.06
52.50	47.50	28.29	23.01
63.33	36.67	33.06	16.80
71.75	28.25	40.57	14.23
77.60	22.40	43.23	11.26
100.00	0.00	60.16	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	**PTT-309-1**	60.07	**309-1**	44.36	12.06	75.98	74.32	76.86
	PTT-309-2	59.39	**309-2**	44.84	12.27	76.81	75.12	76.45
	PTT-309-3	59.12	**309-3**	44.74	12.12	76.64	74.96	76.74
	PTT-309-4	60.17	**309-4**	44.57		76.34	74.67	
	average	**59.69**		**44.63**	**12.15**	**76.44**	**74.77**	**76.68**
	confidence limit	**0.82**		**0.33**	**0.27**	**0.57**	**0.56**	**0.52**
	SD	**0.51**		**0.21**	**0.11**	**0.36**	**0.35**	**0.21**
	CV %	**0.86**		**0.47**	**0.89**	**0.47**	**0.47**	**0.27**
Day 2	**PTT-309-1**	57.62	**309-1**	43.38	12.56	76.20	74.31	74.19
	PTT-309-2	57.73	**309-2**	44.88	12.37	78.63	76.88	74.56
	PTT-309-3	59.11	**309-3**	43.61	12.53	76.57	74.70	74.25
	PTT-309-4	59.06	**309-4**	43.83	12.77	76.93	75.08	73.79
	average	**58.38**		**43.93**	**12.56**	**77.08**	**75.24**	**74.20**
	confidence limit	**1.30**		**1.05**	**0.26**	**1.71**	**1.81**	**0.51**
	SD	**0.82**		**0.66**	**0.16**	**1.07**	**1.14**	**0.32**
	CV %	**1.40**		**1.51**	**1.31**	**1.39**	**1.51**	**0.43**
Day 3	**PTT-309-1**	65.77	**309-1**	48.92	13.86	73.74	73.12	75.06
	PTT-309-2	68.28	**309-2**	47.88	13.98	72.27	71.56	74.85
	PTT-309-3	67.20	**309-3**	48.39	14.05	72.99	72.32	74.74
	PTT-309-4	66.38	**309-4**	49.40	13.78	74.41	73.83	75.20
	average	**66.91**		**48.65**	**13.92**	**73.35**	**72.71**	**74.96**
	confidence limit	**1.73**		**1.05**	**0.19**	**1.48**	**1.56**	**0.33**
	SD	**1.09**		**0.66**	**0.12**	**0.93**	**0.98**	**0.21**
	CV %	**1.62**		**1.35**	**0.87**	**1.27**	**1.35**	**0.27**
Day 4	**PTT-309-1**	61.29	**309-1**	45.74	13.15	76.12	74.89	75.56
	PTT-309-2	60.18	**309-2**	45.94	12.15	76.45	75.22	77.42
	PTT-309-3	61.91	**309-3**	45.45	13.28	75.64	74.42	75.32
	PTT-309-4	60.91	**309-4**	44.65	12.27	74.31	73.11	77.19
	average	**61.07**		**45.45**	**12.71**	**75.63**	**74.41**	**76.37**
	confidence limit	**1.15**		**0.90**	**0.93**	**1.50**	**1.48**	**1.73**
	SD	**0.72**		**0.57**	**0.58**	**0.94**	**0.93**	**1.09**
	CV %	**1.19**		**1.25**	**4.60**	**1.25**	**1.25**	**1.42**
Day 5	**PTT-309-1**	61.92	**309-1**	45.67	13.13	75.74	74.78	73.22
	PTT-309-2	60.87	**309-2**	46.20	13.09	76.62	75.65	73.29
	PTT-309-3	61.20	**309-3**	46.46	13.83	77.05	76.07	71.92
	PTT-309-4	60.81	**309-4**	46.60	13.07	77.28	76.30	73.33
	average	**61.20**		**46.23**	**13.28**	**76.67**	**75.70**	**72.94**
	confidence limit	**0.81**		**0.65**	**0.58**	**1.08**	**1.07**	**1.08**
	SD	**0.51**		**0.41**	**0.37**	**0.68**	**0.67**	**0.68**
	CV %	**0.83**		**0.89**	**2.77**	**0.89**	**0.89**	**0.93**
Day 6	**PTT-309-1**	60.57	**309-1**	44.56	12.49	78.75	74.07	74.18
	PTT-309-2	58.75	**309-2**	42.75	12.43	76.09	71.06	74.30
	PTT-309-3	61.15	**309-3**	44.56	12.44	78.75	74.07	74.28
	PTT-309-4		**309-4**	43.49	12.51	77.18	72.29	74.14
	average	**60.16**		**43.84**	**12.47**	**77.69**	**72.88**	**74.22**
	confidence limit	**3.11**		**1.41**	**0.06**	**2.07**	**2.34**	**0.13**
	SD	**1.25**		**0.88**	**0.04**	**1.30**	**1.47**	**0.08**
	CV %	**2.08**		**2.02**	**0.31**	**1.67**	**2.02**	**0.11**

DAY 1

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
25.36	74.64	13.86	37.80
43.50	56.50	25.92	25.73
58.10	41.90	33.02	20.44
69.69	30.31	39.81	14.65
78.21	21.79	45.67	10.56
100.00	0.00	58.84	0.00

DAY 2

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.96	69.04	17.17	34.45
42.86	57.14	25.00	29.39
49.83	50.17	29.12	24.76
54.48	45.52	32.61	22.71
71.26	28.74		14.31
78.62	21.38	48.13	10.38
100	0.00	61.05	0.00

DAY 3

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
23.36	76.64	14.60	44.01
37.43	62.57	24.57	36.58
43.98	56.02	29.62	32.37
57.88	42.12	39.26	24.33
69.18	30.82	47.93	16.53
76.25	23.75	52.63	13.62
100.00	0.00	69.81	0.00

DAY 4

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
23.54	76.46	11.99	41.57
37.12	62.88	20.97	32.51
48.36	51.64	29.45	27.14
54.59	45.41	32.77	23.61
61.90	38.10	37.34	19.68
79.22	20.78	49.08	10.35
100.00	0.00	62.48	0.00

DAY 5

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
46.15	53.85	27.78	29.00
57.81	42.19	36.33	22.05
64.31	35.69	39.25	19.22
76.60	23.40	46.46	11.58
100.00	0.00	63.29	0.00

DAY 6

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.85	69.15	18.08	37.36
59.06	40.94	35.26	20.34
69.87	30.13	43.32	14.68
70.44	29.56	42.44	14.46
100.00	0.00	61.90	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	PTT-311-1	58.13	311-1	39.92	12.16	68.67	67.85	73.91
	PTT-311-2	57.32	311-2	42.29	14.71	72.75	71.88	68.65
	PTT-311-3	60.31	311-3	41.12	14.71	70.74	69.89	68.65
	PTT-311-4	59.58	311-4	40.03	14.68	68.86	68.04	68.71
	average	58.84		40.84	14.07	70.26	69.41	69.98
	confidence limit	2.16		1.76	2.02	3.03	3.00	4.17
	SD	1.36		1.11	1.27	1.91	1.88	2.62
	CV %	2.31		2.71	9.03	2.71	2.71	3.74
Day 2	PTT-311-1	61.28	311-1	43.13	15.12	71.57	70.65	69.83
	PTT-311-2	61.27	311-2	42.88	14.91	71.16	70.24	70.25
	PTT-311-3	60.60	311-3	42.82	14.85	71.06	70.14	70.37
	PTT-311-4		311-4	42.91	14.95	71.21	70.29	70.17
	average	61.05		42.94	14.96	71.25	70.33	70.15
	confidence limit	0.62		0.22	0.18	0.36	0.35	0.37
	SD	0.39		0.14	0.12	0.22	0.22	0.23
	CV %	0.64		0.32	0.77	0.32	0.32	0.33
Day 3	PTT-311-1	69.22	311-1	47.30	16.64	68.76	67.76	71.09
	PTT-311-2	69.60	311-2	47.56	17.07	69.14	68.13	70.34
	PTT-311-3	70.18	311-3	48.30	17.15	70.21	69.19	70.21
	PTT-311-4	70.22	311-4	46.75	17.33	67.96	66.97	69.89
	average	69.81		47.48	17.05	69.02	68.01	70.38
	confidence limit	0.77		1.02	0.47	1.49	1.47	0.81
	SD	0.48		0.64	0.29	0.94	0.92	0.51
	CV %	0.69		1.36	1.72	1.36	1.36	0.72
Day 4	PTT-311-1	62.65	311-1	43.39	15.59	71.08	69.45	70.46
	PTT-311-2	60.95	311-2	43.71	15.38	71.56	69.96	70.85
	PTT-311-3	63.84	311-3	42.78	15.22	70.16	68.47	71.16
	PTT-311-4		311-4	42.80	14.16	70.19	68.50	73.17
	average	62.48		43.17	15.09	70.75	69.09	71.41
	confidence limit	3.61		0.73	1.01	1.09	1.17	1.92
	SD	1.45		0.46	0.64	0.69	0.73	1.21
	CV %	2.32		1.06	4.22	0.97	1.06	1.69
Day 5	PTT-311-1	64.05	311-1	44.84	15.81	72.29	70.85	69.36
	PTT-311-2	63.05	311-2	44.34	15.73	71.53	70.06	69.51
	PTT-311-3	63.33	311-3	44.93	15.84	72.43	70.99	69.31
	PTT-311-4	62.74	311-4	44.30	16.16	71.47	69.99	68.72
	average	63.29		44.60	15.89	71.93	70.47	69.23
	confidence limit	0.89		0.52	0.30	0.80	0.83	0.55
	SD	0.56		0.33	0.19	0.50	0.52	0.35
	CV %	0.88		0.74	1.19	0.70	0.74	0.50
Day 6	PTT-311-1	62.11	311-1	43.20	14.79	70.66	69.80	69.61
	PTT-311-2	59.68	311-2	42.92	14.54	70.20	69.34	70.09
	PTT-311-3	62.36	311-3	44.14	14.63	72.19	71.31	69.92
	PTT-311-4	63.43	311-4		14.65			69.88
	average	61.90		43.42	14.65	71.02	70.15	69.87
	confidence limit	2.52		1.59	0.16	2.60	2.56	0.31
	SD	1.58		0.64	0.10	1.05	1.03	0.20
	CV %	2.56		1.47	0.71	1.47	1.47	0.28

DAY 1

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
26.62	73.38	13.67	36.44
42.80	57.20	23.87	28.04
53.97	46.03	29.91	22.83
64.72	35.28	36.30	17.14
74.19	25.81	42.79	12.50
78.52	21.48	45.61	10.70
100.00	0.00	59.12	0.00

DAY 2

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
35.56	64.44	20.36	35.55
52.67	47.33	32.36	26.79
62.18	37.82	37.79	20.35
69.91	30.09	44.66	16.31
76.83	23.17	51.40	12.53
81.49	18.51	54.70	9.67
100	0.00	66.29	0.00

DAY 3

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
25.09	74.91	15.45	44.22
32.94	67.06	21.11	38.57
43.17	56.83	26.43	31.26
50.00	50.00	32.20	27.63
66.82	33.18	43.59	18.06
79.43	20.57	53.66	10.74
100.00	0.00	69.52	0.00

DAY 4

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
31.07	68.93	17.71	35.33
49.31	50.69	28.25	26.30
75.99	24.01	47.15	11.71
76.86	23.14	46.37	11.19
84.21	15.79	51.39	7.09
100.00	0.00	61.61	0.00

DAY 5

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
43.32	56.68	24.09	30.04
50.18	49.82	29.53	26.06
61.06	38.94	35.06	20.31
81.41	18.59	50.63	9.69
100.00	0.00	62.02	0.00

DAY 6

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
43.53	56.47	20.70	26.98
55.67	44.33	26.82	20.45
73.89	26.11	40.19	12.48
82.13	17.87	45.89	8.75
100.00	0.00	56.75	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	**PTT-312-1**	58.62	**312-1**	36.43	18.38	64.44	61.62	62.74
	PTT-312-2	59.97	**312-2**	36.94	19.48	65.26	62.49	60.51
	PTT-312-3	58.76	**312-3**	36.37	19.23	64.34	61.52	61.02
	PTT-312-4		**312-4**		20.17			59.11
	average	**59.12**		**36.58**	**19.32**	**64.68**	**61.88**	**60.85**
	confidence limit	**1.84**		**0.78**	**1.18**	**1.26**	**1.32**	**2.38**
	SD	**0.74**		**0.31**	**0.74**	**0.51**	**0.53**	**1.50**
	CV %	**1.26**		**0.86**	**3.83**	**0.78**	**0.86**	**2.46**
Day 2	**PTT-312-1**	65.93	**312-1**	39.70	20.51	62.78	59.89	62.74
	PTT-312-2	67.90	**312-2**	41.65	21.46	65.49	62.83	61.01
	PTT-312-3	64.06	**312-3**	40.24	22.07	63.53	60.71	59.90
	PTT-312-4	67.26	**312-4**	39.77	22.21	62.87	60.00	59.65
	average	**66.29**		**40.34**	**21.56**	**63.67**	**60.86**	**60.82**
	confidence limit	**2.70**		**1.44**	**1.23**	**2.01**	**2.17**	**2.24**
	SD	**1.70**		**0.91**	**0.77**	**1.26**	**1.37**	**1.41**
	CV %	**2.56**		**2.25**	**3.59**	**1.98**	**2.25**	**2.31**
Day 3	**PTT-312-1**	70.40	**312-1**	43.44	21.25	66.25	62.49	60.59
	PTT-312-2	71.02	**312-2**	45.69	20.77	69.33	65.72	61.41
	PTT-312-3	68.17	**312-3**	46.00	20.90	69.75	66.17	61.19
	PTT-312-4	68.48	**312-4**	45.05	20.75	68.45	64.80	61.44
	average	**69.52**		**45.05**	**20.92**	**68.45**	**64.80**	**61.16**
	confidence limit	**2.24**		**1.82**	**0.37**	**2.48**	**2.61**	**0.62**
	SD	**1.41**		**1.14**	**0.23**	**1.56**	**1.64**	**0.39**
	CV %	**2.02**		**2.53**	**1.11**	**2.28**	**2.53**	**0.64**
Day 4	**PTT-312-1**	60.97	**312-1**	37.73	18.32	61.99	61.24	64.02
	PTT-312-2	62.04	**312-2**	38.38	18.96	63.06	62.29	62.77
	PTT-312-3	61.30	**312-3**	39.17	18.89	64.36	63.57	62.90
	PTT-312-4	62.14	**312-4**	39.00	18.90	64.08	63.30	62.88
	average	**61.61**		**38.57**	**18.77**	**63.37**	**62.60**	**63.14**
	confidence limit	**0.91**		**1.04**	**0.48**	**1.71**	**1.69**	**0.94**
	SD	**0.57**		**0.65**	**0.30**	**1.08**	**1.06**	**0.59**
	CV %	**0.92**		**1.70**	**1.60**	**1.70**	**1.70**	**0.93**
Day 5	**PTT-312-1**	62.77	**312-1**	37.77	19.97	63.94	60.90	62.01
	PTT-312-2	61.60	**312-2**	37.02	20.24	62.79	59.69	61.49
	PTT-312-3	61.68	**312-3**	38.47	19.77	65.00	62.03	62.39
	PTT-312-4		**312-4**	37.27	19.89	63.17	60.10	62.16
	average	**62.02**		**37.63**	**19.97**	**63.73**	**60.68**	**62.01**
	confidence limit	**1.62**		**1.02**	**0.32**	**1.55**	**1.64**	**0.60**
	SD	**0.65**		**0.64**	**0.20**	**0.98**	**1.03**	**0.38**
	CV %	**1.05**		**1.70**	**1.00**	**1.53**	**1.70**	**0.61**
Day 6	**PTT-312-1**	56.48	**312-1**	33.80	17.78	65.15	59.56	62.43
	PTT-312-2	56.92	**312-2**	31.83	18.70	62.00	56.09	60.48
	PTT-312-3	56.85	**312-3**	32.74	18.17	63.46	57.69	61.60
	PTT-312-4		**312-4**	34.55	17.49	66.34	60.88	63.04
	average	**56.75**		**33.23**	**18.04**	**64.24**	**58.56**	**61.89**
	confidence limit	**0.59**		**1.90**	**0.83**	**3.03**	**3.34**	**1.76**
	SD	**0.24**		**1.19**	**0.52**	**1.90**	**2.10**	**1.11**
	CV %	**0.42**		**3.59**	**2.90**	**2.97**	**3.59**	**1.79**

LINEAR FIXED RANGE INTEGRATION

Quantification based on PTT and PET peak integration

DAY 1

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
26.17	73.83	11.20	30.41
34.96	65.04	17.87	26.06
48.29	51.71	26.64	20.00
59.64	40.36	33.40	15.48
70.06	29.94	42.67	11.41
77.44	22.56	45.80	8.52
100.00	0.00	62.68	0.00

DAY 2

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
57.08	42.92	32.11	14.69
70.87	29.13	40.07	10.35
75.88	24.12	45.43	9.091
79.95	20.05	48.20	7.463
53.82	46.18	30.00	17.73
30.95	69.05	14.02	26.95
100	0.00	61.35	0.00

DAY 3

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
35.24	64.76	19.00	
52.65	47.35	32.61	
62.90	37.10	40.49	16.63
71.13	28.87	45.75	9.96
73.90	26.10	48.28	7.11
79.66	20.34	52.26	
100.00	0.00	70.92	0.00

DAY 4

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
34.15	65.85	17.52	26.05
45.12	54.88	24.74	20.76
55.30	44.70	31.48	16.90
62.06	37.94	36.87	14.49
72.14	27.86	43.52	10.61
77.58	22.42	47.14	8.73
100.00	0.00	63.96	0.00

DAY 5

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.58	69.42	13.75	26.96
48.19	51.81	25.84	19.11
58.97	41.03	32.97	15.18
80.72	19.28	46.65	6.75
100.00	0.00	61.22	0.00

DAY 6

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
32.63	67.37	17.46	27.03
53.62	46.38	27.94	17.11
63.85	36.15	36.85	13.61
72.73	27.27	42.36	9.96
81.84	18.16	49.51	7.11
100.00	0.00	62.31	0.00

Day	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	**PTT-302-1**	62.74	**302-1**	45.40	9.96	76.33	72.44	73.38
	PTT-302-2	62.59	**302-2**	45.67	10.11	76.70	72.87	73.00
	PTT-302-3	61.77	**302-3**	45.21	9.77	76.07	72.13	73.85
	PTT-302-4	63.60	**302-4**	44.99	9.95	75.77	71.78	73.39
	average	**62.68**		**45.32**	**9.95**	**76.22**	**72.31**	**73.41**
	confidence limit	**1.19**		**0.46**	**0.22**	**0.63**	**0.73**	**0.56**
	SD	**0.75**		**0.29**	**0.14**	**0.39**	**0.46**	**0.35**
	CV %	**1.20**		**0.64**	**1.39**	**0.52**	**0.64**	**0.48**
Day 2	**PTT-302-1**	60.97	**302-1**	44.35	9.84	75.73	72.30	72.22
	PTT-302-2	61.16	**302-2**	44.77	9.82	76.32	72.98	73.71
	PTT-302-3	60.20	**302-3**	43.58	9.79	74.63	71.04	72.44
	PTT-302-4	63.05	**302-4**	44.82	10.14	76.39	73.06	72.44
	average	**61.35**		**44.38**	**9.90**	**75.77**	**72.34**	**72.70**
	confidence limit	**1.93**		**0.91**	**0.26**	**1.29**	**1.49**	**1.08**
	SD	**1.21**		**0.57**	**0.16**	**0.81**	**0.93**	**0.68**
	CV %	**1.97**		**1.29**	**1.65**	**1.07**	**1.29**	**0.93**
Day 3	**PTT-302-1**	71.65	**302-1**	51.32	9.96	78.03	72.37	70.56
	PTT-302-2	70.42	**302-2**	51.79	9.92	78.60	73.03	70.61
	PTT-302-3	70.20	**302-3**	50.50	9.87	77.03	71.21	70.68
	PTT-302-4	71.39	**302-4**	51.42	10.30	78.15	72.51	70.12
	average	**70.92**		**51.26**	**10.01**	**77.96**	**72.28**	**70.49**
	confidence limit	**1.13**		**0.87**	**0.31**	**1.06**	**1.22**	**0.40**
	SD	**0.71**		**0.54**	**0.20**	**0.66**	**0.77**	**0.25**
	CV %	**1.00**		**1.06**	**1.96**	**0.85**	**1.06**	**0.36**
Day 4	**PTT-302-1**	63.69	**302-1**	44.89	9.72	74.72	70.18	74.80
	PTT-302-2	64.50	**302-2**	44.92	9.87	74.76	70.23	74.39
	PTT-302-3	63.43	**302-3**	45.31	9.87	75.30	70.84	74.39
	PTT-302-4	64.22	**302-4**	44.50	9.27	74.18	69.57	75.96
	average	**63.96**		**44.91**	**9.68**	**74.74**	**70.21**	**74.88**
	confidence limit	**0.78**		**0.53**	**0.45**	**0.73**	**0.82**	**1.18**
	SD	**0.49**		**0.33**	**0.29**	**0.46**	**0.52**	**0.74**
	CV %	**0.76**		**0.74**	**2.94**	**0.61**	**0.74**	**0.99**
Day 5	**PTT-302-1**	59.79	**302-1**	45.38	9.93	78.54	74.13	73.75
	PTT-302-2	61.04	**302-2**	45.85	10.04	79.20	74.90	73.45
	PTT-302-3	62.32	**302-3**	44.80	10.18	77.72	73.18	73.08
	PTT-302-4	61.72	**302-4**	45.65	9.30	78.92	74.57	75.40
	average	**61.22**		**45.42**	**9.86**	**78.59**	**74.19**	**73.92**
	confidence limit	**1.73**		**0.73**	**0.62**	**1.02**	**1.19**	**1.63**
	SD	**1.09**		**0.46**	**0.39**	**0.64**	**0.74**	**1.02**
	CV %	**1.77**		**1.00**	**3.92**	**0.82**	**1.00**	**1.38**
Day 6	**PTT-302-1**	61.86	**302-1**	45.44	9.97	77.48	75.10	72.87
	PTT-302-2	62.76	**302-2**	45.01	9.94	76.87	74.38	72.96
	PTT-302-3		**302-3**	44.96	9.90	76.80	74.30	73.05
	PTT-302-4		**302-4**	45.19	10.32	77.13	74.68	71.97
	average	**62.31**		**45.15**	**10.03**	**77.07**	**74.62**	**72.71**
	confidence limit	**5.72**		**0.35**	**0.31**	**0.49**	**0.57**	**0.79**
	SD	**0.64**		**0.22**	**0.19**	**0.31**	**0.36**	**0.50**
	CV %	**1.02**		**0.48**	**1.94**	**0.40**	**0.48**	**0.69**

DAY 1			
PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
37.83	62.17	23.48	33.86
37.99	62.01	22.85	33.94
48.27	51.73	29.04	28.31
65.38	34.62	40.12	19.35
71.62	28.38	43.28	15.61
83.29	16.71	50.83	9.21
100.00	0.00	61.74	0.00

DAY 2			
PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
42.99	57.01	24.02	31.07
49.17	50.83	27.43	27.53
54.32	45.68	32.74	24.24
60.25	39.75	36.63	21.41
69.57	30.43	42.33	16.43
80.31	19.69	46.45	10.21
100.00	0.00	60.06	0.00

DAY 3			
PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
% PTT	% PET	J/g	J/g
0.00	100.00	0.00	
38.38	61.62	25.45	38.62
52.13	47.87	35.72	29.16
59.83	40.17	41.74	24.25
71.52	28.48	50.46	17.91
79.96	20.04	54.99	11.90
100.00	0.00	68.23	0.00

DAY 4			
PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
36.19	63.81	21.49	35.05
42.95	57.05	26.99	32.26
59.54	40.46	36.43	22.26
75.00	25.00	46.58	14.02
76.84	23.16	45.20	15.03
82.34	17.66	51.48	9.78
100.00	0.00	62.08	0.00

DAY 5			
PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
36.96	63.04	22.32	35.61
49.54	50.46	30.80	28.12
64.56	35.44	39.66	18.77
74.61	25.39	47.22	13.88
100.00	0.00	61.94	0.00

DAY 6			
PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
29.81	70.19	17.01	35.02
52.50	47.50	30.67	25.52
71.75	28.25	42.55	15.43
77.60	22.40	45.79	12.36
100.00	0.00	61.27	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	PTT-309-1	62.16	309-1	46.44	13.56	75.96	75.22	75.25
	PTT-309-2	61.32	309-2	47.39	13.83	77.51	76.76	74.76
	PTT-309-3	61.59	309-3					
	PTT-309-4	61.88	309-4	46.87	13.58	76.66	75.92	75.21
	average	**61.74**		**46.90**	**13.66**	**76.71**	**75.97**	**75.07**
	confidence limit	**0.58**		**1.18**	**0.37**	**1.93**	**1.91**	**0.68**
	SD	**0.36**		**0.48**	**0.15**	**0.78**	**0.77**	**0.27**
	CV %	**0.59**		**1.01**	**1.10**	**1.01**	**1.01**	**0.37**
Day 2	PTT-309-1	59.47	309-1	45.53	13.56	76.92	78.09	74.85
	PTT-309-2	59.38	309-2	47.10	13.09	79.47	80.78	75.72
	PTT-309-3	60.89	309-3	45.95	13.42	77.61	78.81	75.11
	PTT-309-4	60.51	309-4	45.86	13.62	77.46	78.66	74.74
	average	**60.06**		**46.11**	**13.42**	**77.87**	**79.08**	**75.10**
	confidence limit	**1.20**		**1.09**	**0.38**	**1.77**	**1.87**	**0.70**
	SD	**0.75**		**0.68**	**0.24**	**1.11**	**1.17**	**0.44**
	CV %	**1.25**		**1.48**	**1.77**	**1.43**	**1.48**	**0.59**
Day 3	PTT-309-1	66.17	309-1	51.21	14.96	74.36	75.06	75.75
	PTT-309-2	69.53	309-2	49.65	14.95	72.09	72.77	75.76
	PTT-309-3	69.15	309-3	50.17	15.22	72.85	73.54	75.32
	PTT-309-4	68.05	309-4	51.28	14.83	74.46	75.16	75.96
	average	**68.23**		**50.58**	**14.99**	**73.44**	**74.13**	**75.70**
	confidence limit	**2.40**		**1.27**	**0.26**	**1.85**	**1.87**	**0.42**
	SD	**1.51**		**0.80**	**0.16**	**1.16**	**1.17**	**0.27**
	CV %	**2.21**		**1.58**	**1.10**	**1.58**	**1.58**	**0.35**
Day 4	PTT-309-1	61.86	309-1	47.48	13.88	76.58	76.49	75.02
	PTT-309-2	61.30	309-2	47.98	12.81	77.39	77.29	76.94
	PTT-309-3	62.94	309-3	46.70	13.74	75.32	75.23	75.27
	PTT-309-4	62.21	309-4	46.39	13.19	74.82	74.73	76.26
	average	**62.08**		**47.14**	**13.41**	**76.03**	**75.93**	**75.87**
	confidence limit	**1.09**		**1.15**	**0.79**	**1.86**	**1.86**	**1.42**
	SD	**0.69**		**0.73**	**0.50**	**1.17**	**1.17**	**0.89**
	CV %	**1.11**		**1.54**	**3.70**	**1.54**	**1.54**	**1.18**
Day 5	PTT-309-1	62.85	309-1	47.50	13.73	76.49	76.69	75.30
	PTT-309-2	61.19	309-2	48.08	13.84	77.42	77.63	75.10
	PTT-309-3	62.18	309-3	48.22	13.82	77.65	77.85	75.14
	PTT-309-4	61.53	309-4	48.53	13.75	78.15	78.35	75.27
	average	**61.94**		**48.08**	**13.79**	**77.43**	**77.63**	**75.20**
	confidence limit	**1.17**		**0.69**	**0.08**	**1.11**	**1.11**	**0.15**
	SD	**0.73**		**0.43**	**0.05**	**0.69**	**0.70**	**0.10**
	CV %	**1.19**		**0.90**	**0.39**	**0.90**	**0.90**	**0.13**
Day 6	PTT-309-1	63.25	309-1	47.57	13.71	79.50	78.55	75.45
	PTT-309-2	60.29	309-2	48.87	13.44	81.67	80.69	75.96
	PTT-309-3	62.97	309-3	48.09	13.73	80.36	79.40	75.42
	PTT-309-4	58.55	309-4	48.41	13.47	80.90	79.93	75.91
	average	**61.27**		**48.24**	**13.59**	**80.61**	**79.64**	**75.69**
	confidence limit	**3.58**		**0.87**	**0.24**	**1.45**	**1.44**	**0.46**
	SD	**2.25**		**0.55**	**0.15**	**0.91**	**0.90**	**0.29**
	CV %	**3.67**		**1.13**	**1.13**	**1.13**	**1.13**	**0.38**

DAY 1			
PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
25.36	74.64	15.03	39.09
43.50	56.50	28.06	27.72
58.10	41.90	35.00	21.48
69.69	30.31	41.72	16.66
78.21	21.79	47.47	11.40
100.00	0.00	59.39	0.00

DAY 2			
PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.96	69.04	18.63	35.48
42.86	57.14	26.75	30.75
49.83	50.17	30.94	26.38
54.48	45.52	34.69	24.14
71.26	28.74	43.49	15.39
78.62	21.38	50.18	11.67
100	0.00	63.09	0.00

DAY 3			
PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
23.36	76.64	16.04	45.31
37.43	62.57	26.40	37.69
43.98	56.02	31.91	33.99
57.88	42.12	41.32	25.44
69.18	30.82	50.21	18.17
76.25	23.75	54.62	14.57
100.00	0.00	71.93	0.00

DAY 4			
PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
23.54	76.46	13.49	41.98
37.12	62.88	22.28	33.54
48.36	51.64	31.29	27.60
54.59	45.41	34.55	24.30
61.90	38.10	39.09	20.45
79.22	20.78	50.75	11.08
100.00	0.00	62.80	0.00

DAY 5			
PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
46.15	53.85	29.44	29.71
57.81	42.19	37.92	23.05
64.31	35.69	40.94	20.09
76.60	23.40	48.09	12.55
100.00	0.00	63.11	0.00

DAY 6			
PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.85	69.15	20.07	37.82
59.06	40.94	37.89	21.95
69.87	30.13	45.25	15.95
70.44	29.56	44.61	15.65
100.00	0.00	63.23	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	PTT-311-1	59.17	311-1	42.06		69.82	70.82	
	PTT-311-2	61.59	311-2	44.25	15.79	73.46	74.50	69.88
	PTT-311-3	59.51	311-3	43.34	15.94	71.95	72.97	69.59
	PTT-311-4	57.30	311-4	41.93	15.68	69.60	70.60	70.09
	average	**59.39**		**42.90**	**15.80**	**71.21**	**72.22**	**69.85**
	confidence limit	**2.80**		**1.76**	**0.32**	**2.92**	**2.96**	**0.62**
	SD	**1.76**		**1.10**	**0.13**	**1.83**	**1.86**	**0.25**
	CV %	**2.96**		**2.58**	**0.83**	**2.58**	**2.58**	**0.36**
Day 2	PTT-311-1		311-1	45.38	16.08	72.34	71.93	69.46
	PTT-311-2	63.12	311-2	45.15	16.34	71.98	71.56	68.96
	PTT-311-3	63.13	311-3	45.17	16.00	72.01	71.60	69.61
	PTT-311-4	63.02	311-4	45.06	15.99	71.83	71.42	69.63
	average	**63.09**		**45.19**	**16.10**	**72.04**	**71.63**	**69.42**
	confidence limit	**0.15**		**0.22**	**0.26**	**0.34**	**0.34**	**0.49**
	SD	**0.06**		**0.14**	**0.16**	**0.22**	**0.21**	**0.31**
	CV %	**0.10**		**0.30**	**1.01**	**0.30**	**0.30**	**0.45**
Day 3	PTT-311-1	70.92	311-1	48.98	17.60	68.20	68.10	70.62
	PTT-311-2	72.38	311-2	49.38	17.59	68.76	68.65	70.63
	PTT-311-3	72.07	311-3	50.29	18.26	70.02	69.92	69.52
	PTT-311-4	72.34	311-4	48.49	17.15	67.52	67.42	71.37
	average	**71.93**		**49.29**	**17.65**	**68.62**	**68.52**	**70.53**
	confidence limit	**1.09**		**1.21**	**0.73**	**1.69**	**1.69**	**1.22**
	SD	**0.69**		**0.76**	**0.46**	**1.06**	**1.06**	**0.76**
	CV %	**0.95**		**1.55**	**2.59**	**1.55**	**1.55**	**1.08**
Day 4	PTT-311-1	63.34	311-1	44.95	16.16	71.24	71.58	70.06
	PTT-311-2	62.36	311-2	45.10	15.97	71.47	71.82	70.41
	PTT-311-3	64.77	311-3	44.23	15.71	70.10	70.43	70.89
	PTT-311-4	61.26	311-4	43.40	15.02	68.78	69.11	72.17
	average	**62.93**		**44.42**	**15.72**	**70.40**	**70.74**	**70.88**
	confidence limit	**2.37**		**1.24**	**0.79**	**1.96**	**1.97**	**1.47**
	SD	**1.49**		**0.78**	**0.50**	**1.23**	**1.24**	**0.92**
	CV %	**2.37**		**1.75**	**3.17**	**1.75**	**1.75**	**1.30**
Day 5	PTT-311-1	65.30	311-1	46.47	16.69	71.49	73.64	69.72
	PTT-311-2	61.68	311-2	45.62	16.18	71.84	72.29	70.64
	PTT-311-3	62.72	311-3	46.81	16.65	73.72	74.18	69.79
	PTT-311-4	62.72	311-4	46.83	16.97	73.75	74.21	69.21
	average	**63.11**		**46.43**	**16.62**	**72.70**	**73.58**	**69.84**
	confidence limit	**2.46**		**0.90**	**0.52**	**1.91**	**1.43**	**0.95**
	SD	**1.54**		**0.57**	**0.33**	**1.20**	**0.90**	**0.59**
	CV %	**2.45**		**1.22**	**1.97**	**1.65**	**1.22**	**0.85**
Day 6	PTT-311-1	63.66	311-1	45.26	15.96	70.98	71.58	70.49
	PTT-311-2	61.33	311-2	42.40	14.99	66.50	67.06	72.29
	PTT-311-3	63.23	311-3	45.02	16.01	70.61	71.20	70.40
	PTT-311-4	64.69	311-4	47.20	16.89	74.03	74.65	68.77
	average	**63.23**		**44.97**	**15.96**	**70.53**	**71.12**	**70.49**
	confidence limit	**2.24**		**3.14**	**1.24**	**4.92**	**4.96**	**2.28**
	SD	**1.41**		**1.97**	**0.78**	**3.09**	**3.12**	**1.44**
	CV %	**2.22**		**4.38**	**4.86**	**4.38**	**4.38**	**2.04**

DAY 1

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
26.62	73.38	14.93	
42.80	57.20	25.46	36.08
53.97	46.03	31.73	26.76
64.72	35.28	38.07	20.64
74.19	25.81	44.20	16.54
78.52	21.48	47.01	12.64
100.00	0.00	60.71	0.00

DAY 2

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
35.56	64.44	21.46	36.09
52.67	47.33	33.68	26.74
62.18	37.82	38.79	20.64
69.91	30.09	45.68	16.54
76.83	23.17	52.40	12.64
81.49	18.51	55.91	10.06
100	0.00	66.16	0.00

DAY 3

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
25.09	74.91	16.95	45.17
32.94	67.06	22.71	40.02
43.17	56.83	28.56	32.84
50.00	50.00	33.81	28.95
66.82	33.18	44.86	19.07
79.43	20.57	55.30	11.81
100.00	0.00	70.59	0.00

DAY 4

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
31.07	68.93	19.00	35.40
49.31	50.69	29.94	26.81
63.86	36.14	27.38	26.39
75.99	24.01	48.63	12.22
76.86	23.14	47.46	11.51
84.21	15.79	52.56	7.24
100.00	0.00	61.58	0.00

DAY 5

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
43.32	56.68	25.53	30.27
50.18	49.82	30.90	26.35
61.06	38.94	36.30	20.37
81.41	18.59	51.75	9.31
100.00	0.00	61.23	0.00

DAY 6

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
43.53	56.47	22.04	27.36
55.67	44.33	29.79	22.83
73.89	26.11	41.83	12.97
82.13	17.87	50.22	8.85
100.00	0.00	59.71	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	**PTT-312-1**	60.16	**312-1**	39.98	21.47	66.92	65.86	63.33
	PTT-312-2	59.29	**312-2**	38.21	22.05	63.96	62.94	62.44
	PTT-312-3	61.92	**312-3**	39.13	22.28	65.50	64.46	62.09
	PTT-312-4	61.45	**312-4**	38.69	22.36	64.76	63.73	61.96
	average	**60.71**		**39.00**	**22.04**	**65.29**	**64.25**	**62.46**
	confidence limit	**1.91**		**1.20**	**0.64**	**2.00**	**1.97**	**0.98**
	SD	**1.20**		**0.75**	**0.40**	**1.26**	**1.24**	**0.62**
	CV %	**1.98**		**1.93**	**1.82**	**1.93**	**1.93**	**0.99**
Day 2	**PTT-312-1**	65.84	**312-1**	41.10	21.49	63.04	62.12	61.40
	PTT-312-2	67.66	**312-2**	43.19	22.03	66.01	65.28	60.43
	PTT-312-3	64.11	**312-3**	41.98	22.29	64.29	63.45	59.96
	PTT-312-4	67.02	**312-4**	41.81	21.01	64.05	63.20	62.26
	average	**66.16**		**42.02**	**21.71**	**64.35**	**63.52**	**61.01**
	confidence limit	**2.48**		**1.38**	**0.91**	**1.96**	**2.09**	**1.63**
	SD	**1.56**		**0.87**	**0.57**	**1.23**	**1.31**	**1.03**
	CV %	**2.36**		**2.07**	**2.63**	**1.91**	**2.07**	**1.68**
Day 3	**PTT-312-1**	71.77	**312-1**	45.70	21.50	66.17	64.74	63.61
	PTT-312-2	71.30	**312-2**	47.76	21.01	69.16	67.66	64.44
	PTT-312-3	69.31	**312-3**	47.85	20.99	69.29	67.79	64.47
	PTT-312-4	69.96	**312-4**	47.09	20.85	68.19	66.71	64.71
	average	**70.59**		**47.10**	**21.09**	**68.20**	**66.73**	**64.31**
	confidence limit	**1.82**		**1.58**	**0.45**	**2.29**	**2.24**	**0.77**
	SD	**1.14**		**0.99**	**0.28**	**1.44**	**1.41**	**0.48**
	CV %	**1.62**		**2.11**	**1.35**	**2.11**	**2.11**	**0.75**
Day 4	**PTT-312-1**	59.97	**312-1**	39.50	18.82	63.56	64.15	63.47
	PTT-312-2	62.11	**312-2**	40.22	19.28	64.71	65.32	62.58
	PTT-312-3	61.16	**312-3**	40.70	19.08	65.49	66.10	62.97
	PTT-312-4	63.07	**312-4**	40.89	19.47	65.79	66.40	62.21
	average	**61.58**		**40.33**	**19.16**	**64.89**	**65.49**	**62.81**
	confidence limit	**2.11**		**0.99**	**0.44**	**1.59**	**1.60**	**0.86**
	SD	**1.33**		**0.62**	**0.28**	**1.00**	**1.01**	**0.54**
	CV %	**2.15**		**1.54**	**1.45**	**1.54**	**1.54**	**0.86**
Day 5	**PTT-312-1**	62.34	**312-1**	39.53	20.48	64.61	64.56	61.26
	PTT-312-2	62.09	**312-2**	38.81	20.23	63.47	63.39	61.73
	PTT-312-3		**312-3**	40.45	19.87	66.07	66.07	62.41
	PTT-312-4	59.25	**312-4**	38.99	19.83	63.76	63.68	62.49
	average	**61.23**		**39.45**	**20.10**	**64.48**	**64.42**	**61.97**
	confidence limit	**2.73**		**1.17**	**0.49**	**1.86**	**1.91**	**0.93**
	SD	**1.72**		**0.74**	**0.31**	**1.17**	**1.20**	**0.59**
	CV %	**2.80**		**1.87**	**1.54**	**1.81**	**1.87**	**0.94**
Day 6	**PTT-312-1**	57.89	**312-1**	39.77	19.90	70.39	66.61	60.88
	PTT-312-2	58.86	**312-2**	39.68	19.86	70.26	66.45	60.96
	PTT-312-3	63.60	**312-3**	39.23	20.42	69.58	65.70	59.86
	PTT-312-4	58.49	**312-4**	36.80	18.64	65.92	61.63	63.36
	average	**59.71**		**38.87**	**19.71**	**69.04**	**65.10**	**61.26**
	confidence limit	**4.18**		**2.23**	**1.20**	**3.36**	**3.73**	**2.36**
	SD	**2.62**		**1.40**	**0.75**	**2.11**	**2.34**	**1.48**
	CV %	**4.39**		**3.60**	**3.83**	**3.06**	**3.60**	**2.42**

SIGMOIDAL FIXED RANGE INTEGRATION

Quantification based on PTT and PET peak integration

DAY 1

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
26.17	73.83	17.12	30.29
34.96	65.04	22.47	25.89
48.29	51.71	30.81	19.93
59.64	40.36	36.97	15.47
70.06	29.94	45.44	11.35
77.44	22.56	47.93	8.49
100.00	0.00	65.40	0.00

DAY 2

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.95	69.05	19.01	26.87
53.82	46.18	33.73	17.66
57.08	42.92	35.47	14.61
70.87	29.13	42.56	10.28
75.88	24.12	47.67	9.05
79.95	20.05	50.04	7.44
100	0.00	63.81	0.00

DAY 3

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
35.24	64.76	25.18	28.57
52.65	47.35	37.12	19.68
62.90	37.10	44.27	15.25
71.13	28.87	49.31	12.24
73.90	26.10	51.59	10.79
79.66	20.34	55.36	8.30
100.00	0.00	73.16	0.00

DAY 4

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
34.15	65.85	22.02	25.94
45.12	54.88	28.63	20.64
55.30	44.70	34.78	16.86
62.06	37.94	39.88	14.44
72.14	27.86	45.62	10.56
77.58	22.42	48.69	8.67
100.00	0.00	65.88	0.00

DAY 5

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.58	69.42	18.55	26.76
48.19	51.81	29.83	19.01
58.97	41.03	35.88	14.96
80.72	19.28	48.22	6.71
100.00	0.00	62.67	0.00

DAY 6

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
32.63	67.37	20.81	26.90
53.62	46.38	31.08	16.90
63.85	36.15	39.13	13.53
72.73	27.27	44.02	9.93
81.84	18.16	51.25	7.11
100.00	0.00	64.18	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	**PTT-302-1**	65.24	**302-1**	47.68	10.01	73.90	72.91	73.04
	PTT-302-2	65.20	**302-2**	48.13	10.16	74.60	73.59	72.65
	PTT-302-3	64.33	**302-3**	47.97	9.83	74.35	73.35	73.49
	PTT-302-4	66.83	**302-4**	47.54	10.02	73.68	72.69	73.01
	average	**65.40**		**47.83**	**10.01**	**74.13**	**73.13**	**73.05**
	confidence limit	**1.66**		**0.43**	**0.21**	**0.66**	**0.65**	**0.55**
	SD	**1.04**		**0.27**	**0.13**	**0.42**	**0.41**	**0.34**
	CV %	**1.59**		**0.56**	**1.34**	**0.56**	**0.56**	**0.47**
Day 2	**PTT-302-1**	63.51	**302-1**	46.95	9.92	75.07	75.68	74.06
	PTT-302-2	63.21	**302-2**	47.25	9.82	75.55	76.16	74.30
	PTT-302-3	62.77	**302-3**	45.96	9.90	73.49	74.08	74.11
	PTT-302-4	65.73	**302-4**	47.27	10.20	75.58	76.19	73.31
	average	**63.81**		**46.86**	**9.96**	**74.92**	**75.53**	**73.94**
	confidence limit	**2.10**		**0.98**	**0.26**	**1.57**	**1.58**	**0.69**
	SD	**1.32**		**0.62**	**0.17**	**0.98**	**0.99**	**0.43**
	CV %	**2.07**		**1.31**	**1.67**	**1.31**	**1.31**	**0.59**
Day 3	**PTT-302-1**	73.73	**302-1**	55.35	11.68	77.98	78.11	71.85
	PTT-302-2	73.10	**302-2**	54.72	11.01	77.15	77.23	73.46
	PTT-302-3	72.44	**302-3**	56.05	11.56	78.91	79.10	72.14
	PTT-302-4	73.35	**302-4**	54.07	11.55	76.29	76.31	72.16
	average	**73.16**		**55.05**	**11.45**	**77.58**	**77.69**	**72.40**
	confidence limit	**0.86**		**1.35**	**0.48**	**1.79**	**1.91**	**1.15**
	SD	**0.54**		**0.85**	**0.30**	**1.12**	**1.20**	**0.72**
	CV %	**0.74**		**1.54**	**2.61**	**1.45**	**1.54**	**1.00**
Day 4	**PTT-302-1**	65.83	**302-1**	46.79	9.81	72.95	71.02	74.45
	PTT-302-2	66.38	**302-2**	46.66	9.97	72.75	70.82	74.04
	PTT-302-3	65.30	**302-3**	47.30	9.97	73.74	71.79	74.04
	PTT-302-4	66.02	**302-4**	46.61	9.35	72.67	70.75	75.64
	average	**65.88**		**46.84**	**9.77**	**73.03**	**71.10**	**74.54**
	confidence limit	**0.72**		**0.50**	**0.46**	**0.78**	**0.76**	**1.21**
	SD	**0.45**		**0.32**	**0.29**	**0.49**	**0.48**	**0.76**
	CV %	**0.68**		**0.67**	**2.98**	**0.67**	**0.67**	**1.02**
Day 5	**PTT-302-1**	61.11	**302-1**	47.71	9.48	77.68	76.13	73.03
	PTT-302-2	62.48	**302-2**	48.13	10.08	78.36	76.80	71.41
	PTT-302-3	63.64	**302-3**	47.17	10.22	76.80	75.26	71.04
	PTT-302-4	63.46	**302-4**	48.05	9.37	78.23	76.67	73.30
	average	**62.67**		**47.77**	**9.79**	**77.77**	**76.21**	**72.19**
	confidence limit	**1.85**		**0.69**	**0.68**	**1.13**	**1.11**	**1.81**
	SD	**1.16**		**0.44**	**0.43**	**0.71**	**0.70**	**1.14**
	CV %	**1.85**		**0.91**	**4.34**	**0.91**	**0.91**	**1.57**
Day 6	**PTT-302-1**	64.03	**302-1**	47.30	10.01	76.24	73.70	72.59
	PTT-302-2	64.32	**302-2**	47.04	9.96	75.86	73.30	72.70
	PTT-302-3		**302-3**	47.31	9.92	76.26	73.72	72.83
	PTT-302-4		**302-4**	47.67	10.34	76.78	74.28	71.73
	average	**64.18**		**47.33**	**10.06**	**76.29**	**73.75**	**72.46**
	confidence limit	**0.33**		**0.41**	**0.31**	**0.60**	**0.64**	**0.79**
	SD	**0.21**		**0.26**	**0.19**	**0.38**	**0.40**	**0.50**
	CV %	**0.32**		**0.55**	**1.91**	**0.50**	**0.55**	**0.68**

DAY 1

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
37.83	62.17	24.56	34.55
37.99	62.01	24.18	34.38
48.27	51.73	30.36	28.84
65.38	34.62	41.26	19.86
71.62	28.38	44.36	16.15
83.29	16.71	51.73	9.64
100.00	0.00	62.76	0.00

DAY 2

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
42.99	57.01	25.09	31.81
49.17	50.83	28.33	28.12
54.32	45.68	33.37	24.85
60.25	39.75	37.12	22.05
69.57	30.43	42.83	16.94
80.31	19.69	47.01	10.63
100	0.00	58.31	0.00

DAY 3

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
38.38	61.62	26.85	39.30
52.13	47.87	37.25	30.06
59.83	40.17	42.85	25.40
71.52	28.48	51.46	18.48
79.96	20.04	55.83	12.35
100.00	0.00	68.84	0.00

DAY 4

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
36.19	63.81	22.60	35.65
42.95	57.05	28.26	32.63
59.54	40.46	37.42	22.56
75.00	25.00	47.51	14.41
82.34	17.66	52.03	10.05
100.00	0.00	62.58	0.00

DAY 5

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
36.96	63.04	23.51	36.02
49.54	50.46	31.80	28.54
64.56	35.44	40.98	19.10
74.61	25.39	48.15	14.31
100.00	0.00	62.41	0.00

DAY 6

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
29.81	70.19	18.38	35.63
52.50	47.50	32.20	25.22
71.75	28.25	43.91	16.02
77.60	22.40	47.37	12.88
100.00	0.00	63.11	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	PTT-309-1	63.14	309-1	47.72	14.19	76.13	76.04	74.60
	PTT-309-2	62.29	309-2	48.70	14.29	77.70	77.60	74.42
	PTT-309-3	62.97	309-3					
	PTT-309-4	62.63	309-4	48.16	14.01	76.83	76.74	74.92
	average	62.76		48.19	14.16	76.89	76.79	74.65
	confidence limit	0.60		0.78	0.23	1.25	1.24	0.40
	SD	0.38		0.49	0.14	0.78	0.78	0.25
	CV %	0.60		1.02	1.00	1.02	1.02	0.34
Day 2	PTT-309-1	57.54	309-1	44.12	13.99	74.57	75.67	74.70
	PTT-309-2	57.54	309-2	45.37	13.49	76.80	77.81	75.60
	PTT-309-3	59.16	309-3	44.05	13.79	74.44	75.55	75.06
	PTT-309-4	58.98	309-4	44.75	13.99	75.69	76.75	74.70
	average	58.31		44.57	13.82	75.37	76.45	75.01
	confidence limit	1.41		0.98	0.38	1.75	1.69	0.68
	SD	0.89		0.62	0.24	1.10	1.06	0.43
	CV %	1.52		1.39	1.71	1.46	1.39	0.57
Day 3	PTT-309-1	66.57	309-1	52.31	15.36	74.47	75.99	75.78
	PTT-309-2	70.06	309-2	50.52	15.25	71.92	73.39	75.96
	PTT-309-3	69.97	309-3	51.01	15.01	72.62	74.10	76.34
	PTT-309-4	68.75	309-4	52.18	15.17	74.29	75.80	76.08
	average	68.84		51.51	15.20	73.33	74.82	76.04
	confidence limit	2.59		1.40	0.23	1.99	2.03	0.37
	SD	1.63		0.88	0.15	1.25	1.28	0.23
	CV %	2.36		1.71	0.97	1.71	1.71	0.31
Day 4	PTT-309-1	62.09	309-1	48.26	14.13	76.48	77.11	74.96
	PTT-309-2	61.94	309-2	48.49	13.23	76.85	77.48	76.56
	PTT-309-3	63.47	309-3	47.37	13.87	75.07	75.69	75.43
	PTT-309-4	62.83	309-4	47.31	13.58	74.98	75.60	75.94
	average	62.58		47.86	13.70	75.84	76.47	75.72
	confidence limit	1.13		0.96	0.62	1.53	1.54	1.09
	SD	0.71		0.61	0.39	0.96	0.97	0.69
	CV %	1.13		1.26	2.82	1.26	1.26	0.91
Day 5	PTT-309-1	63.26	309-1	48.50	13.95	76.55	77.71	75.27
	PTT-309-2	61.50	309-2	49.07	14.14	77.45	78.62	74.94
	PTT-309-3	62.72	309-3	49.29	14.06	77.79	78.97	75.08
	PTT-309-4	62.17	309-4	49.18	13.97	77.62	78.80	75.24
	average	62.41		49.01	14.03	77.35	78.53	75.13
	confidence limit	1.20		0.56	0.14	0.88	0.90	0.25
	SD	0.75		0.35	0.09	0.56	0.56	0.16
	CV %	1.21		0.72	0.62	0.72	0.72	0.21
Day 6	PTT-309-1	64.48	309-1	49.22	14.23	79.44	77.99	74.91
	PTT-309-2	61.04	309-2	50.03	13.88	80.75	79.27	75.56
	PTT-309-3	63.81	309-3	49.57	14.24	80.00	78.55	74.89
	PTT-309-4		309-4	49.49	13.89	79.87	78.42	75.54
	average	63.11		49.58	14.06	80.02	78.56	75.22
	confidence limit	2.90		0.54	0.32	0.86	0.85	0.60
	SD	1.82		0.34	0.20	0.54	0.53	0.38
	CV %	2.89		0.68	1.44	0.68	0.68	0.50

DAY 1

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
25.36	74.64	16.33	39.28
43.50	56.50	29.52	28.12
58.10	41.90	36.24	21.70
69.69	30.31	42.91	15.83
78.21	21.79	48.57	11.53
100.00	0.00	61.69	0.00

DAY 2

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.96	69.04	19.81	35.68
42.86	57.14	28.04	30.98
49.83	50.17	32.17	26.7
54.48	45.52	35.98	24.41
71.26	28.74	44.85	15.58
78.62	21.38	51.46	11.87
100	0.00	64.26	0.00

DAY 3

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
23.36	76.64	17.69	45.46
37.43	62.57	28.06	37.80
43.98	56.02	33.16	34.18
57.88	42.12	42.50	25.56
69.18	30.82	51.52	18.40
76.25	23.75	55.62	14.69
100.00	0.00	72.83	0.00

DAY 4

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
23.54	76.46	14.85	42.02
37.12	62.88	23.77	33.69
48.36	51.64	32.46	27.69
54.59	45.41	35.77	24.39
61.90	38.10	40.20	20.57
79.22	20.78	51.68	11.20
100.00	0.00	64.83	0.00

DAY 5

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
46.15	53.85	30.40	29.81
57.81	42.19	38.70	23.22
64.31	35.69	41.85	20.27
76.60	23.40	49.00	12.74
100.00	0.00	63.16	0.00

DAY 6

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.85	69.15	21.54	37.87
59.06	40.94	39.45	22.27
69.87	30.13	46.15	16.19
70.44	29.56	45.87	15.80
100.00	0.00	63.82	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	PTT-311-1	61.18	311-1	43.29		68.74	70.18	
	PTT-311-2	60.44	311-2	45.46	15.90	72.40	73.69	69.26
	PTT-311-3	62.71	311-3	44.81	16.06	71.30	72.64	68.95
	PTT-311-4	62.42	311-4	43.18	15.78	68.56	70.00	69.49
	average	61.69		44.19	15.91	70.25	71.63	69.23
	confidence limit	1.69		1.80	0.22	3.03	2.91	0.43
	SD	1.06		1.13	0.14	1.90	1.83	0.27
	CV %	1.72		2.56	0.88	2.71	2.56	0.39
Day 2	PTT-311-1		311-1	46.82	16.17	72.53	72.86	69.56
	PTT-311-2	64.07	311-2	46.66	16.44	72.29	72.62	69.05
	PTT-311-3	64.22	311-3	46.58	16.10	72.16	72.49	69.69
	PTT-311-4	64.48	311-4	47.03	16.09	72.86	73.19	69.71
	average	64.26		46.77	16.20	72.46	72.79	69.50
	confidence limit	0.33		0.32	0.26	0.49	0.49	0.49
	SD	0.21		0.20	0.16	0.31	0.31	0.31
	CV %	0.32		0.42	1.01	0.42	0.42	0.44
Day 3	PTT-311-1	71.62	311-1	49.83	17.65	67.75	68.42	70.67
	PTT-311-2	73.68	311-2	50.33	17.65	68.43	69.10	70.67
	PTT-311-3	72.81	311-3	51.32	18.32	69.78	70.46	69.55
	PTT-311-4	73.22	311-4	49.44	17.21	67.22	67.88	71.40
	average	72.83		50.23	17.71	68.29	68.97	70.57
	confidence limit	1.41		1.29	0.73	1.76	1.78	1.21
	SD	0.88		0.81	0.46	1.11	1.12	0.76
	CV %	1.21		1.62	2.59	1.62	1.62	1.08
Day 4	PTT-311-1	63.78	311-1	45.80	16.19	70.32	70.65	70.10
	PTT-311-2	63.22	311-2	45.82	16.03	70.35	70.68	70.39
	PTT-311-3	65.28	311-3	45.02	15.75	69.12	69.45	70.91
	PTT-311-4	67.03	311-4	44.10	15.08	67.71	68.03	72.15
	average	64.83		45.19	15.76	69.38	69.70	70.89
	confidence limit	2.72		1.29	0.78	1.99	2.00	1.44
	SD	1.71		0.81	0.49	1.25	1.26	0.91
	CV %	2.63		1.80	3.11	1.80	1.80	1.28
Day 5	PTT-311-1	65.58	311-1	47.42	16.77	72.96	75.08	69.77
	PTT-311-2	61.39	311-2	46.31	16.22	71.13	73.32	70.76
	PTT-311-3	62.68	311-3	47.92	16.71	73.79	75.87	69.88
	PTT-311-4	63.00	311-4	47.61	17.03	73.28	75.38	69.30
	average	63.16		47.32	16.68	72.79	74.91	69.93
	confidence limit	2.79		1.12	0.54	1.84	1.77	0.97
	SD	1.76		0.70	0.34	1.16	1.11	0.61
	CV %	2.78		1.48	2.03	1.59	1.48	0.87
Day 6	PTT-311-1	64.22	311-1	46.64	16.09	70.55	73.08	70.44
	PTT-311-2	62.35	311-2	43.79	15.07	65.91	68.61	72.32
	PTT-311-3	63.66	311-3	46.38	16.13	70.12	72.67	70.37
	PTT-311-4	65.05	311-4	49.12	17.01	74.62	76.97	68.75
	average	63.82		46.48	16.08	70.30	72.83	70.47
	confidence limit	1.80		3.47	1.26	5.66	5.43	2.32
	SD	1.13		2.18	0.79	3.56	3.41	1.46
	CV %	1.78		4.69	4.93	5.06	4.69	2.07

DAY 1			
PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
26.62	73.38	15.94	10.15
42.80	57.20	26.22	36.00
53.97	46.03	32.59	26.79
64.72	35.28	38.83	20.64
74.19	25.81	44.75	16.60
78.52	21.48	47.64	12.67
100.00	0.00	59.75	0.00

DAY 2			
PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
35.56	64.44	22.03	36.02
52.67	47.33	34.36	26.78
62.18	37.82	39.23	20.64
69.91	30.09	46.13	16.6
76.83	23.17	52.80	12.66
81.49	18.51	56.38	10.13
100	0.00	66.09	0.00

DAY 3			
PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
25.09	74.91	18.35	45.33
32.94	67.06	23.74	40.20
43.17	56.83	29.88	33.07
50.00	50.00	34.62	29.14
66.82	33.18	45.40	19.27
79.43	20.57	55.90	11.92
100.00	0.00	71.32	0.00

DAY 4			
PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
31.07	68.93	19.94	35.43
49.31	50.69	30.80	26.85
75.99	24.01	49.29	12.34
76.86	23.14	47.93	11.62
84.21	15.79	53.07	7.37
100.00	0.00	61.79	0.00

DAY 5			
PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
43.32	56.68	26.37	30.33
50.18	49.82	31.55	26.44
61.06	38.94	36.90	20.39
81.41	18.59	52.24	9.35
100.00	0.00	61.68	0.00

DAY 6			
PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
43.53	56.47	22.82	27.46
55.67	44.33	31.72	23.45
73.89	26.11	42.49	13.11
82.13	17.87	50.74	8.91
100.00	0.00	59.17	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	**PTT-312-1**	59.36	**312-1**	40.90	21.57	67.92	68.45	64.01
	PTT-312-2	62.19	**312-2**	39.15	22.00	65.01	65.52	63.32
	PTT-312-3	59.88	**312-3**	40.20	22.26	66.76	67.28	62.90
	PTT-312-4	57.58	**312-4**	39.67	22.34	65.88	66.39	62.78
	average	**59.75**		**39.98**	**22.04**	**66.39**	**66.91**	**63.25**
	confidence limit	**3.02**		**1.19**	**0.55**	**1.98**	**1.99**	**0.88**
	SD	**1.90**		**0.75**	**0.35**	**1.24**	**1.25**	**0.55**
	CV %	**3.18**		**1.87**	**1.57**	**1.87**	**1.87**	**0.88**
Day 2	**PTT-312-1**	65.82	**312-1**	41.78	21.58	63.51	63.22	61.24
	PTT-312-2	67.45	**312-2**	43.89	21.98	66.56	66.41	60.52
	PTT-312-3	64.13	**312-3**	42.93	22.27	65.18	64.96	60.00
	PTT-312-4	66.95	**312-4**	43.05	21.04	65.35	65.14	62.21
	average	**66.09**		**42.91**	**21.72**	**65.15**	**64.93**	**61.00**
	confidence limit	**2.34**		**1.38**	**0.85**	**2.00**	**2.09**	**1.52**
	SD	**1.47**		**0.87**	**0.53**	**1.26**	**1.31**	**0.96**
	CV %	**2.23**		**2.02**	**2.45**	**1.93**	**2.02**	**1.57**
Day 3	**PTT-312-1**	72.34	**312-1**	46.78	21.51	66.57	68.35	63.79
	PTT-312-2	71.44	**312-2**	48.75	20.99	69.38	68.35	64.66
	PTT-312-3	71.03	**312-3**	48.75	21.00	69.38	67.44	64.65
	PTT-312-4	70.48	**312-4**	48.10	20.86	68.45	67.43	64.88
	average	**71.32**		**48.10**	**21.09**	**68.44**	**67.89**	**64.49**
	confidence limit	**1.25**		**1.48**	**0.46**	**2.10**	**0.84**	**0.77**
	SD	**0.78**		**0.93**	**0.29**	**1.32**	**0.53**	**0.48**
	CV %	**1.10**		**1.93**	**1.36**	**1.93**	**0.78**	**0.75**
Day 4	**PTT-312-1**	60.55	**312-1**	40.41	18.78	64.31	65.40	63.65
	PTT-312-2	62.30	**312-2**	41.14	19.25	65.47	66.58	62.74
	PTT-312-3	61.17	**312-3**	41.44	19.07	65.95	67.07	63.09
	PTT-312-4	63.13	**312-4**	41.85	19.43	66.60	67.73	62.39
	average	**61.79**		**41.21**	**19.13**	**65.58**	**66.70**	**62.96**
	confidence limit	**1.83**		**0.97**	**0.44**	**1.54**	**1.56**	**0.85**
	SD	**1.15**		**0.61**	**0.28**	**0.97**	**0.98**	**0.54**
	CV %	**1.86**		**1.47**	**1.45**	**1.47**	**1.47**	**0.85**
Day 5	**PTT-312-1**	62.48	**312-1**	40.44	20.45	65.00	65.56	61.41
	PTT-312-2	62.42	**312-2**	39.73	20.27	63.85	64.41	61.75
	PTT-312-3		**312-3**	41.19	19.89	66.20	66.78	62.46
	PTT-312-4	60.15	**312-4**	39.98	19.83	64.26	64.81	62.58
	average	**61.68**		**40.34**	**20.11**	**64.83**	**65.39**	**62.05**
	confidence limit	**2.11**		**1.02**	**0.48**	**1.64**	**1.65**	**0.90**
	SD	**1.33**		**0.64**	**0.30**	**1.03**	**1.04**	**0.56**
	CV %	**2.15**		**1.59**	**1.49**	**1.59**	**1.59**	**0.91**
Day 6	**PTT-312-1**	58.60	**312-1**	40.78	19.90	71.92	68.92	60.73
	PTT-312-2	59.69	**312-2**	40.77	19.88	71.90	68.90	60.77
	PTT-312-3	59.22	**312-3**	40.43	20.42	71.36	68.33	59.64
	PTT-312-4		**312-4**	37.72	18.62	66.99	63.75	63.39
	average	**59.17**		**39.93**	**19.71**	**70.54**	**67.48**	**61.13**
	confidence limit	**0.87**		**2.35**	**1.22**	**3.79**	**3.98**	**2.53**
	SD	**0.55**		**1.48**	**0.77**	**2.38**	**2.50**	**1.59**
	CV %	**0.92**		**3.70**	**3.88**	**3.38**	**3.70**	**2.61**

PERPENDICULAR DROP FIXED RANGE INTEGRATION

Quantification based on PTT and PET peak integration

DAY 1

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
26.17	73.83	21.11	35.30
34.96	65.04	25.11	29.97
48.29	51.71	32.99	23.41
59.64	40.36	38.68	18.27
70.06	29.94	46.32	13.48
77.44	22.56	48.19	9.97
100.00	0.00	62.35	0.00

DAY 2

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
% PTT	% PET	J/g	J/g
30.95	69.05	22.16	31.65
53.82	46.18	35.46	21.07
57.08	42.92	37.08	17.83
70.87	29.13	43.45	12.49
75.88	24.12	48.38	11.08
79.95	20.05	50.61	9.047
100	0.00	61.19	0.00

DAY 3

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
35.24	64.76	29.05	34.15
52.65	47.35	39.35	24.12
62.90	37.10	45.71	18.56
71.13	28.87	50.23	15.18
73.90	26.10	51.88	12.64
79.66	20.34	55.92	10.42
100.00	0.00	71.63	0.00

DAY 4

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
34.15	65.85	25.02	31.32
45.12	54.88	31.17	25.19
55.30	44.70	37.09	20.92
62.06	37.94	41.70	18.07
72.14	27.86	46.87	13.13
77.58	22.42	49.58	10.77
100.00	0.00	63.96	0.00

DAY 5

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.58	69.42	21.65	32.11
48.19	51.81	32.50	23.55
58.97	41.03	37.61	18.73
80.72	19.28	48.66	8.35
100.00	0.00	59.73	0.00

DAY 6

PTT 302 %	PET 302 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
32.63	67.37	24.06	32.03
53.62	46.38	33.61	21.53
63.85	36.15	40.77	16.62
72.73	27.27	44.79	11.86
81.84	18.16	51.44	8.63
100.00	0.00	61.62	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	**PTT-302-1**	62.41	**302-1**	48.28	11.69	74.17	77.43	74.77
	PTT-302-2	62.15	**302-2**	48.73	11.88	74.99	78.16	74.36
	PTT-302-3	61.53	**302-3**	48.63	11.85	74.81	78.00	74.43
	PTT-302-4	63.31	**302-4**	48.24	11.89	74.09	77.37	74.34
	average	**62.35**		**48.47**	**11.83**	**74.51**	**77.74**	**74.48**
	confidence limit	**1.18**		**0.39**	**0.15**	**0.72**	**0.63**	**0.32**
	SD	**0.74**		**0.25**	**0.09**	**0.45**	**0.40**	**0.20**
	CV %	**1.18**		**0.51**	**0.79**	**0.61**	**0.51**	**0.27**
Day 2	**PTT-302-1**	60.76	**302-1**	47.60	11.92	74.82	77.79	73.78
	PTT-302-2	61.04	**302-2**	47.66	11.71	74.93	77.89	74.25
	PTT-302-3	60.09	**302-3**	46.61	11.86	73.06	76.18	73.92
	PTT-302-4	62.86	**302-4**	47.90	12.15	75.36	78.28	73.28
	average	**61.19**		**47.44**	**11.91**	**74.54**	**77.54**	**73.81**
	confidence limit	**1.88**		**0.91**	**0.29**	**1.61**	**1.48**	**0.64**
	SD	**1.18**		**0.57**	**0.18**	**1.01**	**0.93**	**0.40**
	CV %	**1.94**		**1.20**	**1.53**	**1.36**	**1.20**	**0.54**
Day 3	**PTT-302-1**	72.29	**302-1**	56.00	13.96	78.17	78.18	72.92
	PTT-302-2	71.23	**302-2**	54.92	13.13	76.52	76.67	74.53
	PTT-302-3	70.81	**302-3**	55.51	13.95	77.42	77.50	72.94
	PTT-302-4	72.19	**302-4**	54.84	13.69	76.40	76.56	73.45
	average	**71.63**		**55.32**	**13.68**	**77.13**	**77.23**	**73.46**
	confidence limit	**1.16**		**0.87**	**0.62**	**1.32**	**1.21**	**1.20**
	SD	**0.73**		**0.54**	**0.39**	**0.83**	**0.76**	**0.75**
	CV %	**1.01**		**0.98**	**2.84**	**1.08**	**0.98**	**1.03**
Day 4	**PTT-302-1**	63.70	**302-1**	47.63	11.83	72.48	74.47	74.84
	PTT-302-2	64.51	**302-2**	47.45	11.73	72.18	74.18	75.05
	PTT-302-3	63.43	**302-3**	48.15	12.11	73.37	75.28	74.25
	PTT-302-4	64.21	**302-4**	47.31	11.78	71.94	73.97	74.95
	average	**63.96**		**47.64**	**11.86**	**72.49**	**74.47**	**74.77**
	confidence limit	**0.78**		**0.58**	**0.27**	**1.00**	**0.91**	**0.58**
	SD	**0.49**		**0.37**	**0.17**	**0.63**	**0.57**	**0.36**
	CV %	**0.76**		**0.77**	**1.43**	**0.87**	**0.77**	**0.48**
Day 5	**PTT-302-1**	58.46	**302-1**	48.39	12.33	78.29	81.01	73.08
	PTT-302-2	59.42	**302-2**	48.72	12.28	78.94	81.57	73.19
	PTT-302-3	60.57	**302-3**	47.70	12.46	76.94	79.86	72.80
	PTT-302-4	60.47	**302-4**	48.82	12.10	79.14	81.73	73.59
	average	**59.73**		**48.41**	**12.29**	**78.33**	**81.04**	**73.17**
	confidence limit	**1.58**		**0.81**	**0.24**	**1.58**	**1.35**	**0.52**
	SD	**0.99**		**0.51**	**0.15**	**0.99**	**0.85**	**0.33**
	CV %	**1.66**		**1.05**	**1.21**	**1.27**	**1.05**	**0.44**
Day 6	**PTT-302-1**	61.95	**302-1**	47.89	11.77	75.78	77.72	74.81
	PTT-302-2	61.41	**302-2**	47.46	11.80	75.03	77.02	74.74
	PTT-302-3	61.09	**302-3**	47.63	11.88	75.32	77.30	74.57
	PTT-302-4	62.03	**302-4**	47.93	12.45	75.85	77.78	73.35
	average	**61.62**		**47.73**	**11.98**	**75.49**	**77.45**	**74.37**
	confidence limit	**0.71**		**0.35**	**0.51**	**0.62**	**0.57**	**1.09**
	SD	**0.45**		**0.22**	**0.32**	**0.39**	**0.36**	**0.69**
	CV %	**0.73**		**0.47**	**2.67**	**0.52**	**0.47**	**0.92**

DAY 1

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
37.83	62.17	34.06	23.66
37.99	62.01	23.81	34.86
48.27	51.73	29.58	28.83
65.38	34.62	40.57	19.78
71.62	28.38	43.55	15.87
83.29	16.71	50.90	9.27
100.00	0.00	61.74	0.00

DAY 2

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
42.99	57.01	24.79	32.22
49.17	50.83	28.37	28.52
54.32	45.68	33.26	25.05
60.25	39.75	36.89	21.89
69.57	30.43	42.67	16.95
80.31	19.69	46.82	10.7
100	0.00	60.01	0.00

DAY 3

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
38.38	61.62	26.30	40.15
52.13	47.87	36.28	30.75
59.83	40.17	40.76	
71.52	28.48	50.74	18.54
79.96	20.04	55.08	12.18
100.00	0.00	68.21	0.00

DAY 4

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
42.95	57.05	28.12	33.32
59.54	40.46	37.08	22.89
75.00	25.00	46.92	14.34
76.84	23.16	45.01	14.87
82.34	17.66	51.59	9.89
100.00	0.00	62.09	0.00

DAY 5

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
36.96	63.04	23.15	36.43
49.54	50.46	31.26	28.60
64.56	35.44	39.94	18.70
74.61	25.39	47.54	14.20
100.00	0.00	61.94	0.00

DAY 6

PTT 309 %	PET 309 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
29.81	70.19	17.81	36.07
52.50	47.50	31.26	25.21
63.33	36.67	35.08	17.82
71.75	28.25	42.99	15.98
77.60	22.40	46.59	13.22
100.00	0.00	61.20	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	**PTT-309-1**	62.16	**309-1**	47.00	14.33	76.44	76.13	74.47
	PTT-309-2	61.32	**309-2**	47.72	14.15	77.61	77.29	74.80
	PTT-309-3	61.59	**309-3**					
	PTT-309-4	61.88	**309-4**	47.27	13.96	76.87	76.57	75.13
	average	**61.74**		**47.33**	**14.15**	**76.97**	**76.66**	**74.80**
	confidence limit	**0.58**		**0.90**	**0.46**	**1.47**	**1.46**	**0.82**
	SD	**0.36**		**0.36**	**0.19**	**0.59**	**0.59**	**0.33**
	CV %	**0.59**		**0.77**	**1.31**	**0.77**	**0.77**	**0.44**
Day 2	**PTT-309-1**	59.42	**309-1**	45.73	13.83	75.98	76.20	75.19
	PTT-309-2	59.33	**309-2**	47.49	13.53	78.90	79.14	75.73
	PTT-309-3	60.84	**309-3**	46.60	14.08	77.42	77.65	74.74
	PTT-309-4	60.45	**309-4**	46.19	14.00	76.74	76.97	74.88
	average	**60.01**		**46.50**	**13.86**	**77.26**	**77.49**	**75.13**
	confidence limit	**1.20**		**1.19**	**0.39**	**1.98**	**1.98**	**0.69**
	SD	**0.75**		**0.75**	**0.24**	**1.24**	**1.25**	**0.44**
	CV %	**1.25**		**1.61**	**1.76**	**1.61**	**1.61**	**0.58**
Day 3	**PTT-309-1**	66.17	**309-1**	51.31	15.26	74.39	75.22	76.39
	PTT-309-2	69.51	**309-2**	49.83	15.22	72.25	73.05	76.45
	PTT-309-3	69.13	**309-3**	51.99	17.17	75.38	76.22	73.43
	PTT-309-4	68.04	**309-4**	51.53	15.26	74.71	75.54	76.39
	average	**68.21**		**51.17**	**15.73**	**74.18**	**75.01**	**75.66**
	confidence limit	**2.38**		**1.49**	**1.53**	**2.15**	**2.18**	**2.37**
	SD	**1.50**		**0.93**	**0.96**	**1.35**	**1.37**	**1.49**
	CV %	**2.20**		**1.83**	**6.12**	**1.83**	**1.83**	**1.97**
Day 4	**PTT-309-1**	61.87	**309-1**	47.80	14.19	76.39	76.99	75.73
	PTT-309-2	61.31	**309-2**	47.47	12.34	75.87	76.45	78.92
	PTT-309-3	62.95	**309-3**	47.21	14.21	75.45	76.03	75.70
	PTT-309-4	62.23	**309-4**	46.46	13.26	74.25	74.83	77.34
	average	**62.09**		**47.24**	**13.50**	**75.49**	**76.08**	**76.92**
	confidence limit	**1.09**		**0.91**	**1.42**	**1.45**	**1.46**	**2.44**
	SD	**0.69**		**0.57**	**0.89**	**0.91**	**0.92**	**1.54**
	CV %	**1.11**		**1.21**	**6.60**	**1.21**	**1.21**	**2.00**
Day 5	**PTT-309-1**	62.85	**309-1**	48.19	14.36	77.09	75.15	73.26
	PTT-309-2	61.19	**309-2**	48.48	14.19	77.56	75.61	73.56
	PTT-309-3	62.18	**309-3**	48.75	14.32	77.99	76.03	73.33
	PTT-309-4	61.53	**309-4**	48.88	14.08	78.20	76.23	73.76
	average	**61.94**		**48.58**	**14.24**	**77.71**	**75.75**	**73.48**
	confidence limit	**1.17**		**0.49**	**0.20**	**0.78**	**0.76**	**0.36**
	SD	**0.73**		**0.31**	**0.13**	**0.49**	**0.48**	**0.23**
	CV %	**1.19**		**0.63**	**0.90**	**0.63**	**0.63**	**0.31**
Day 6	**PTT-309-1**	63.18	**309-1**	46.00	14.13	77.13	75.16	75.10
	PTT-309-2	60.23	**309-2**	49.04	13.65	81.84	80.13	76.00
	PTT-309-3	62.92	**309-3**	48.38	14.08	80.82	79.05	75.20
	PTT-309-4	58.48	**309-4**	48.51	13.63	81.02	79.26	76.03
	average	**61.20**		**47.98**	**13.87**	**80.21**	**78.40**	**75.58**
	confidence limit	**3.58**		**2.15**	**0.43**	**3.33**	**3.52**	**0.80**
	SD	**2.25**		**1.35**	**0.27**	**2.10**	**2.21**	**0.50**
	CV %	**3.68**		**2.82**	**1.94**	**2.61**	**2.82**	**0.66**

DAY 1

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
25.36	74.64	16.13	40.06
43.50	56.50	28.57	28.22
58.10	41.90	35.86	22.30
69.69	30.31	42.47	16.36
78.21	21.79	48.21	12.11
100.00	0.00	59.47	0.00

DAY 2

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.96	69.04	19.57	36.49
42.86	57.14	27.50	31.48
49.83	50.17	31.40	26.91
54.48	45.52	35.32	24.79
71.26	28.74	44.32	16.19
78.62	21.38	50.79	12.28
100	0.00	63.09	0.00

DAY 3

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
23.36	76.64	17.48	46.71
37.43	62.57	27.82	39.11
43.98	56.02	32.62	34.74
57.88	29.48	42.09	26.20
69.18	30.82	50.31	18.21
76.25	23.75	55.01	14.95
100.00	0.00	72.06	0.00

DAY 4

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
23.54	76.46	15.42	43.78
37.12	62.88	23.52	34.70
48.36	51.64	32.51	28.75
54.59	45.41	35.68	25.36
61.90	38.10	40.01	21.32
79.22	20.78	51.27	11.58
100.00	0.00	62.96	0.00

DAY 5

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
46.15	53.85	30.34	30.47
57.81	42.19	38.21	23.25
64.31	35.69	41.47	20.53
76.60	23.40	48.27	12.76
100.00	0.00	63.11	0.00

DAY 6

PTT 311 %	PET 311 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
30.85	69.15	22.10	39.65
59.06	40.94	38.64	22.62
69.87	30.13	45.31	15.98
70.44	29.56	45.32	16.28
100.00	0.00	63.23	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	PTT-311-1	59.17	311-1	42.43	13.68	68.38	71.35	74.01
	PTT-311-2	61.59	311-2	44.69	16.19	72.34	75.15	69.25
	PTT-311-3	59.51	311-3	44.07	16.62	71.25	74.11	68.43
	PTT-311-4	57.30	311-4	42.61	16.32	68.69	71.65	69.00
	average	59.39		43.45	15.70	70.16	73.07	70.18
	confidence limit	2.80		1.76	2.16	3.09	2.96	4.11
	SD	1.76		1.11	1.36	1.94	1.86	2.58
	CV %	2.96		2.54	8.66	2.76	2.54	3.68
Day 2	PTT-311-1	63.12	311-1	46.21	16.92	72.78	73.24	68.74
	PTT-311-2	63.13	311-2	45.91	17.10	72.31	72.77	68.40
	PTT-311-3	63.02	311-3	45.70	16.32	71.98	72.44	69.84
	PTT-311-4		311-4	46.57	17.44	73.35	73.82	67.78
	average	63.09		46.10	16.95	72.61	73.07	68.69
	confidence limit	0.10		0.60	0.75	0.95	0.95	1.38
	SD	0.06		0.38	0.47	0.60	0.60	0.87
	CV %	0.10		0.82	2.77	0.82	0.82	1.26
Day 3	PTT-311-1	70.79	311-1	49.33	17.87	67.96	68.45	70.97
	PTT-311-2	72.19	311-2	49.97	18.27	68.84	69.34	70.32
	PTT-311-3	71.90	311-3	50.76	18.75	69.93	70.44	69.54
	PTT-311-4	72.10	311-4	49.03	17.70	67.54	68.04	71.25
	average	71.75		49.77	18.15	68.57	69.07	70.52
	confidence limit	1.03		1.22	0.74	1.68	1.69	1.21
	SD	0.65		0.77	0.47	1.06	1.06	0.76
	CV %	0.90		1.54	2.58	1.54	1.54	1.08
Day 4	PTT-311-1	63.36	311-1	45.70	16.86	71.07	72.59	69.98
	PTT-311-2	62.40	311-2	45.53	16.38	70.81	72.32	70.84
	PTT-311-3	64.78	311-3	44.86	16.30	69.77	71.25	70.98
	PTT-311-4	61.30	311-4	43.68	15.39	67.93	69.38	72.60
	average	62.96		44.94	16.23	69.90	71.38	71.10
	confidence limit	2.35		1.46	0.98	2.27	2.32	1.74
	SD	1.48		0.92	0.61	1.43	1.46	1.09
	CV %	2.35		2.04	3.78	2.04	2.04	1.54
Day 5	PTT-311-1	65.29	311-1	46.89	17.09	72.85	74.30	69.59
	PTT-311-2	61.71	311-2	46.02	16.61	71.42	72.92	70.44
	PTT-311-3	62.71	311-3	47.55	17.34	73.93	75.35	69.14
	PTT-311-4	62.72	311-4	47.20	17.28	73.36	74.79	69.25
	average	63.11		46.92	17.08	72.89	74.34	69.60
	confidence limit	2.43		1.04	0.53	1.72	1.65	0.94
	SD	1.53		0.65	0.33	1.08	1.04	0.59
	CV %	2.42		1.40	1.94	1.48	1.40	0.85
Day 6	PTT-311-1	63.66	311-1	45.86	16.52	70.79	72.53	70.60
	PTT-311-2	61.33	311-2	43.42	15.92	66.68	68.67	71.67
	PTT-311-3	63.23	311-3	45.70	16.57	70.52	72.28	70.52
	PTT-311-4	64.69	311-4	48.43	18.01	75.16	76.60	67.95
	average	63.23		45.85	16.76	70.79	72.52	70.19
	confidence limit	2.24		3.26	1.41	5.51	5.15	2.51
	SD	1.41		2.05	0.89	3.47	3.24	1.58
	CV %	2.22		4.47	5.30	4.90	4.47	2.25

DAY 1

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
26.62	73.38	15.49	38.33
42.80	57.20	25.53	29.59
53.97	46.03	32.09	24.39
64.72	35.28	38.30	18.23
74.19	25.81	44.56	13.33
78.52	21.48	47.40	11.36
100.00	0.00	59.28	0.00

DAY 2

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
35.56	64.44	22.67	37.35
52.67	47.33	34.49	27.6
62.18	37.82	39.42	21.28
69.91	30.09	46.02	16.9
76.83	23.17	52.82	13.08
81.49	18.51	56.04	10.2
100	0.00	66.09	0.00

DAY 3

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
25.09	74.91	18.42	46.48
32.94	67.06	23.39	40.62
43.17	56.83	29.48	33.64
50.00	50.00	34.14	29.21
66.82	33.18	44.82	19.08
79.43	20.57	54.94	11.51
100.00	0.00	70.81	0.00

DAY 4

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
31.07	68.93	20.20	36.69
49.31	50.69	30.93	27.80
63.86	36.14	28.76	27.39
75.99	24.01	48.93	12.54
76.86	23.14	47.66	11.73
84.21	15.79	52.37	7.01
100.00	0.00	61.35	0.00

DAY 5

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
43.32	56.68	26.47	31.16
50.18	49.82	31.52	26.93
61.06	38.94	37.08	21.12
81.41	18.59	52.32	10.08
100.00	0.00	61.23	0.00

DAY 6

PTT 312 %	PET 312 %	PTT enthalpy J/g	PET enthalpy J/g
0.00	100.00	0.00	
43.53	56.47	22.75	27.99
55.67	44.33	30.06	23.04
73.89	26.11	42.20	13.44
82.13	17.87	50.63	9.46
100.00	0.00	59.21	0.00

	JRC code	PTT enthalpy J/g	JRC code	PTT enthalpy J/g	PET enthalpy J/g	PTT % calibration	PTT % single point	PTT % calibration (PET)
Day 1	PTT-312-1	59.15	312-1	40.49	19.80	67.90	68.30	62.05
	PTT-312-2	61.38	312-2	39.32	20.62	65.94	66.33	60.48
	PTT-312-3	59.40	312-3	40.27	20.68	67.53	67.93	60.36
	PTT-312-4	57.19	312-4	39.90	21.32	66.91	67.31	59.13
	average	59.28		40.00	20.61	67.07	67.47	60.50
	confidence limit	2.73		0.81	0.99	1.37	1.37	1.90
	SD	1.71		0.51	0.62	0.86	0.86	1.19
	CV %	2.89		1.28	3.02	1.28	1.28	1.97
Day 2	PTT-312-1	65.84	312-1	41.21	21.60	62.29	62.35	62.40
	PTT-312-2	67.59	312-2	44.58	23.47	67.18	67.45	59.14
	PTT-312-3	64.04	312-3	43.43	23.79	65.51	65.71	58.58
	PTT-312-4	66.89	312-4	43.58	24.20	65.73	65.94	57.87
	average	66.09		43.20	23.27	65.18	65.37	59.50
	confidence limit	2.46		2.26	1.83	3.29	3.42	3.18
	SD	1.54		1.42	1.15	2.07	2.15	2.00
	CV %	2.34		3.29	4.94	3.17	3.29	3.36
Day 3	PTT-312-1	71.75	312-1	47.11	22.81	67.82	66.53	60.38
	PTT-312-2	71.31	312-2	49.44	22.59	71.18	69.82	60.73
	PTT-312-3	70.20	312-3	49.01	22.05	70.56	69.22	61.60
	PTT-312-4	69.97	312-4	48.29	21.93	69.52	68.20	61.80
	average	70.81		48.46	22.35	69.77	68.44	61.13
	confidence limit	1.37		1.62	0.67	2.33	2.29	1.08
	SD	0.86		1.02	0.42	1.47	1.44	0.68
	CV %	1.21		2.10	1.89	2.10	2.10	1.11
Day 4	PTT-312-1	59.72	312-1	41.00	20.42	65.51	66.83	61.64
	PTT-312-2	61.92	312-2	41.62	20.77	66.68	67.84	60.98
	PTT-312-3	60.93	312-3	41.73	20.20	66.85	68.02	62.05
	PTT-312-4	62.84	312-4	42.48	21.16	68.06	69.24	60.25
	average	61.35		41.71	20.64	66.77	67.98	61.23
	confidence limit	2.13		0.97	0.67	1.66	1.57	1.26
	SD	1.34		0.61	0.42	1.04	0.99	0.79
	CV %	2.18		1.46	2.04	1.56	1.46	1.29
Day 5	PTT-312-1	62.34	312-1	41.07	21.91	65.49	64.40	59.79
	PTT-312-2	62.09	312-2	40.06	21.42	63.89	62.82	60.69
	PTT-312-3		312-3	41.33	20.67	65.90	64.81	62.07
	PTT-312-4	59.25	312-4	40.39	21.11	64.41	63.33	61.26
	average	61.23		40.71	21.28	64.92	63.84	60.95
	confidence limit	2.73		0.94	0.83	1.49	1.47	1.52
	SD	1.72		0.59	0.52	0.93	0.92	0.96
	CV %	2.80		1.45	2.45	1.44	1.45	1.57
Day 6	PTT-312-1	57.89	312-1	36.61	18.86	64.84	61.83	62.82
	PTT-312-2	58.87	312-2	34.67	17.71	61.80	58.55	65.08
	PTT-312-3	61.60	312-3	36.15	19.29	64.12	61.05	61.97
	PTT-312-4	58.49	312-4	38.06	19.66	67.09	64.28	61.24
	average	59.21		36.37	18.88	64.46	61.43	62.78
	confidence limit	2.61		2.22	1.35	3.46	3.75	2.65
	SD	1.64		1.40	0.85	2.18	2.36	1.67
	CV %	2.77		3.84	4.48	3.38	3.84	2.66

Annex VI

Mechanical properties and elasticity

Elongation at break of pure PTT samples - Instron dynamometer

JRC code	Load at break mN	Elong. at break %	Elong. at maximum load %	Maximum load mN
293-1	2427.15	56.50	56.50	2598.32
293-2	2335.32	50.70	50.70	2458.21
293-3	2486.48	56.20	56.20	2496.28
293-4	2440.03	50.47	50.47	2512.56
293-5	2383.98	50.63	50.63	2489.62
293-6	2407.05	49.37	49.37	2587.24
293-7	2458.61	52.77	52.77	2633.22
293-8	2417.10	53.07	52.07	2418.94
293-9	2349.66	48.10	48.10	2489.66
293-10	2381.81	51.33	50.33	2391.97
average	**2408.72**	**51.91**	**51.71**	**2507.60**
confidence limit	**33.84**	**1.97**	**1.97**	**55.83**
SD	**47.30**	**2.75**	**2.76**	**78.05**
CV %	**1.96**	**5.30**	**5.33**	**3.11**

JRC code	Load at break mN	Elong. at break %	Elong. at maximum load %	Maximum load mN
294-1	1719.41	49.97	49.30	1734.19
294-2	1363.84	48.07	43.00	1686.36
294-3	1578.17	38.67	37.67	1584.53
294-4	1050.12	46.93	40.23	1585.00
294-5	1450.73	39.77	38.50	1505.43
294-6	1288.59	47.37	42.10	1650.92
294-7	1628.05	44.03	43.37	1642.25
294-8	1716.12	52.30	48.90	1771.74
294-9	1518.65	47.63	41.87	1590.64
294-10	1594.03	42.23	41.10	1620.82
average	**1490.77**	**45.70**	**42.60**	**1637.19**
confidence limit	**149.63**	**3.15**	**2.77**	**56.14**
SD	**209.16**	**4.41**	**3.87**	**78.48**
CV %	**14.03**	**9.65**	**9.10**	**4.79**

JRC code	Load at break mN	Elong. at break %	Elong. at maximum load %	Maximum load mN
295-1	2563.87	33.83	32.83	2572.69
295-2	2509.23	32.40	32.40	2509.23
295-3	2470.58	32.77	32.77	2470.58
295-4	2415.15	30.20	29.20	2426.96
295-5	2441.21	29.40	29.40	2441.21
295-6	2291.50	32.47	30.93	2317.47
295-7	2265.91	32.53	30.43	2364.26
295-8	2590.62	34.47	33.47	2599.44
295-9	2272.37	36.80	34.67	2544.89
295-10	2475.58	34.67	34.67	2475.58
295-11	2573.67	33.10	32.37	2588.02
average	**2442.70**	**32.97**	**32.10**	**2482.76**
confidence limit	**80.59**	**1.37**	**1.28**	**61.48**
SD	**119.96**	**2.04**	**1.90**	**91.51**
CV %	**4.91**	**6.20**	**5.91**	**3.69**

JRC code	Load at break mN	Elong. at break %	Elong. at maximum load %	Maximum load mN
296-1	2040.11	44.30	40.80	2485.87
296-2	2570.93	38.87	38.87	2570.93
296-3	2363.24	41.30	38.27	2530.59
296-4	2594.71	44.77	44.13	2642.68
296-5	2304.69	44.10	40.17	2539.74
296-6	2577.49	43.00	43.00	2577.49
296-7	2481.80	38.97	38.47	2513.52
296-8	2348.04	45.40	42.53	2653.51
296-9	2522.04	46.10	44.57	2544.43
296-10	2154.45	46.80	42.73	2636.21
average	**2395.75**	**43.36**	**41.35**	**2569.50**
confidence limit	**135.52**	**1.79**	**1.50**	**36.85**
SD	**189.44**	**2.66**	**2.23**	**54.85**
CV %	**7.91**	**6.13**	**5.39**	**2.13**

JRC code	Load at break mN	Elong. at break %	Elong. at maximum load %	Maximum load mN
297-1	8392.89	103.83	76.47	23613.69
297-2	4313.03	101.63	75.00	23238.75
297-3	4494.52	99.83	63.63	23194.74
297-4	2890.76	85.87	64.90	23355.19
297-5	21425.79	72.27	60.67	23224.92
297-6	1632.46	100.97	66.50	23834.30
297-7	890.38	480.23	72.70	23482.00
297-8	2978.78	87.27	66.43	24536.68
297-9	22466.43	69.23	67.23	23280.52
297-10	5277.43	96.57	60.37	22309.16
average	**7476.25**	**129.77**	**67.39**	**23407.00**
confidence limit	**5656.12**	**88.52**	**4.03**	**401.67**
SD	**7906.71**	**123.75**	**5.63**	**561.50**
CV %	**105.76**	**95.36**	**8.35**	**2.40**

JRC code	Load at break mN	Elong. at break %	Elong. at maximum load %	Maximum load mN
300-1	1564.93	64.27	62.97	1643.68
300-2	1655.31	65.87	63.90	1725.52
300-3	1569.08	69.37	65.57	1701.83
300-4	1541.22	64.87	61.30	1643.77
300-5	1516.98	56.50	56.50	1516.98
300-6	1631.12	64.30	64.30	1631.12
300-7	1489.68	61.70	58.40	1545.85
300-8	1641.02	64.77	63.93	1669.25
300-9	1521.34	60.33	56.47	1579.60
300-10	1716.90	65.17	65.17	1716.90
average	**1584.76**	**63.72**	**61.85**	**1637.45**
confidence limit	**52.21**	**2.49**	**2.51**	**50.92**
SD	**72.99**	**3.49**	**3.50**	**71.18**
CV %	**4.61**	**5.47**	**5.66**	**4.35**

JRC code	Load at break mN	Elong. at break %	Elong. at maximum load %	Maximum load mN
317-1	2384.82	43.70	43.70	2456.30
317-2	2469.41	45.90	45.90	2354.84
317-3	2218.83	38.23	38.23	2285.41
317-4	2458.65	47.73	47.73	2574.24
317-5	2362.06	44.47	43.47	2367.85
317-6	2502.10	48.00	48.00	2598.62
317-7	2492.14	47.53	47.53	2522.14
317-8	2381.13	42.53	42.53	2473.54
317-9	2369.03	43.13	43.13	2399.13
317-10	2376.57	46.43	46.43	2397.75
average	**2401.47**	**44.77**	**44.67**	**2442.98**
confidence limit	**60.09**	**2.17**	**2.19**	**71.95**
SD	**84.00**	**3.03**	**3.06**	**100.58**
CV %	**3.50**	**6.77**	**6.84**	**4.12**

Elongation at break of PTT manually separated from fabrics - Instron dynamometer

JRC code	Load at break mN	Elong. at break %	Elong. at maximum load %	Maximum load mN
301-1	2455.22	60.73	60.73	2675.02
301-2	2489.70	57.00	57.00	2541.24
301-3	2509.32	59.97	59.97	2721.54
301-4	2549.61	60.20	60.20	2648.11
301-5	2463.62	57.67	57.67	2463.62
301-6	2495.90	60.53	60.53	2552.65
301-7	2492.98	59.70	59.70	2623.54
301-8	2473.18	60.13	59.13	2611.47
301-9	2510.37	58.63	58.63	2674.16
301-10	2458.39	57.90	57.90	2488.98
average	**2489.83**	**59.25**	**59.15**	**2600.03**
confidence limit	**20.75**	**0.95**	**0.93**	**61.04**
SD	**29.01**	**1.33**	**1.30**	**85.32**
CV %	**1.17**	**2.25**	**2.19**	**3.28**

JRC code	Load at break mN	Elong. at break %	Elong. at maximum load %	Maximum load mN
302-1	2112.72	70.87	56.00	2254.21
302-2	2326.60	65.67	59.50	2451.47
302-3	2184.73	70.97	55.00	2204.67
302-4	2311.94	59.67	59.67	2400.51
302-5	2203.73	70.20	57.53	2226.54
302-6	2179.34	68.70	60.63	2264.88
302-7	2203.11	62.17	60.10	2106.52
302-8	2369.68	121.40	114.10	2456.34
302-9	2010.34	58.30	47.03	2084.55
302-10	2185.48	67.30	56.57	2235.00
average	**2208.77**	**71.53**	**62.61**	**2268.47**
confidence limit	**75.81**	**12.96**	**13.25**	**93.40**
SD	**105.97**	**18.11**	**18.52**	**130.57**
CV %	**4.80**	**25.33**	**29.58**	**5.76**

JRC code	Load at break mN	Elong. at break %	Elong. at maximum load %	Maximum load mN
309-1	2919.82	75.50	75.50	3012.55
309-2	2709.06	69.17	69.17	2698.41
309-3	2860.95	74.23	74.23	2914.66
309-4	2859.62	75.10	75.10	2975.07
309-5	2674.14	72.83	72.83	2705.10
309-6	2813.72	73.97	73.97	2952.28
309-7	2640.91	69.93	69.93	2641.23
309-8	2792.62	74.90	74.90	2845.54
309-9	2798.53	76.13	76.13	2763.63
309-10	2746.68	71.40	71.40	2802.52
average	**2781.61**	**73.32**	**73.32**	**2831.10**
confidence limit	**63.43**	**1.72**	**1.72**	**92.48**
SD	**88.66**	**2.41**	**2.41**	**129.28**
CV %	**3.19**	**3.28**	**3.28**	**4.57**

JRC code	Load at break mN	Elong. at break %	Elong. at maximum load %	Maximum load mN
311-1	2205.59	86.47	69.27	2289.89
311-2	2100.00	167.53	59.00	2154.03
311-3	2154.68	126.63	63.47	2203.85
311-4	2157.39	112.70	70.90	2145.66
311-5	2375.27	74.53	67.80	2475.89
311-6	2324.76	130.97	70.97	2399.54
311-7	2164.06	61.43	57.37	2223.74
311-8	2436.83	84.20	72.73	2501.79
311-9	2308.50	125.47	66.13	2365.58
311-10	2433.24	82.33	71.90	2425.87
average	**2266.03**	**105.23**	**66.95**	**2318.58**
confidence limit	**89.26**	**23.42**	**3.87**	**94.95**
SD	**124.78**	**32.74**	**5.41**	**132.73**
CV %	**5.51**	**31.11**	**8.08**	**5.72**

JRC code	Load at break mN	Elong. at break %	Elong. at maximum load %	Maximum load mN
312-1	2370.72	71.97	63.40	2432.25
312-2	2404.13	76.70	73.13	2404.13
312-3	2344.19	116.37	58.80	2455.81
312-4	2216.71	122.30	62.67	2286.41
312-5	2470.55	65.90	65.90	2530.21
312-6	2266.43	71.63	69.07	2354.70
312-7	2077.34	104.53	53.37	2072.51
312-8	2344.20	131.20	65.90	2401.88
312-9	2321.42	66.03	65.00	2425.61
312-10	2217.46	108.00	69.53	2247.55
312-11	2356.65	96.07	64.33	2388.94
average	**2301.91**	**95.87**	**64.77**	**2356.78**
confidence limit	**79.77**	**17.41**	**4.02**	**91.62**
SD	**111.52**	**24.34**	**5.62**	**128.08**
CV %	**4.84**	**25.39**	**8.67**	**5.43**

JRC code	Load at break mN	Elong. at break %	Elong. at maximum load %	Maximum load mN
322-1	2062.07	70.70	68.80	2202.19
322-2	2269.30	71.43	71.43	2269.30
322-3	2241.97	71.13	69.53	2276.22
322-4	1989.57	67.80	62.93	2163.13
322-5	1991.02	69.77	65.57	2208.20
322-6	2129.52	65.53	65.53	2129.52
322-7	2253.00	71.23	71.23	2253.00
322-8	2097.09	67.83	64.07	2117.77
322-9	2196.08	70.07	67.07	2217.49
322-10	2195.78	67.10	64.90	2225.14
average	**2142.54**	**69.26**	**67.11**	**2206.20**
confidence limit	**75.00**	**1.47**	**2.14**	**39.28**
SD	**104.84**	**2.05**	**3.00**	**54.91**
CV %	**4.89**	**2.96**	**4.47**	**2.49**

Elastic recovery of pure PTT samples - Instron dynamometer

JRC code	Force C1 cN	Force C2 cN	SD 1 %	La mm	Lb mm	Imm. rec. 1 %	Imm. PD 1 %	Rec. 1 %	PD 1 %
293-1	138.93	127.33	8.35	56.83	53.68	45.36	13.66	70.56	7.36
293-2	135.43	124.00	8.44	56.98	53.74	44.16	13.96	70.08	7.48
293-3	134.63	123.21	8.49	56.98	53.93	44.16	13.96	68.56	7.87
293-4	137.95	126.36	8.40	56.88	53.76	44.96	13.76	69.92	7.52
293-5	140.12	128.24	8.48	56.81	53.59	45.52	13.62	71.28	7.18
293-6	151.04	139.28	7.79	55.57	53.00	55.44	11.14	76.00	6.00
293-7	154.46	142.80	7.55	55.43	52.92	56.56	10.86	76.64	5.84
293-8	156.79	145.27	7.34	54.82	52.71	61.44	9.64	78.32	5.42
293-9	151.66	139.85	7.78	55.69	53.06	54.48	11.38	75.52	6.12
293-10	152.72	141.07	7.62	55.40	52.97	56.80	10.80	76.24	5.93
293-11	145.35	133.48	8.17	56.40	53.24	48.80	12.80	74.08	6.49
293-12	155.61	144.11	7.39	54.65	52.42	62.80	9.30	80.64	4.85
average	**146.22**	**134.58**	**7.98**	**56.04**	**53.25**	**51.71**	**12.07**	**73.99**	**6.51**
confidence limit	**5.33**	**5.31**	**0.29**	**0.55**	**0.30**	**4.43**	**1.11**	**2.44**	**0.61**
SD	**8.38**	**8.35**	**0.45**	**0.87**	**0.48**	**6.98**	**1.74**	**3.84**	**0.96**
CV %	**5.73**	**6.21**	**5.63**	**1.56**	**0.90**	**13.49**	**14.45**	**5.19**	**14.75**

JRC code	Force C5 cN	Force C6 cN	SD 2 %	Le mm	Lf mm	Imm. PD 3 %	Imm. rec 3 %	PD 3 %	Rec. 3 %
293-1	125.55	118.46	5.64	57.15	54.24	14.30	42.80	8.47	66.08
293-2	122.13	115.02	5.83	57.28	54.31	14.56	41.76	8.62	65.52
293-3	121.59	114.58	5.77	57.30	54.47	14.60	41.60	8.94	64.24
293-4	124.66	117.54	5.71	57.20	54.31	14.40	42.40	8.62	65.52
293-5	126.27	119.18	5.62	57.13	54.16	14.26	42.96	8.33	66.72
293-6	137.17	129.97	5.25	55.96	53.60	11.92	52.32	7.20	71.20
293-7	140.78	133.24	5.35	55.83	53.51	11.66	53.36	7.02	71.92
293-8	143.56	136.26	5.08	55.26	53.32	10.52	57.92	6.65	73.44
293-9	137.70	130.33	5.35	56.07	53.66	12.14	51.44	7.32	70.72
293-10	139.17	131.88	5.24	55.80	53.55	11.60	53.60	7.10	71.60
293-11	131.02	123.67	5.61	56.50	53.91	13.50	46.00	7.68	69.28
293-12	142.42	135.42	4.92	0.56	0.30	10.18	59.28	6.21	75.12
average	**132.67**	**125.46**	**5.45**	**0.78**	**0.42**	**12.80**	**48.79**	**7.68**	**69.28**
confidence limit	**5.28**	**5.22**	**0.18**	**1.39**	**0.78**	**1.05**	**4.18**	**0.57**	**2.27**
SD	**8.31**	**8.22**	**0.29**	**0.82**	**0.45**	**1.65**	**6.58**	**0.89**	**3.57**
CV %	**6.26**	**6.55**	**5.33**	**12.85**	**11.61**	**12.85**	**13.49**	**11.62**	**5.15**

JRC code	Force C1 cN	Force C2 cN	SD 1 %	La mm	Lb mm	Imm. rec. 1 %	Imm. PD 1 %	Rec. 1 %	PD 1 %
294-1	120.29	110.51	8.13	54.88	52.56	62.00	9.50	80.32	4.92
294-2	121.67	111.44	8.41	54.86	52.41	61.20	9.70	79.12	5.22
294-3	122.44	112.12	8.43	54.78	52.42	65.12	8.72	82.08	4.48
294-4	125.38	114.79	8.45	54.79	52.30	61.68	9.58	78.88	5.28
294-5	121.49	111.16	8.50	54.84	52.49	66.96	8.26	80.08	4.98
294-6	137.68	125.35	8.95	54.75	52.46	60.96	9.76	79.52	5.12
294-7	136.45	126.27	7.46	54.85	52.61	61.12	9.72	80.72	4.82
294-8	137.77	127.25	7.64	54.36	52.24	61.76	9.56	80.64	4.84
294-9	136.21	125.86	7.60	54.79	52.64	61.68	9.58	81.60	4.60
294-10	133.13	123.09	7.54	54.13	52.49	61.28	9.68	80.08	4.98
average	**129.25**	**118.78**	**8.11**	**54.70**	**52.46**	**62.38**	**9.41**	**80.30**	**4.92**
confidence limit	**5.43**	**5.23**	**0.37**	**0.18**	**0.09**	**1.43**	**0.36**	**0.73**	**0.18**
SD	**7.59**	**7.31**	**0.52**	**0.25**	**0.13**	**2.01**	**0.50**	**1.01**	**0.25**
CV %	**5.87**	**6.15**	**6.36**	**0.46**	**0.24**	**3.22**	**5.33**	**1.26**	**5.15**

JRC code	Force C5 cN	Force C6 cN	SD 2 %	Le mm	Lf mm	Imm. PD 3 %	Imm. rec 3 %	PD 3 %	Rec. 3 %
294-1	106.86	101.20	5.30	55.30	53.21	10.30	58.80	6.44	74.24
294-2	107.52	101.57	5.53	55.25	53.08	10.84	56.64	6.68	73.28
294-3	107.97	102.30	5.25	55.18	53.08	10.24	59.04	6.50	74.00
294-4	110.80	104.58	5.62	55.18	53.01	11.24	55.04	7.08	71.68
294-5	107.41	101.52	5.48	55.28	53.16	10.62	57.52	6.36	74.56
294-6	121.97	116.49	4.49	55.15	53.22	10.60	57.60	6.42	74.32
294-7	122.56	116.50	4.94	55.42	53.34	10.50	58.00	6.16	75.36
294-8	123.43	117.14	5.09	55.12	53.25	10.36	58.56	6.16	75.36
294-9	122.43	116.05	5.21	55.62	53.54	10.36	58.56	6.02	75.92
294-10	119.69	113.98	4.77	55.31	53.18	10.56	57.76	6.32	74.72
average	**115.06**	**109.13**	**5.17**	**55.28**	**53.21**	**10.56**	**57.75**	**6.41**	**74.34**
confidence limit	**5.34**	**5.27**	**0.25**	**0.11**	**0.11**	**0.21**	**0.85**	**0.22**	**0.86**
SD	**7.46**	**7.37**	**0.36**	**0.15**	**0.15**	**0.30**	**1.19**	**0.30**	**1.21**
CV %	**6.48**	**6.76**	**6.87**	**0.27**	**0.28**	**2.82**	**2.06**	**4.70**	**1.62**

JRC code	Force C1 cN	Force C2 cN	SD 1 %	La mm	Lb mm	Imm. rec. 1 %	Imm. PD 1 %	Rec. 1 %	PD 1 %
295-1	182.15	167.99	7.78	55.92	52.84	52.64	11.84	77.28	5.67
295-2	185.87	171.12	7.93	55.87	52.81	53.04	11.74	77.52	5.63
295-3	182.54	167.21	8.40	56.24	52.73	50.08	12.48	78.16	5.46
295-4	185.94	170.75	8.17	56.24	52.85	50.08	12.48	77.20	5.69
295-5	187.80	172.10	8.36	56.18	52.59	50.56	12.36	79.28	5.19
295-6	181.51	166.46	8.29	56.27	52.85	49.84	12.54	77.20	5.69
295-7	188.71	173.27	8.18	56.15	52.60	50.80	12.30	79.20	5.21
295-8	184.87	169.60	8.26	56.19	52.71	50.48	12.38	78.32	5.43
295-9	178.75	164.22	8.13	56.33	53.06	49.36	12.66	75.52	6.12
295-10	183.07	167.55	8.48	56.25	52.81	50.00	12.50	77.52	5.62
average	**184.12**	**169.03**	**8.20**	**56.16**	**52.79**	**50.69**	**12.33**	**77.72**	**5.57**
confidence limit	**2.19**	**2.01**	**0.15**	**0.11**	**0.10**	**0.86**	**0.22**	**0.78**	**0.19**
SD	**3.07**	**2.82**	**0.21**	**0.15**	**0.14**	**1.21**	**0.30**	**1.10**	**0.27**
CV %	**1.67**	**1.67**	**2.61**	**0.27**	**0.26**	**2.38**	**2.45**	**1.41**	**4.84**

JRC code	Force C5 cN	Force C6 cN	SD 2 %	Le mm	Lf mm	Imm. PD 3 %	Imm. rec 3 %	PD 3 %	Rec. 3 %
295-1	163.09	154.17	5.47	56.25	53.39	12.50	50.00	6.78	72.88
295-2	165.31	156.50	5.33	56.22	53.37	12.44	50.24	6.74	73.04
295-3	160.86	151.93	5.55	56.58	53.31	13.16	47.36	6.62	73.52
295-4	164.71	155.63	5.51	56.56	53.40	13.12	47.52	6.79	72.80
295-5	165.32	156.02	5.63	56.48	53.18	12.96	48.16	6.37	74.56
295-6	160.88	152.23	5.38	56.61	53.42	13.22	47.12	6.84	72.64
295-7	166.67	157.35	5.59	56.46	53.19	12.92	48.32	6.37	74.48
295-8	162.86	153.68	5.64	56.54	53.28	13.08	47.68	6.55	73.76
295-9	159.31	150.50	5.53	56.67	53.61	13.34	46.64	7.21	71.12
295-10	161.66	152.72	5.53	56.57	53.39	13.14	47.44	6.78	72.88
average	**163.07**	**154.07**	**5.52**	**56.49**	**53.35**	**12.99**	**48.05**	**6.71**	**73.17**
confidence limit	**1.71**	**1.61**	**0.07**	**0.11**	**0.09**	**0.21**	**0.85**	**0.18**	**0.71**
SD	**2.39**	**2.25**	**0.10**	**0.15**	**0.12**	**0.30**	**1.19**	**0.25**	**1.00**
CV %	**1.47**	**1.46**	**1.82**	**0.26**	**0.23**	**2.30**	**2.48**	**3.68**	**1.36**

JRC code	Force C1 cN	Force C2 cN	SD 1 %	La mm	Lb mm	Imm. rec. 1 %	Imm. PD 1 %	Rec. 1 %	PD 1 %
296-1	126.98	116.16	8.52	55.01	53.20	67.60	9.12	75.20	6.20
296-2	134.71	123.10	8.62	55.23	53.23	63.04	9.96	70.80	7.30
296-3	133.97	122.38	8.65	52.95	52.33	74.00	10.04	76.96	5.76
296-4	120.91	110.39	8.70	54.56	52.84	63.52	10.24	76.96	5.76
296-5	137.83	127.33	7.62	55.47	53.21	57.92	9.12	75.92	6.02
296-6	125.43	113.98	9.13	55.63	52.92	56.40	10.24	74.80	6.30
296-7	135.33	123.11	9.03	54.62	52.78	60.88	8.30	77.28	5.68
296-8	130.20	118.44	9.03	54.56	52.65	60.96	9.48	79.52	5.12
296-9	141.06	129.85	7.95	55.49	52.93	54.80	9.78	76.08	5.98
296-10	144.46	132.80	8.07	54.80	52.65	62.40	10.42	80.24	4.94
average	**133.09**	**121.75**	**8.53**	**54.83**	**52.87**	**62.15**	**9.67**	**76.38**	**5.91**
confidence limit	**5.20**	**5.08**	**0.36**	**0.55**	**0.21**	**4.00**	**0.47**	**1.87**	**0.47**
SD	**7.28**	**7.11**	**0.50**	**0.77**	**0.29**	**5.59**	**0.66**	**2.62**	**0.65**
CV %	**0.05**	**0.06**	**0.06**	**1.41**	**0.55**	**0.09**	**0.07**	**0.03**	**0.11**

JRC code	Force C5 cN	Force C6 cN	SD 2 %	Le mm	Lf mm	Imm. PD 3 %	Imm. rec 3 %	PD 3 %	Rec. 3 %
296-1	113.63	106.64	6.15	55.53	53.20	106.40	55.76	6.40	74.40
296-2	120.20	112.73	6.21	53.79	53.23	106.46	69.68	6.46	74.16
296-3	118.28	110.88	6.26	52.76	52.33	104.66	77.92	4.66	81.36
296-4	117.49	110.08	6.31	55.62	53.84	107.68	55.04	7.68	69.28
296-5	125.55	117.65	6.29	55.93	53.41	106.82	52.56	6.82	72.72
296-6	122.13	114.84	5.97	55.26	53.28	106.56	57.92	6.56	73.76
296-7	121.59	113.26	6.85	56.17	53.95	107.90	50.64	7.90	68.40
296-8	115.65	109.58	5.25	56.20	53.99	107.98	50.40	7.98	68.08
296-9	118.65	111.89	5.70	55.75	53.74	107.48	54.00	7.48	70.08
296-10	121.65	112.56	7.47	55.26	53.54	107.08	57.92	7.08	71.68
average	**119.48**	**112.01**	**6.25**	**55.23**	**53.45**	**106.90**	**58.18**	**6.90**	**72.39**
confidence limit	**2.48**	**2.16**	**0.43**	**0.79**	**0.35**	**0.70**	**8.85**	**0.70**	**2.82**
SD	**3.47**	**3.02**	**0.60**	**1.11**	**0.49**	**0.98**	**6.33**	**0.98**	**3.94**
CV %	**0.03**	**0.03**	**0.10**	**2.00**	**0.92**	**0.01**	**0.15**	**0.14**	**0.05**

JRC code	Force C1 cN	Force C2 cN	SD 1 %	La mm	Lb mm	Imm. rec. 1 %	Imm. PD 1 %	Rec. 1 %	PD 1 %
297-1	878.82	768.30	12.58	52.80	53.01	77.60	5.60	75.92	6.32
297-2	1139.12	1024.28	10.08	53.87	53.67	69.04	7.74	70.64	6.97
297-3	1338.23	1216.11	9.13	54.03	52.50	67.76	8.06	80.00	5.01
297-4	816.06	709.17	13.10	53.40	53.76	72.80	6.80	69.92	7.51
297-5	1109.32	990.93	10.67	50.23	52.99	98.16	0.46	76.08	5.98
297-6	511.43	444.03	13.18	52.76	53.55	77.92	5.52	71.60	7.11
297-7	884.49	768.59	13.10	51.00	52.09	92.00	2.00	83.28	4.18
297-8	633.34	541.99	14.42	54.23	53.83	66.16	8.46	69.36	7.66
297-9	766.85	657.46	14.27	55.76	53.73	53.92	11.52	70.16	7.46
297-10	654.17	557.24	14.82	54.51	54.09	63.92	9.02	67.28	8.17
average	**873.18**	**767.81**	**12.54**	**53.26**	**53.32**	**73.93**	**6.52**	**73.42**	**6.64**
confidence limit	**183.75**	**173.97**	**1.39**	**1.18**	**0.46**	**9.44**	**2.36**	**3.71**	**0.90**
SD	**256.87**	**243.20**	**1.94**	**1.65**	**0.65**	**13.20**	**3.30**	**5.18**	**1.26**
CV %	**29.42**	**31.67**	**15.49**	**3.10**	**1.22**	**17.85**	**50.61**	**7.06**	**19.05**

JRC code	Force C5 cN	Force C6 cN	SD 2 %	Le mm	Lf mm	Imm. PD 3 %	Imm. rec 3 %	PD 3 %	Rec. 3 %
297-1	801.98	731.35	8.81	55.56	53.65	11.12	55.52	8.31	70.80
297-2	1068.89	988.64	7.51	55.14	53.85	10.28	58.88	9.34	69.20
297-3	1254.47	1170.67	6.68	54.01	53.40	8.02	67.92	6.81	72.80
297-4	734.02	669.98	8.72	54.61	54.85	9.22	63.12	9.70	61.20
297-5	1029.01	950.21	7.66	53.67	53.94	7.34	70.64	7.89	68.48
297-6	441.87	407.10	7.87	51.49	55.00	2.98	88.08	10.00	60.00
297-7	798.91	727.44	8.95	52.20	53.33	4.40	82.40	6.67	73.36
297-8	529.31	481.98	8.94	55.47	54.93	10.94	56.24	9.86	60.56
297-9	676.47	611.96	9.54	56.68	54.73	13.36	46.56	9.46	62.16
297-10	561.29	509.32	9.26	55.07	55.16	10.14	59.44	10.32	58.72
average	**789.62**	**724.87**	**8.39**	**54.39**	**54.28**	**8.78**	**64.88**	**8.84**	**65.73**
confidence limit	**185.98**	**175.44**	**0.65**	**1.14**	**0.51**	**2.27**	**9.09**	**0.95**	**4.10**
SD	**259.99**	**245.25**	**0.91**	**1.59**	**0.72**	**3.18**	**12.71**	**1.33**	**5.73**
CV %	**32.93**	**33.83**	**10.86**	**2.92**	**1.32**	**36.19**	**19.59**	**15.07**	**8.72**

JRC code	Force C1 cN	Force C2 cN	SD 1 %	La mm	Lb mm	Imm. rec. 1 %	Imm. PD 1 %	Rec. 1 %	PD 1 %
300-1	76.54	70.93	7.32	54.05	53.10	67.60	9.12	75.20	6.20
300-2	84.56	77.87	7.91	54.62	53.65	63.04	9.96	70.80	7.30
300-3	83.87	77.15	8.01	53.25	52.88	74.00	10.04	76.96	5.76
300-4	70.65	65.16	7.77	54.56	52.88	63.52	10.24	76.96	5.76
300-5	87.59	82.10	6.26	55.26	53.01	57.92	9.12	75.92	6.02
300-6	75.18	68.74	8.56	55.45	53.15	56.40	10.24	74.80	6.30
300-7	85.08	76.84	9.68	54.89	52.84	60.88	8.30	77.28	5.68
300-8	79.95	72.51	9.30	54.88	52.56	60.96	9.48	79.52	5.12
300-9	90.81	84.54	6.90	55.65	52.99	54.80	9.78	76.08	5.98
300-10	94.21	88.63	5.92	54.70	52.47	62.40	10.42	80.24	4.94
average	**82.84**	**76.45**	**7.76**	**54.73**	**52.95**	**62.15**	**9.67**	**76.38**	**5.91**
confidence limit	**5.22**	**5.23**	**0.87**	**0.50**	**0.23**	**4.00**	**0.47**	**1.87**	**0.47**
SD	**7.30**	**7.31**	**1.22**	**0.70**	**0.33**	**5.59**	**0.66**	**2.62**	**0.65**
CV %	**0.09**	**0.10**	**0.16**	**1.28**	**0.62**	**0.09**	**0.07**	**0.03**	**0.11**

JRC code	Force C5 cN	Force C6 cN	SD 2 %	Le mm	Lf mm	Imm. PD 3 %	Imm. rec 3 %	PD 3 %	Rec. 3 %
300-1	63.43	58.45	7.85	54.76	53.28	6.30	61.92	6.56	73.76
300-2	70.00	64.54	7.80	55.13	53.50	6.92	58.96	7.00	72.00
300-3	68.08	62.69	7.92	53.76	53.26	7.08	69.92	6.52	73.92
300-4	67.29	61.89	8.03	55.07	53.56	6.48	59.44	7.12	71.52
300-5	75.35	69.46	7.82	55.77	53.41	6.74	53.84	6.82	72.72
300-6	72.56	65.84	9.26	55.96	53.84	5.84	52.32	7.68	69.28
300-7	70.99	65.98	7.06	55.40	54.01	5.76	56.80	8.02	67.92
300-8	64.58	61.58	4.65	55.39	53.74	5.98	56.88	7.48	70.08
300-9	68.45	63.70	6.94	56.16	53.83	5.74	50.72	7.66	69.36
300-10	71.45	64.37	9.91	55.21	53.85	5.56	58.32	7.70	69.20
average	**69.22**	**63.85**	**7.72**	**55.26**	**53.63**	**6.24**	**57.91**	**7.26**	**70.98**
confidence limit	**2.59**	**2.14**	**1.01**	**0.49**	**0.19**	**0.39**	**3.89**	**0.38**	**1.50**
SD	**3.62**	**2.99**	**1.41**	**0.68**	**0.26**	**0.54**	**5.43**	**0.52**	**2.10**
CV %	**0.05**	**0.05**	**0.18**	**1.23**	**0.49**	**0.09**	**0.09**	**0.07**	**0.03**

JRC code	Force C1 cN	Force C2 cN	SD 1 %	La mm	Lb mm	Imm. rec. 1 %	Imm. PD 1 %	Rec. 1 %	PD 1 %
317-1	145.26	134.48	7.42	54.56	52.87	63.52	9.12	77.04	5.74
317-2	158.74	143.69	9.48	54.98	53.05	60.16	9.96	75.60	6.10
317-3	156.40	144.87	7.37	55.02	52.98	59.84	10.04	76.16	5.96
317-4	166.04	155.78	6.18	55.12	53.25	59.04	10.24	74.00	6.50
317-5	160.54	147.81	7.93	54.56	52.67	63.52	9.12	78.64	5.34
317-6	159.98	146.55	8.39	55.12	52.54	59.04	10.24	79.68	5.08
317-7	143.81	133.11	7.44	54.15	53.01	66.80	8.30	75.92	6.02
317-8	145.88	135.04	7.43	54.74	52.68	62.08	9.48	78.56	5.36
317-9	159.20	148.67	6.61	54.89	53.07	60.88	9.78	75.44	6.14
317-10	160.01	148.54	7.17	55.21	52.98	58.32	10.42	76.16	5.96
average	**155.59**	**143.85**	**7.54**	**54.84**	**52.91**	**61.32**	**9.67**	**76.72**	**5.82**
confidence limit	**5.52**	**5.29**	**0.66**	**0.24**	**0.16**	**1.90**	**0.47**	**1.25**	**0.31**
SD	**7.72**	**7.40**	**0.92**	**0.33**	**0.22**	**2.65**	**0.66**	**1.75**	**0.44**
CV %	**0.05**	**0.05**	**0.12**	**0.61**	**0.41**	**0.04**	**0.07**	**0.02**	**0.08**

JRC code	Force C5 cN	Force C6 cN	SD 2 %	Le mm	Lf mm	Imm. PD 3 %	Imm. rec 3 %	PD 3 %	Rec. 3 %
317-1	135.25	124.58	7.89	54.03	53.15	6.30	67.76	6.30	74.80
317-2	137.89	124.48	9.73	54.33	53.46	6.92	65.36	6.92	72.32
317-3	140.69	131.56	6.49	53.53	53.54	7.08	71.76	7.08	71.68
317-4	145.75	133.48	8.42	54.07	53.24	6.48	67.44	6.48	74.08
317-5	151.98	139.75	8.05	53.66	53.37	6.74	70.72	6.74	73.04
317-6	142.54	130.88	8.18	53.35	52.92	5.84	73.20	5.84	76.64
317-7	129.54	118.66	8.40	53.63	52.88	5.76	70.96	5.76	76.96
317-8	129.77	121.65	6.26	53.47	52.99	5.98	72.24	5.98	76.08
317-9	140.55	126.98	9.65	53.32	52.87	5.74	73.44	5.74	77.04
317-10	141.74	129.02	8.97	53.17	52.78	5.56	74.64	5.56	77.76
average	**139.57**	**128.10**	**8.20**	**53.66**	**53.12**	**6.24**	**70.75**	**6.24**	**75.04**
confidence limit	**4.92**	**4.41**	**0.82**	**0.27**	**0.19**	**0.39**	**2.14**	**0.39**	**1.55**
SD	**6.88**	**6.17**	**1.15**	**0.37**	**0.27**	**0.54**	**2.99**	**0.54**	**2.17**
CV %	**0.05**	**0.05**	**0.14**	**0.70**	**0.51**	**0.09**	**0.04**	**0.09**	**0.03**

Elastic recovery of pure PTT samples - Favimat dynamometer

JRC code	Force C1 cN	Force C2 cN	SD1 %	Imm. rec. 1 %	Imm. PD 1 %	Rec. 1 %	PD 1 %	Force C3 cN	Force C4 cN	SD2 %	Imm. rec. 2 %
293-1	146.31	120.68	17.52	62.72	9.18	78.59	5.27	135.23	118.24	12.57	59.83
293-2	130.24	106.14	18.50	61.98	9.37	79.12	5.14	117.60	102.71	12.66	59.02
293-3	145.12	119.66	17.54	62.90	9.14	79.12	5.14	134.35	117.32	12.68	60.13
293-4	145.79	120.37	17.44	63.16	9.08	80.19	4.88	134.41	117.94	12.26	60.33
293-5	147.33	121.66	17.43	63.30	9.04	79.14	5.14	136.27	119.24	12.50	60.38
293-6	146.38	121.11	17.26	63.50	8.99	79.05	5.16	134.85	118.70	11.97	60.76
293-7	143.97	119.10	17.27	64.30	8.80	79.67	5.01	132.77	116.72	12.08	61.56
293-8	141.66	117.18	17.28	64.56	8.73	79.86	4.96	130.68	114.82	12.13	61.85
293-9	143.35	118.41	17.40	65.13	8.59	81.38	4.59	132.13	116.03	12.18	62.34
293-10	141.15	116.46	17.49	63.80	8.92	79.72	5.00	130.19	114.15	12.32	61.04
average	**142.78**	**117.79**	**17.51**	**63.63**	**8.96**	**79.69**	**5.00**	**131.47**	**115.29**	**12.31**	**60.82**
confidence limit	**3.67**	**3.36**	**0.27**	**0.68**	**0.17**	**0.54**	**0.13**	**3.98**	**3.58**	**0.18**	**0.72**
SD	**5.14**	**4.69**	**0.38**	**0.95**	**0.23**	**0.75**	**0.18**	**5.57**	**5.01**	**0.25**	**1.01**
CV %	**3.60**	**3.98**	**2.19**	**1.49**	**2.61**	**0.94**	**3.66**	**4.24**	**4.34**	**2.06**	**1.66**

JRC code	Imm. PD 2 %	Rec. 2 %	PD 2 %	Force C5 cN	Force C6 cN	SD3 %	Imm. PD 3 %	Imm. Rec. 3 %	PD 3 %	Rec. 3 %	Force C7 cN
293-1	9.90	75.36	6.07	130.75	116.29	11.06	10.23	58.48	6.51	73.56	128.84
293-2	10.09	75.46	6.05	113.04	100.29	11.28	10.45	57.58	6.44	73.87	110.23
293-3	9.82	75.87	5.94	129.83	115.38	11.13	10.16	58.76	6.35	74.20	127.55
293-4	9.78	76.57	5.77	131.12	116.06	11.48	10.12	58.93	6.16	75.01	128.21
293-5	9.76	75.93	5.93	131.87	117.29	11.06	10.10	58.99	6.35	74.24	129.35
293-6	9.66	75.91	5.93	131.13	116.82	10.91	9.98	59.47	6.33	74.30	129.55
293-7	9.47	76.55	5.78	128.95	114.86	10.93	9.75	60.42	6.15	75.04	127.56
293-8	9.40	76.93	5.68	127.54	113.03	11.38	9.72	60.54	6.10	75.25	124.77
293-9	9.28	78.14	5.38	129.12	114.19	11.56	9.61	60.97	5.81	76.41	126.17
293-10	9.60	76.56	5.77	126.53	112.32	11.23	9.91	59.78	6.18	74.91	124.34
average	**9.65**	**76.44**	**5.80**	**127.68**	**113.36**	**11.22**	**9.98**	**59.49**	**6.21**	**74.80**	**125.30**
confidence limit	**0.18**	**0.57**	**0.14**	**4.12**	**3.70**	**0.17**	**0.19**	**0.76**	**0.14**	**0.55**	**4.25**
SD	**0.25**	**0.79**	**0.20**	**5.76**	**5.17**	**0.23**	**0.26**	**1.06**	**0.19**	**0.77**	**5.94**
CV %	**2.55**	**1.03**	**3.39**	**4.51**	**4.56**	**2.06**	**2.63**	**1.78**	**3.04**	**1.02**	**4.74**

JRC code	Force C1 cN	Force C2 cN	SD1 %	Imm. rec. 1 %	Imm. PD 1 %	Rec. 1 %	PD 1 %	Force C3 cN	Force C4 cN	SD2 %	Imm. rec. 2 %
294-1	138.21	114.77	16.97	68.53	7.75	82.80	4.24	124.56	111.73	10.30	65.89
294-2	133.21	110.84	16.79	67.09	8.11	81.30	4.61	120.55	108.18	10.26	64.43
294-3	135.52	112.51	16.98	67.27	8.07	82.35	4.35	122.87	109.73	10.69	64.66
294-4	135.73	112.83	16.87	67.62	7.98	81.95	4.45	122.55	109.96	10.27	65.17
294-5	136.23	113.12	16.96	68.23	7.82	82.68	4.27	123.25	110.19	10.60	65.52
294-6	136.73	113.54	16.96	68.95	7.65	83.13	4.16	123.48	110.61	10.42	66.20
294-7	134.60	111.71	17.01	68.13	7.85	82.60	4.29	121.59	108.88	10.45	65.44
294-8	133.53	110.95	16.91	67.59	7.98	81.78	4.49	121.04	107.91	10.85	65.04
294-9	134.27	111.42	17.02	67.70	7.96	81.67	4.52	121.75	108.69	10.72	64.93
294-10	134.53	111.68	16.98	68.10	7.86	83.09	4.17	121.63	108.91	10.46	65.34
average	**135.26**	**112.34**	**16.95**	**67.92**	**7.90**	**82.34**	**4.36**	**122.33**	**109.48**	**10.50**	**65.26**
confidence limit	**1.10**	**0.89**	**0.05**	**0.41**	**0.10**	**0.45**	**0.11**	**0.88**	**0.85**	**0.15**	**0.38**
SD	**1.54**	**1.25**	**0.07**	**0.57**	**0.14**	**0.63**	**0.16**	**1.23**	**1.18**	**0.20**	**0.54**
CV %	**1.14**	**1.11**	**0.41**	**0.84**	**1.81**	**0.77**	**3.56**	**1.01**	**1.08**	**1.95**	**0.82**

JRC code	Imm. PD 2 %	Rec. 2 %	PD 2 %	Force C5 cN	Force C6 cN	SD3 %	Imm. PD 3 %	Imm. Rec. 3 %	PD 3 %	Rec. 3 %	Force C7 cN
294-1	8.40	79.59	5.03	120.44	109.45	9.12	8.74	64.51	5.45	77.89	118.78
294-2	8.76	78.21	5.37	117.62	106.19	9.72	9.10	63.06	5.78	76.54	114.70
294-3	8.71	78.97	5.18	118.66	107.60	9.32	9.01	63.42	5.58	77.35	116.36
294-4	8.58	78.89	5.20	118.72	107.82	9.18	8.92	63.80	5.57	77.37	116.31
294-5	8.49	79.48	5.05	118.93	107.96	9.23	8.83	64.17	5.45	77.87	116.52
294-6	8.32	79.99	4.93	119.99	108.38	9.68	8.66	64.83	5.35	78.29	117.11
294-7	8.52	79.36	5.08	117.59	106.72	9.24	8.86	64.05	5.51	77.63	115.40
294-8	8.61	78.58	5.28	116.42	105.67	9.24	8.92	63.79	5.68	76.93	114.63
294-9	8.64	78.33	5.34	117.70	106.59	9.44	8.98	63.53	5.74	76.69	115.11
294-10	8.54	79.78	4.98	117.72	106.81	9.27	8.90	63.87	5.39	78.11	115.38
average	**8.56**	**79.12**	**5.14**	**118.38**	**107.32**	**9.34**	**8.89**	**63.90**	**5.55**	**77.47**	**116.03**
confidence limit	**0.10**	**0.44**	**0.11**	**0.87**	**0.81**	**0.15**	**0.09**	**0.37**	**0.11**	**0.43**	**0.91**
SD	**0.13**	**0.62**	**0.15**	**1.21**	**1.13**	**0.21**	**0.13**	**0.52**	**0.15**	**0.60**	**1.27**
CV %	**1.57**	**0.78**	**2.98**	**1.03**	**1.06**	**2.20**	**1.44**	**0.81**	**2.65**	**0.77**	**1.10**

JRC code	Force C1 cN	Force C2 cN	SD1 %	Imm. rec. 1 %	Imm. PD 1 %	Rec. 1 %	PD 1 %	Force C3 cN	Force C4 cN	SD2 %	Imm. rec. 2 %
295-1	202.66	171.33	15.46	69.03	7.63	82.46	4.32	183.28	167.37	8.68	66.63
295-2	196.80	166.44	15.43	68.33	7.80	82.45	4.32	178.47	162.67	8.85	66.03
295-3	198.77	167.77	15.60	68.53	7.76	82.35	4.35	180.17	163.91	9.03	66.29
295-4	204.55	172.87	15.49	69.07	7.62	82.30	4.36	185.81	169.01	9.04	66.76
295-5	202.39	170.97	15.52	68.66	7.72	82.18	4.39	182.85	166.98	8.68	66.37
295-6	200.54	169.49	15.48	68.35	7.80	82.34	4.35	182.53	165.58	9.28	65.92
295-7	196.52	165.66	15.70	68.63	7.73	82.27	4.37	177.54	161.78	8.88	66.28
295-8	196.88	167.14	15.11	68.17	7.84	81.77	4.49	179.34	163.54	8.81	65.74
295-9	196.76	166.30	15.48	67.32	8.06	80.88	4.71	178.88	162.58	9.11	64.88
295-10	200.06	168.49	15.78	68.17	7.84	82.12	4.41	180.63	164.47	8.95	65.78
average	**199.59**	**168.65**	**15.51**	**68.43**	**7.78**	**82.11**	**4.41**	**180.95**	**164.79**	**8.93**	**66.07**
confidence limit	**2.08**	**1.74**	**0.13**	**0.36**	**0.09**	**0.34**	**0.08**	**1.86**	**1.70**	**0.14**	**0.38**
SD	**2.91**	**2.44**	**0.18**	**0.50**	**0.13**	**0.48**	**0.12**	**2.59**	**2.37**	**0.19**	**0.54**
CV %	**1.46**	**1.45**	**1.15**	**0.73**	**1.61**	**0.58**	**2.67**	**1.43**	**1.44**	**2.13**	**0.81**

JRC code	Imm. PD 2 %	Rec. 2 %	PD 2 %	Force C5 cN	Force C6 cN	SD3 %	Imm. PD 3 %	Imm. Rec. 3 %	PD 3 %	Rec. 3 %	Force C7 cN
295-1	8.22	79.93	4.94	179.19	164.44	8.23	8.55	65.28	5.31	78.44	174.88
295-2	8.37	79.89	4.96	173.69	159.85	7.97	8.65	64.89	5.33	78.37	170.56
295-3	8.31	79.74	5.00	175.16	160.99	8.09	8.58	65.21	5.34	78.35	172.84
295-4	8.19	79.66	5.01	179.80	165.95	7.70	8.49	65.54	5.34	78.33	176.45
295-5	8.28	79.53	5.04	178.75	164.08	8.21	8.61	65.05	5.37	78.19	174.68
295-6	8.39	79.66	5.01	176.06	162.54	7.68	8.67	64.79	5.35	78.27	173.71
295-7	8.31	79.53	5.04	173.01	158.92	8.14	8.60	65.08	5.37	78.22	169.55
295-8	8.44	79.12	5.14	174.10	160.75	7.67	8.74	64.50	5.46	77.83	171.12
295-9	8.66	78.21	5.37	173.56	159.73	7.97	8.94	63.71	5.74	76.71	170.35
295-10	8.43	79.41	5.07	175.84	161.48	8.17	8.70	64.69	5.39	78.11	171.95
average	**8.36**	**79.47**	**5.06**	**175.92**	**161.87**	**7.98**	**8.65**	**64.87**	**5.40**	**78.08**	**172.61**
confidence limit	**0.10**	**0.36**	**0.09**	**1.80**	**1.65**	**0.16**	**0.09**	**0.36**	**0.09**	**0.37**	**1.63**
SD	**0.13**	**0.50**	**0.12**	**2.51**	**2.31**	**0.22**	**0.12**	**0.51**	**0.13**	**0.51**	**2.28**
CV %	**1.61**	**0.63**	**2.43**	**1.43**	**1.43**	**2.81**	**1.44**	**0.78**	**2.34**	**0.66**	**1.32**

JRC code	Force C1 cN	Force C2 cN	SD1 %	Imm. rec. 1 %	Imm. PD 1 %	Rec. 1 %	PD 1 %	Force C3 cN	Force C4 cN	SD2 %	Imm. rec. 2 %
296-1	145.39	119.77	17.62	62.09	9.34	79.11	5.15	133.82	117.37	12.29	59.21
296-2	148.31	122.64	17.31	63.45	9.00	79.07	5.16	135.86	119.90	11.75	60.95
296-3	149.67	123.26	17.64	63.43	9.01	79.61	5.02	137.33	120.61	12.18	60.60
296-4	145.81	120.17	17.59	62.07	9.35	78.51	5.30	133.92	117.66	12.14	59.21
296-5	145.98	120.44	16.85	61.79	9.91	78.39	5.14	132.12	118.48	12.51	58.83
296-6	145.20	119.87	17.45	62.30	9.29	77.96	5.43	133.55	117.40	12.09	59.27
296-7	148.96	123.10	17.36	63.54	8.98	78.79	5.22	137.14	120.55	12.10	60.77
296-8	151.39	125.06	17.39	63.29	9.04	79.07	5.16	139.76	122.46	12.37	60.37
296-9	145.07	120.55	16.90	64.23	8.82	79.59	5.03	134.27	118.13	12.02	61.39
296-10	144.73	119.64	17.34	62.96	9.13	79.94	4.94	133.13	117.18	11.98	60.21
average	**147.05**	**121.45**	**17.35**	**62.92**	**9.19**	**79.00**	**5.16**	**135.09**	**118.97**	**12.14**	**60.08**
confidence limit	**1.67**	**1.36**	**0.20**	**0.58**	**0.22**	**0.44**	**0.10**	**1.69**	**1.29**	**0.15**	**0.63**
SD	**2.34**	**1.90**	**0.27**	**0.81**	**0.31**	**0.61**	**0.14**	**2.36**	**1.80**	**0.21**	**0.89**
CV %	**1.59**	**1.56**	**1.59**	**1.28**	**3.35**	**0.77**	**2.75**	**1.75**	**1.51**	**1.76**	**1.47**

JRC code	Imm. PD 2 %	Rec. 2 %	PD 2 %	Force C5 cN	Force C6 cN	SD3 %	Imm. PD 3 %	Imm. Rec. 3 %	PD 3 %	Rec. 3 %	Force C7 cN
296-1	10.05	75.55	6.02	130.36	115.50	11.40	9.34	57.70	6.41	73.99	127.68
296-2	9.62	75.86	5.95	132.35	117.88	10.94	9.00	59.69	6.28	74.52	129.76
296-3	9.71	76.32	5.83	133.94	118.61	11.45	9.01	59.13	6.25	74.63	131.64
296-4	10.05	75.03	6.15	130.10	115.72	11.05	9.35	57.73	6.61	73.16	127.83
296-5	10.64	75.58	6.75	129.42	117.06	10.92	9.91	58.38	6.21	73.74	128.10
296-6	10.03	74.40	6.31	129.90	115.49	11.09	9.29	57.76	6.73	72.69	127.55
296-7	9.66	75.64	6.00	133.68	118.58	11.29	8.98	59.37	6.46	73.77	130.55
296-8	9.76	75.72	5.98	134.86	120.36	10.76	9.04	59.02	6.42	73.95	133.34
296-9	9.52	76.22	5.86	130.27	116.23	10.77	8.82	59.83	6.29	74.48	127.92
296-10	9.80	76.98	5.67	129.36	115.30	10.87	9.13	58.80	6.09	75.29	127.27
average	**9.88**	**75.73**	**6.05**	**131.42**	**117.07**	**11.05**	**9.19**	**58.74**	**6.38**	**74.02**	**129.16**
confidence limit	**0.23**	**0.51**	**0.22**	**1.49**	**1.23**	**0.18**	**0.22**	**0.58**	**0.14**	**0.54**	**1.48**
SD	**0.33**	**0.71**	**0.30**	**2.08**	**1.72**	**0.25**	**0.31**	**0.81**	**0.19**	**0.75**	**2.07**
CV %	**3.29**	**0.93**	**4.97**	**1.58**	**1.47**	**2.27**	**3.35**	**1.38**	**3.01**	**1.02**	**1.61**

JRC code	Force C1 cN	Force C2 cN	SD1 %	Imm. rec. 1 %	Imm. PD 1 %	Rec. 1 %	PD 1 %	Force C3 cN	Force C4 cN	SD2 %	Imm. rec. 2 %
300-1	72.02	60.34	16.22	57.49	10.47	75.35	6.07	70.76	60.91	13.92	55.41
300-2	95.32	77.89	18.28	61.52	9.48	75.97	5.92	88.22	76.46	13.34	58.37
300-3	95.76	78.31	18.22	61.55	9.47	75.97	5.92	88.10	76.83	12.79	58.46
300-4	96.36	79.01	18.01	62.09	9.34	76.39	5.81	88.86	77.57	12.71	59.03
300-5	94.01	76.59	18.53	61.02	9.60	75.66	5.99	86.54	75.16	13.15	57.85
300-6	94.54	77.26	18.28	61.31	9.53	75.74	5.98	87.16	75.82	13.01	58.22
300-7	94.02	76.77	18.35	60.94	9.62	75.39	6.06	86.62	75.34	13.01	57.82
300-8	93.53	76.31	18.41	60.99	9.62	75.63	6.01	86.21	74.92	13.10	57.78
300-9	96.22	78.43	18.49	62.11	9.33	77.03	5.66	88.42	76.94	12.99	59.02
300-10	87.46	71.38	18.39	61.40	9.51	76.31	5.84	79.55	69.40	12.76	58.38
average	**91.92**	**75.23**	**18.12**	**61.04**	**9.60**	**75.94**	**5.93**	**85.04**	**73.94**	**13.08**	**58.03**
confidence limit	**5.32**	**4.04**	**0.49**	**0.94**	**0.23**	**0.37**	**0.09**	**4.06**	**3.66**	**0.25**	**0.73**
SD	**7.44**	**5.65**	**0.68**	**1.31**	**0.32**	**0.52**	**0.13**	**5.68**	**5.11**	**0.35**	**1.02**
CV %	**8.10**	**7.51**	**3.77**	**2.15**	**3.37**	**0.68**	**2.14**	**6.68**	**6.91**	**2.70**	**1.76**

JRC code	Imm. PD 2 %	Rec. 2 %	PD 2 %	Force C5 cN	Force C6 cN	SD3 %	Imm. PD 3 %	Imm. Rec. 3 %	PD 3 %	Rec. 3 %	Force C7 cN
300-1	10.99	72.13	6.87	70.58	61.24	13.22	10.47	54.49	7.27	70.50	70.41
300-2	10.26	72.22	6.85	85.25	75.25	11.73	9.48	56.88	7.33	70.26	83.97
300-3	10.23	72.25	6.84	85.53	75.62	11.58	9.47	56.94	7.32	70.31	84.12
300-4	10.09	72.84	6.69	86.39	76.37	11.60	9.34	57.51	7.17	70.90	84.80
300-5	10.38	71.83	6.94	83.96	73.98	11.88	9.60	56.27	7.42	69.87	82.77
300-6	10.29	72.00	6.90	84.62	74.65	11.78	9.53	56.64	7.38	70.03	83.04
300-7	10.39	71.64	6.99	84.18	74.20	11.86	9.62	56.15	7.48	69.62	82.76
300-8	10.41	71.86	6.94	83.80	73.78	11.96	9.62	56.31	7.43	69.87	82.38
300-9	10.10	73.18	6.61	85.99	75.73	11.93	9.33	57.40	7.08	71.26	84.40
300-10	10.25	72.53	6.77	77.17	68.14	11.71	9.51	56.87	7.25	70.59	75.65
average	**10.34**	**72.25**	**6.84**	**82.75**	**72.90**	**11.93**	**9.60**	**56.55**	**7.31**	**70.32**	**81.43**
confidence limit	**0.18**	**0.34**	**0.09**	**3.58**	**3.36**	**0.34**	**0.23**	**0.61**	**0.09**	**0.36**	**3.34**
SD	**0.25**	**0.48**	**0.12**	**5.00**	**4.69**	**0.47**	**0.32**	**0.85**	**0.12**	**0.51**	**4.66**
CV %	**2.46**	**0.66**	**1.74**	**6.04**	**6.44**	**3.97**	**3.37**	**1.51**	**1.70**	**0.72**	**5.73**

Elastic recovery of PTT manually separated from fabrics-Favimat dynamometer

JRC code	Force C1 cN	Force C2 cN	SD1 %	Imm. rec. 1 %	Imm. PD 1 %	Rec. 1 %	PD 1 %	Force C3 cN	Force C4 cN	SD2 %	Imm. rec. 2 %
301-1	126.82	109.07	14.00	61.89	9.39	74.62	6.25	121.80	108.37	11.03	58.68
301-2	124.68	107.65	13.66	62.22	9.31	75.05	6.15	120.18	107.00	10.96	59.02
301-3	124.20	106.94	13.89	61.50	9.49	74.29	6.33	119.76	106.28	11.26	58.36
301-4	124.34	107.48	13.56	62.39	9.26	75.27	6.09	119.69	106.82	10.75	59.21
301-5	119.61	102.99	13.90	62.15	9.33	75.14	6.13	114.82	102.29	10.92	58.96
301-6	124.86	107.79	13.67	62.17	9.31	74.91	6.18	120.19	107.09	10.90	58.87
301-7	123.79	106.51	13.97	61.60	9.46	74.31	6.33	119.04	105.80	11.13	58.43
301-8	123.94	106.88	13.77	62.10	9.34	74.94	6.17	119.20	106.16	10.94	58.85
301-9	125.48	108.18	13.78	62.66	9.20	75.48	6.04	120.83	107.49	11.04	59.40
301-10	122.86	106.01	13.71	62.32	9.28	75.22	6.10	118.69	105.36	11.23	59.19
average	**124.06**	**106.95**	**13.79**	**62.10**	**9.34**	**74.92**	**6.18**	**119.42**	**106.27**	**11.02**	**58.90**
confidence limit	**1.35**	**1.17**	**0.10**	**0.25**	**0.06**	**0.29**	**0.07**	**1.33**	**1.18**	**0.11**	**0.24**
SD	**1.89**	**1.64**	**0.15**	**0.35**	**0.09**	**0.40**	**0.10**	**1.85**	**1.64**	**0.16**	**0.34**
CV %	**1.52**	**1.53**	**1.05**	**0.57**	**0.95**	**0.54**	**1.60**	**1.55**	**1.55**	**1.42**	**0.57**

JRC code	Imm. PD 2 %	Rec. 2 %	PD 2 %	Force C5 cN	Force C6 cN	SD3 %	Imm. PD 3 %	Imm. Rec. 3 %	PD 3 %	Rec. 3 %	Force C7 cN
301-1	10.18	70.76	7.21	120.24	107.63	10.48	10.58	57.08	7.71	68.71	118.42
301-2	10.09	71.25	7.08	118.11	106.27	10.03	10.50	57.39	7.60	69.13	117.06
301-3	10.26	70.33	7.31	117.35	105.54	10.07	10.67	56.70	7.80	68.33	116.77
301-4	10.05	71.35	7.06	117.85	106.12	9.95	10.45	57.59	7.57	69.26	116.51
301-5	10.11	71.13	7.11	113.41	101.61	10.41	10.51	57.33	7.60	69.15	112.39
301-6	10.13	71.03	7.13	118.13	106.33	9.99	10.55	57.15	7.64	68.96	117.00
301-7	10.24	70.48	7.27	116.88	105.06	10.11	10.64	56.81	7.77	68.45	115.70
301-8	10.14	71.05	7.13	117.58	105.45	10.32	10.53	57.27	7.63	69.02	115.88
301-9	10.00	71.63	6.99	118.94	106.76	10.23	10.39	57.81	7.50	69.56	117.33
301-10	10.05	71.43	7.04	116.54	104.63	10.22	10.45	57.55	7.54	69.40	115.06
average	**10.13**	**71.04**	**7.13**	**117.50**	**105.54**	**10.18**	**10.53**	**57.27**	**7.64**	**69.00**	**116.21**
confidence limit	**0.06**	**0.30**	**0.07**	**1.27**	**1.16**	**0.13**	**0.06**	**0.25**	**0.07**	**0.28**	**1.17**
SD	**0.08**	**0.41**	**0.10**	**1.78**	**1.63**	**0.18**	**0.09**	**0.35**	**0.10**	**0.40**	**1.64**
CV %	**0.83**	**0.58**	**1.43**	**1.52**	**1.54**	**1.78**	**0.83**	**0.60**	**1.27**	**0.58**	**1.41**

JRC code	Force C1 cN	Force C2 cN	SD1 %	Imm. rec. 1 %	Imm. PD 1 %	Rec. 1 %	PD 1 %	Force C3 cN	Force C4 cN	SD2 %	Imm. rec. 2 %
302-1	89.56	76.52	14.56	49.98	12.32	63.87	8.90	85.71	75.61	11.78	60.13
302-2	87.56	74.90	14.46	49.67	12.40	63.67	8.95	83.86	74.01	11.75	59.94
302-3	84.22	71.83	14.71	48.71	12.64	63.17	9.07	80.39	70.78	11.96	59.27
302-4	88.53	75.68	14.51	49.48	12.45	63.74	8.93	85.17	74.76	12.22	59.90
302-5	82.18	70.09	14.70	48.74	12.63	63.13	9.08	78.89	69.19	12.29	59.31
302-6	85.92	73.28	14.71	48.37	12.72	63.03	9.11	82.50	72.33	12.32	59.11
302-7	87.98	74.87	14.90	59.86	9.89	75.75	5.98	83.87	73.90	11.88	72.26
302-8	88.11	75.30	14.55	49.54	12.43	63.76	8.93	84.44	74.40	11.89	59.89
302-9	86.43	74.76	14.61	49.01	12.55	63.54	9.20	82.12	72.48	12.45	59.31
302-10	87.02	74.61	14.31	49.29	12.04	63.12	8.71	82.71	73.69	11.61	59.48
average	**86.75**	**74.18**	**14.60**	**50.27**	**12.21**	**64.68**	**8.69**	**82.97**	**73.12**	**12.02**	**60.86**
confidence limit	**1.57**	**1.38**	**0.12**	**2.44**	**0.60**	**2.79**	**0.69**	**1.52**	**1.39**	**0.20**	**2.88**
SD	**2.19**	**1.93**	**0.16**	**3.41**	**0.84**	**3.90**	**0.96**	**2.12**	**1.95**	**0.28**	**4.02**
CV %	**2.53**	**2.60**	**1.12**	**6.78**	**6.86**	**6.03**	**11.06**	**2.55**	**2.67**	**2.36**	**6.61**

JRC code	Imm. PD 2 %	Rec. 2 %	PD 2 %	Force C5 cN	Force C6 cN	SD3 %	Imm. PD 3 %	Imm. Rec. 3 %	PD 3 %	Rec. 3 %	Force C7 cN
302-1	13.00	60.13	9.82	84.30	75.02	11.00	13.33	58.09	10.32	58.09	83.51
302-2	13.06	59.94	9.87	82.64	73.45	11.12	13.41	57.97	10.36	57.97	81.78
302-3	13.34	59.27	10.03	79.09	70.00	11.50	13.68	57.24	10.53	57.24	77.96
302-4	13.16	59.90	9.88	83.47	74.16	11.15	13.47	57.88	10.38	57.88	82.61
302-5	13.33	59.31	10.02	77.28	68.62	11.21	13.65	57.31	10.52	57.31	76.46
302-6	13.37	59.11	10.07	81.18	71.75	11.61	13.75	57.13	10.56	57.13	80.15
302-7	10.61	72.26	6.84	82.48	73.31	11.12	10.98	70.44	7.28	70.44	81.64
302-8	13.13	59.89	9.88	83.17	73.84	11.22	13.48	57.93	10.36	57.93	82.37
302-9	13.56	59.31	10.14	80.86	71.83	11.51	13.76	57.22	10.63	57.22	80.41
302-10	13.02	59.48	9.77	81.73	72.45	11.03	13.09	57.04	10.21	57.04	80.89
average	**12.96**	**60.86**	**9.63**	**81.62**	**72.44**	**11.25**	**13.26**	**58.83**	**10.12**	**58.83**	**80.78**
confidence limit	**0.60**	**2.88**	**0.71**	**1.52**	**1.41**	**0.15**	**0.59**	**2.93**	**0.72**	**2.93**	**1.55**
SD	**0.84**	**4.02**	**0.99**	**2.13**	**1.96**	**0.22**	**0.83**	**4.10**	**1.00**	**4.10**	**2.17**
CV %	**6.52**	**6.61**	**10.26**	**2.61**	**2.71**	**1.91**	**6.24**	**6.97**	**9.93**	**6.97**	**2.68**

JRC code	Force C1 cN	Force C2 cN	SD1 %	Imm. rec. 1 %	Imm. PD 1 %	Rec. 1 %	PD 1 %	Force C3 cN	Force C4 cN	SD2 %	Imm. rec. 2 %
309-1	80.24	65.50	18.38	41.77	14.34	61.30	9.53	74.91	63.97	14.60	36.70
309-2	78.42	63.85	18.59	42.56	14.15	62.65	9.20	72.82	62.27	14.48	37.39
309-3	79.73	65.04	18.42	41.95	14.30	61.50	9.49	74.38	63.53	14.58	37.03
309-4	81.00	66.02	18.49	42.46	14.17	62.48	9.24	75.37	64.44	14.51	37.32
309-5	81.06	66.11	18.45	41.89	14.32	61.53	9.48	75.56	64.56	14.56	36.95
309-6	78.31	63.73	18.61	41.78	14.34	61.54	9.47	72.86	62.18	14.65	36.58
309-7	76.86	62.55	18.63	41.41	14.43	61.51	9.48	71.61	61.02	14.79	36.32
309-8	79.29	64.58	18.56	41.79	14.34	61.46	9.49	73.74	62.98	14.59	36.67
309-9	80.34	65.46	18.53	42.00	14.29	61.83	9.41	74.87	63.91	14.63	37.16
309-10	79.00	64.50	18.35	41.77	14.34	61.69	9.44	73.68	62.92	14.61	36.91
average	**79.43**	**64.73**	**18.50**	**41.94**	**14.30**	**61.75**	**9.42**	**73.98**	**63.18**	**14.60**	**36.90**
confidence limit	**0.95**	**0.80**	**0.07**	**0.24**	**0.06**	**0.32**	**0.08**	**0.91**	**0.80**	**0.06**	**0.24**
SD	**1.32**	**1.13**	**0.10**	**0.34**	**0.08**	**0.45**	**0.11**	**1.27**	**1.12**	**0.08**	**0.34**
CV %	**1.67**	**1.74**	**0.53**	**0.81**	**0.59**	**0.73**	**1.19**	**1.72**	**1.78**	**0.58**	**0.92**

JRC code	Imm. PD 2 %	Rec. 2 %	PD 2 %	Force C5 cN	Force C6 cN	SD3 %	Imm. PD 3 %	Imm. Rec. 3 %	PD 3 %	Rec. 3 %	Force C7 cN
309-1	15.59	54.43	11.23	72.80	63.07	13.38	16.16	34.39	12.09	50.90	71.59
309-2	15.42	55.54	10.95	71.16	61.38	13.73	16.03	34.92	11.84	51.94	69.50
309-3	15.52	54.62	11.18	72.28	62.64	13.33	16.07	34.77	12.02	51.22	71.21
309-4	15.44	55.43	10.98	73.37	63.52	13.42	16.02	34.96	11.86	51.87	72.08
309-5	15.53	54.69	11.16	73.55	63.66	13.44	16.10	34.64	12.03	51.19	72.35
309-6	15.62	54.57	11.19	70.76	61.27	13.41	16.19	34.27	12.06	51.02	69.81
309-7	15.68	54.55	11.19	69.48	60.12	13.47	16.23	34.10	12.05	51.07	68.46
309-8	15.60	54.52	11.20	72.20	62.10	13.99	16.16	34.39	12.08	50.96	70.94
309-9	15.48	55.04	11.08	73.13	63.04	13.80	16.07	34.79	11.93	51.57	71.97
309-10	15.54	54.71	11.16	71.96	62.01	13.82	16.12	34.57	12.00	51.30	70.80
average	**15.54**	**54.81**	**11.13**	**72.07**	**62.28**	**13.58**	**16.12**	**34.58**	**12.00**	**51.30**	**70.87**
confidence limit	**0.06**	**0.28**	**0.07**	**0.92**	**0.80**	**0.17**	**0.05**	**0.21**	**0.06**	**0.26**	**0.90**
SD	**0.08**	**0.39**	**0.10**	**1.29**	**1.12**	**0.23**	**0.07**	**0.29**	**0.09**	**0.37**	**1.26**
CV %	**0.53**	**0.72**	**0.87**	**1.78**	**1.81**	**1.71**	**0.43**	**0.83**	**0.74**	**0.72**	**1.78**

JRC	Force C1	Force C2	SD1	Imm. rec. 1	Imm. PD 1	Rec. 1	PD 1	Force C3	Force C4	SD2	Imm. rec. 2
code	cN	cN	%	%	%	%	%	cN	cN	%	%
311-1	76.88	64.50	16.11	44.91	13.57	60.00	9.85	72.91	63.45	12.98	41.63
311-2	76.33	63.98	16.18	50.11	12.30	63.32	9.04	72.29	62.92	12.97	47.35
311-3	63.59	52.68	17.15	48.05	12.80	61.43	9.50	59.14	51.51	12.89	45.41
311-4	77.99	65.58	15.91	45.19	13.50	60.66	9.69	74.08	64.54	12.88	41.81
311-5	76.29	64.09	15.99	45.72	13.38	61.27	9.55	72.52	63.07	13.03	42.51
311-6	73.39	61.45	16.27	43.92	13.81	60.25	9.79	69.37	60.40	12.94	40.70
311-7	74.85	62.87	16.01	44.79	13.60	60.32	9.78	71.00	61.86	12.87	41.64
311-8	73.10	60.95	16.62	42.60	14.14	58.90	10.12	68.89	59.86	13.11	39.42
311-9	71.63	59.64	16.73	42.41	14.18	58.78	10.15	67.58	58.59	13.30	39.03
311-10	74.44	62.25	16.38	43.28	13.98	59.46	9.99	70.71	61.19	13.46	40.00
average	**73.85**	**61.80**	**16.34**	**45.10**	**13.53**	**60.44**	**9.75**	**69.85**	**60.74**	**13.04**	**41.95**
confidence limit	**2.93**	**2.63**	**0.28**	**1.73**	**0.42**	**0.96**	**0.24**	**3.05**	**2.66**	**0.14**	**1.88**
SD	**4.09**	**3.68**	**0.39**	**2.42**	**0.59**	**1.35**	**0.33**	**4.26**	**3.71**	**0.20**	**2.62**
CV %	**5.54**	**5.95**	**2.40**	**5.36**	**4.37**	**2.23**	**3.38**	**6.10**	**6.11**	**1.50**	**6.25**

JRC	Imm. PD 2	Rec. 2	PD 2	Force C5	Force C6	SD3	Imm. PD 3	Imm. Rec. 3	PD 3	Rec. 3	Force C7
code	%	%	%	cN	cN	%	%	%	%	%	cN
311-1	11.01	55.30	11.01	71.50	62.83	12.13	14.76	40.07	11.59	52.95	70.58
311-2	9.93	59.74	9.93	70.84	62.29	12.06	13.28	46.14	10.34	58.06	69.94
311-3	10.37	57.92	10.37	57.59	50.82	11.76	13.76	44.15	10.77	56.29	56.71
311-4	10.87	55.86	10.87	72.70	63.91	12.09	14.75	40.13	11.46	53.48	71.98
311-5	10.69	56.63	10.69	71.01	62.46	12.04	14.56	40.93	11.30	54.18	69.86
311-6	11.00	55.34	11.00	68.20	59.81	12.30	15.00	39.10	11.61	52.87	67.23
311-7	10.92	55.67	10.92	69.60	61.26	11.98	14.76	40.12	11.49	53.39	68.81
311-8	11.34	53.96	11.34	67.50	59.24	12.23	15.33	37.74	11.92	51.58	66.68
311-9	11.38	53.78	11.38	66.10	57.98	12.28	15.43	37.34	11.99	51.33	65.36
311-10	11.16	54.73	11.16	68.99	60.56	12.22	15.17	38.44	11.77	52.26	68.18
average	**10.87**	**55.89**	**10.87**	**68.40**	**60.12**	**12.11**	**14.68**	**40.42**	**11.42**	**53.64**	**67.53**
confidence limit	**0.32**	**1.30**	**0.32**	**3.07**	**2.67**	**0.12**	**0.49**	**1.99**	**0.37**	**1.50**	**3.06**
SD	**0.44**	**1.82**	**0.44**	**4.29**	**3.73**	**0.16**	**0.68**	**2.78**	**0.51**	**2.09**	**4.28**
CV %	**4.08**	**3.25**	**4.08**	**6.27**	**6.21**	**1.35**	**4.63**	**6.88**	**4.49**	**3.91**	**6.34**

JRC	Force C1	Force C2	SD1	Imm. rec. 1	Imm. PD 1	Rec. 1	PD 1	Force C3	Force C4	SD2	Imm. rec. 2
code	cN	cN	%	%	%	%	%	cN	cN	%	%
312-1	97.27	83.50	14.16	52.34	11.74	66.36	8.29	93.62	82.63	11.74	49.73
312-2	97.71	83.56	14.49	51.86	11.86	66.31	8.30	93.98	82.68	12.02	49.25
312-3	98.57	84.69	14.09	52.50	11.70	66.53	8.25	95.16	83.85	11.88	49.90
312-4	95.36	81.19	14.87	50.80	12.12	65.49	8.50	91.41	80.25	12.21	48.15
312-5	90.89	78.00	14.18	51.68	11.91	66.02	8.37	85.43	74.99	12.22	48.98
312-6	99.23	84.87	14.47	52.38	11.73	66.72	8.20	95.85	84.02	12.35	49.65
312-7	96.47	82.64	14.34	51.33	11.99	65.75	8.44	92.79	81.76	11.89	48.77
312-8	99.06	84.59	14.60	53.18	11.54	67.37	8.04	95.42	83.71	12.27	50.63
312-9	179.13	152.49	14.88	57.17	10.55	72.61	6.75	171.89	150.62	12.38	54.36
312-10	97.79	83.77	14.25	52.49	11.79	66.72	8.34	93.81	82.71	11.82	50.01
average	**105.15**	**89.93**	**14.43**	**52.57**	**11.69**	**66.99**	**8.15**	**100.94**	**88.72**	**12.08**	**49.94**
confidence limit	**18.68**	**15.79**	**0.20**	**1.25**	**0.31**	**1.46**	**0.36**	**17.96**	**15.67**	**0.17**	**1.22**
SD	**26.11**	**22.08**	**0.28**	**1.75**	**0.43**	**2.05**	**0.51**	**25.11**	**21.91**	**0.24**	**1.70**
RSD %	**24.83**	**24.55**	**1.96**	**3.33**	**3.70**	**3.05**	**6.23**	**24.88**	**24.70**	**1.95**	**3.41**

JRC	Imm. PD 2	Rec. 2	PD 2	Force C5	Force C6	SD3	Imm. PD 3	Imm. Rec. 3	PD 3	Rec. 3	Force C7
code	%	%	%	cN	cN	%	%	%	%	%	cN
312-1	12.38	62.80	9.17	92.25	82.05	11.05	12.70	48.45	9.60	61.04	91.47
312-2	12.51	62.71	9.19	92.74	82.12	11.45	12.84	47.89	9.64	60.90	91.96
312-3	12.34	63.08	9.10	93.58	83.26	11.02	12.65	48.66	9.54	61.29	93.02
312-4	12.77	61.94	9.38	90.11	79.67	11.58	13.09	46.88	9.81	60.20	89.43
312-5	12.57	62.44	9.25	81.97	72.34	11.76	12.88	47.72	9.72	60.56	79.52
312-6	12.40	63.17	9.07	94.24	83.41	11.49	12.72	48.35	9.51	61.38	93.34
312-7	12.62	62.19	9.32	91.43	81.18	11.21	12.96	47.41	9.75	60.41	90.63
312-8	12.16	64.26	8.81	93.79	83.10	11.40	12.48	49.35	9.22	62.57	93.33
312-9	11.24	69.37	7.55	169.10	149.48	11.60	11.57	53.04	7.97	67.63	167.50
312-10	12.45	62.89	9.21	92.81	82.25	11.12	12.76	49.06	9.69	61.52	91.61
average	**12.34**	**63.49**	**9.01**	**99.20**	**87.89**	**11.37**	**12.67**	**48.68**	**9.45**	**61.75**	**98.18**
confidence limit	**0.30**	**1.55**	**0.38**	**17.75**	**15.66**	**0.18**	**0.30**	**1.22**	**0.39**	**1.55**	**17.66**
SD	**0.42**	**2.16**	**0.53**	**24.82**	**21.89**	**0.25**	**0.42**	**1.71**	**0.54**	**2.17**	**24.69**
RSD %	**3.42**	**3.41**	**5.94**	**25.01**	**24.90**	**2.24**	**3.32**	**3.50**	**5.76**	**3.52**	**25.15**

Annex VII

Validation study

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Health and Consumer Protection (IHCP)
Chemical Assessment and Testing Unit (CAT)

Ispra, 15th July 2013

Subject: Invitation to join the method validation to quantify polytrimethylene terephthalate (PTT) in mixture with polyethylene terephthalate (PET)

Dear Expert,

As agreed during the 12th meeting of the European Network of National Experts on Textile Labelling (ENNETL) held in Ispra on 30th November 2012, the JRC is organising a collaborative trial to validate the method for the quantification of PTT in binary mixtures with PET, which is based on Differential Scanning Calorimetric (DSC) analysis.

Six blind fabric samples will be sent and each of them must be quantified in triplicate.
Independent calibration curves (one for each sample) must be prepared using 100% PTT and PET yarns, provided by the JRC after manual separation from the sample itself.
For each sample 14 DSC runs have to be performed:

3 analyses of the pure 100 % PTT yarn
3 analyses of the pure 100 % PET yarn
3 analyses of the fabric sample
5 calibration points

In total 84 DSC analyses have to be made.

Please be aware that all the analyses must be performed under repeatability conditions, which means by the same operator, under the same conditions and in a short time interval, in particular the set of 14 analyses to quantify each one of the blind samples must be carried out on the same day.

Each participant will receive specimens by carrier and will have to analyse them <u>at the latest by 31th August 2013</u>.

I would kindly ask you to fill in the following registration form in capital letters with all details and to <u>send back to me the signed copy, together with this letter</u>, by fax (+39-0332-785707) or e-mail (paola.piccinini@jrc.ec.europa.eu), so that we can post samples by this week.

Thank you very much for your precious collaboration.

Best regards,

Paola Piccinini

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Health and Consumer Protection (IHCP)
Chemical Assessment and Testing Unit (CAT)

COLLABORATIVE TRIAL'S REGISTRATION FORM

VALIDATION OF DSC METHOD TO QUANTIFY BINARY MIXTURES PTT/PET

Contact person: …………………………………………………

Institution: ……………………………………………………

Address: ………………………………………………………

Postal code: …………………………………………………...

City: …………………………………………………………..

State: ………………………………………………………….

Phone: …………………………………………………………

Fax: ……………………………………………………………

E-mail: ………………………………………………………...

Total price: 1800-3000€*

*to be confirmed according to the total number of laboratories taking part in the validation (max 20 laboratories)

- ○ I accept to take part in the validation of the DSC method to quantify PTT in mixture with PET and all the conditions described in the invitation letter

- ○ I do not accept to take part in the validation of the DSC method to quantify PTT in mixture with PET

Signature: ……………………………………………………

Date: …………………………………………………………

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Health and Consumer Protection (IHCP)
Chemical Assessment and Testing Unit (CAT)

Ispra, 24th July 2013

Dear Expert,

The samples for the validation of the DSC method to quantify textile binary mixtures made of PTT and PET have been sent by carrier in the last days.

Please notice that experimental results must be sent by email (paola.piccinini@jrc.ec.europa.eu) by the end of August 2013 at the latest.

As soon as you receive the samples please send us back by fax (+39-0332-785707) or e-mail the sample acknowledgement form.

The test method protocol and the provided instructions must be strictly followed. In principle, they contain all the information needed to perform the analyses. However, if you experience any problems or if you have any doubt please do not hesitate to contact us and we will do our best to help.

In attachment you will find:

- sample acknowledgement form;
- test method protocol;
- general requirements and instructions;
- Excel file to report results.

I would like to thank you very much for your collaboration.

I am looking forwards to receiving your results.

Best regards,

Paola Piccinini

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Health and Consumer Protection (IHCP)
Chemical Assessment and Testing Unit (CAT)

SAMPLES ACKNOWLEDGMENT FORM

Please fill in this form and send it back by fax (+39-0332-785707) or e-mail (paola.piccinini@jrc.ec.europa.eu), as soon as you receive the samples.

Contact person: …………………………………………………

Institution: ……………………………………………………

Address: ……………………………………………………..

City: ………………………………………………………...

State: ……………………………………………………..

Phone: ……………………………………………………

Fax: ………………………………………………………

E-mail: …………………………………………………...

I declare that I received the samples for the collaborative trial to validate a DSC based method for the quantification of PTT in mixtures with PET in date ____ /____ / 2013.

Signature: …………………………………………………..

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Health and Consumer Protection (IHCP)
Chemical Assessment and Testing Unit (CAT)

GENERAL REQUIREMENTS AND INSTRUCTIONS

Before starting the analysis, please carefully read the instructions, the provided test method and the Excel data reporting sheet in order to collect all required data.

Provided samples

The following samples have been shipped:

- Six fabric samples (A, B, C, D, E and F) made of binary mixture PTT – PET (about one gram)
- Six yarn samples (A, B, C, D, E and F) made of pure PTT manually separated from the correspondent fabric sample (about 150-200 mg)
- Six yarn samples (A, B, C, D, E and F) made of pure PET manually separated from the correspondent fabric sample (about 150-200 mg)

Specimens are marked with codes, e.g. LC17-Sample A-35 PET. Labels indicate the laboratory's code (LC17), the sample code (A), a random number (35), used just for JRC's preparation purposes and not relevant for the method validation, and the indication of the nature of the sample (either FABRIC, or PTT or PET).

The PTT and PET yarns marked with the code A (manually separated from the fabric sample A) shall be used to build up the calibration curve used to quantify sample A.

The provided sample quantity is largely enough to carry out all the determinations.

Requested number of analyses

In total 84 DSC analyses have to be run.

An independent calibration curve with 7 points has to be built up for each of the six blind samples to be quantified (in total six calibration curves). The calibration samples shall be prepared using the PTT and PET yarns which have been manually separated from the fabric sample to be quantified.

To quantify each fabric sample a set of 14 DSC runs have to be performed:

- 3 replicates of pure PTT, corresponding to the 100% PTT calibration point;
- 3 replicates of pure PET, corresponding to the 0% PTT calibration point;

- One replicate for each of the 5 calibration points, each one made of a mixture of PTT and PET with approximately the following PTT mass percentage, 20 %, 40 %, 55 %, 70 % and 85 % (e.g., 1 mg of PTT mixed with 4 mg of PET, corresponds to 20 % PTT and 80 % PET);
- 3 replicates of the fabric sample to be quantified.

Instructions

The protocol of the test method has to be strictly applied.

The analyses **must** be performed in the same laboratory by the same operator, using the same equipment and at in short time interval (each set of 14 DSC runs, needed to quantify one sample, shall be carried out within one day). In other words, test results have to be obtained in **repeatability conditions**.

In case of any problem during the analysis of some replicates, please be aware that all the three replicates must be repeated.

The area of the melting peaks of both PTT (at about 225 °C) and PET (at about 250 °C) shall be integrated.

Integration shall be performed drawing a linear baseline between fixed ranges of temperature as described in the test method.

Reporting

The Excel file "data_reporting_DSC_results.xls" contains the following worksheets:

1. EXAMPLE
2. Sample A
3. Sample B
4. Sample C
5. Sample D
6. Sample E
7. Sample F
8. General information

The worksheets from 2 to 8 have to be filled-in with results.

The PTT and PET's mass percentages in the fabric samples and in the calibration points will be automatically calculated based on the input data.

Please return the filled-in Excel file and your comments and remarks by e-mail (paola.piccinini@jrc.ec.europa.eu) by the end of August 2013 at the latest.

EUROPEAN COMMISSION
JOINT RESEARCH CENTRE

Institute for Health and Consumer Protection (IHCP)
Chemical Assessment and Testing Unit (CAT)

Method for the quantification of textile binary mixtures made of polytrimethylene terephthalate (PTT) and polyethylene terephthalate (PET) by DSC

IMPORTANT NOTE: Before starting the analysis, please carefully read the instructions, test method and Excel data reporting sheet in order to collect all required data.

1 SCOPE

This standard describes a method for the quantification of textile binary mixtures made of PTT and PET via DSC analysis.

2 FIELD OF APPLICATION

This method is applicable to textile binary mixtures of:

1. polyethylene terephthalate (PET)

with

2. polytrimethylene terephthalate (PTT)

3 PRINCIPLE

The method is based on differential scanning calorimetry (DSC) analysis of textile binary mixtures PTT/PET. The quantification is based on the integration of the melting peak of PTT (about 225 °C) and PET (about 250 °C) obtained during the heating cycle. The calibration curve is built using both pure PTT and PET manually separated from the binary mixture to be quantified.

4 APPARATUS AND MATERIALS

All specimens and pans shall be handled using clean gloves or tweezers. However, if sampling textile samples is too difficult with gloves, touch the test specimens only with clean hands.

4.1 Differential Scanning Calorimeter (DSC), the main features of which are as follows:

a) the capability to generate constant heating rate of 5 °C/min;
b) the capability to maintain the test temperature constant to within ± 0.5 °C for at least 5 min;
c) the capability to carry out step heating or any other heating mode;
d) a gas-flow rate of 50 ml/min, controllable to ± 10%;
e) temperature signals with 0.1 °C resolution and noise below 0.5 °C;
f) a recording device which is capable of recording the DSC curve, and of integrating the area between the curve and the visual baseline with an error of less than 2%;
g) a specimen holder assembly which has one or more holders for pans.

4.2 Aluminum pans, for test specimens and reference specimens, all made of the same material and of equal mass. The pans shall be physically and chemically inert under the measurement conditions to both the test specimen and the atmosphere.

The pans shall be able to be fitted with lids and sealed.

4.3 Analytical balance, capable of measuring the specimen mass with an accuracy of ± 0.01 mg

4.4 Standard reference materials

At least one of reference material, which has a suitable melting point in or near the temperature range to be examined, shall be used for the temperature calibration of the instrument.

At least one standard reference material, which has a suitable heat capacity and a melting point in or near the temperature range to be examined, shall be used for the energy or thermal-power calibration of the instrument.

4.4 Gas supply, nitrogen, analytical grade

4 SAMPLE PREPARATION

4.1 Manual separation

Manual separation of the binary mixture is performed to get a small quantity of pure PTT and PET needed to build up the calibration curve.

IMPORTANT NOTE: In this case the manual separation of the samples has been already done by the JRC and the extracted yarns of pure PTT and PET have been already homogenised.

4.2 Sampling

Use the specimens (both fabric and yarns) as received (do not pre-treat or dry them).

The test specimen shall be representative of the sample being examined and shall be prepared and handle with care, if possible with gloves or tweezers otherwise with clean hands.

After having weighed the first test specimen try to prepare the other test specimens with a similar weight.

4.2.1 Sampling of fabrics

Cut small squares (as little as possible) from the fabric sample (cuts must be parallel to the sample warp or weft). The squares must be cut at least one centimeter away from the selvedges (selvages).

Weigh and record the weight of one or more squares in order to reach the desired test specimen weight (5-8 mg).

Load the weighed test specimen into the pan as described in 7.2.

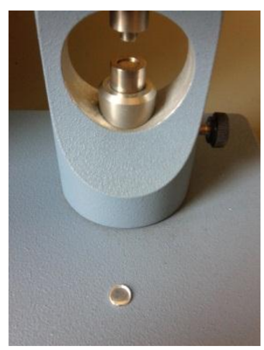
The typical procedure can be seen in the following pictures.

1

2

3

4



5

4.2.2 Sampling of yarns

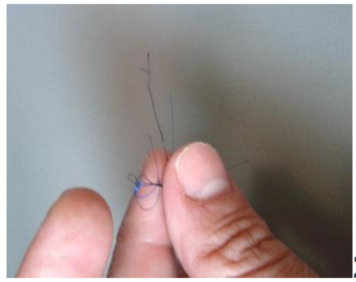
Weigh and record the weight of one or more yarns in order to reach the desired test specimen weight (5-8 mg).

Using your fingers roll those yarns up until you obtain a small ball that has to be closed in a DSC pan.

Load the weighed test specimen into the pan as described in 7.2.

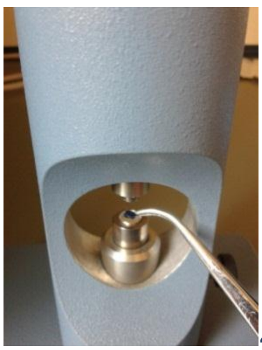
The typical procedure can be seen in the following pictures.

1



2

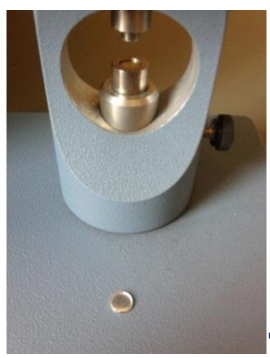
3



4

5

6



7

4.2.3 Sampling of calibration samples

4.2.3.1 Sampling of calibration sample 100 % PTT or 100 % PET

To prepare the test specimen containing 100 % PTT or 100 % PET, weigh and record the weight of one or more yarns of pure PTT or PET in order to reach the desired test specimen weight (5-8 mg).

Using your fingers roll those yarns up until you obtain a small ball that has to be closed in a DSC pan.

Load the weighed test specimen into the pan as described in 7.2.

4.2.3.1 Sampling of calibration samples containing both PTT and PET

Prepare five calibration samples, containing both PTT and PET, with mass percentage of PTT approximately of 20 %, 40 %, 55 %, 70 %, 85 %.

Weigh and record the weight of one or more yarns of pure PTT.

Successively, weigh and record the weight of one or more yarns of pure PET, so that the total mass of the calibration specimen (PTT + PET) is in the range 5-8 mg.

Using your fingers roll up both the weighed PTT and PET yarns together until you obtain a small ball that has to be closed in a DSC pan.

Load the weighed calibration specimen into the pan as described in 7.2.

Use the weight of pure PTT and PET to calculate the mass percentages of the calibration samples in terms of both PTT and PET percentages.

Note: *inserting, in the Excel file to report results, the mass of PTT and PET yarns used to prepare the calibration points their mass percentages will be automatically calculated.*

5 CALIBRATION OF THE INSTRUMENT

5.1 General

Calibrate the energy and temperature measurement devices of the calorimeter at least in accordance with the instrument manufacturer's recommendations.

Note: *Calibration is affected by:*

- *the type of calorimeter used;*
- *the gas used and its flow rate;*
- *the type of specimen pan used, its dimensions and its position in the specimen holder;*
- *the mass of the test specimen;*
- *the heating and cooling rates;*
- *the type of cooling system used.*

It is therefore advisable to define the conditions of the actual determination as precisely as possible and carry out the calibration using the same conditions.

It is advisable carry out calibrations regularly. It is considered good practice to check the temperature and energy measurement devices using standard reference materials *which have melting points close to the temperature range used for the material being analysed.*

5.2 Temperature calibration

Before starting analyses, carry out the temperature calibration as follows:

- use at least one standard reference material, if possible use two or more standard reference materials;

- determine the transition temperatures for the standard reference materials under the same conditions as those to be used for the test specimen (temperature program 150 °C – 5 °C/min – 300 °C; nitrogen flow rate, 50 ml/min). The transition temperatures of the standard reference materials are defined as the intercept of the extrapolated baseline and the tangent to the leading flank of the transition peak at the point of maximum gradient (i.e. the extrapolated onset temperature);

- determine the temperature calibration function by comparison of the nominal values with the recorded values, unless it can be obtained automatically by an associated computer system by feeding in the nominal and recorded values

Temperature calibration shall be performed each time the test conditions are changed. Temperature calibration checks shall be carried out at regular intervals. The repeatability of such checks shall be better than 2%.

5.3 Energy or thermal-power calibration

Before starting analyses, carry out the energy or thermal-power calibration as follows:

- use at least one standard reference material, if possible use two or more standard reference materials;

- examine the standard materials under the same conditions as those which will be used for the test specimen (temperature program 150 °C – 5 °C/min – 300 °C; nitrogen flow rate, 50 ml/min);

- record the plot of E versus temperature for the heat of transition or heat capacity;

- determine the energy or thermal-power calibration function by comparison of the nominal values with the recorded values, unless it can be obtained automatically by an associated computer system by feeding in the nominal and recorded values.

Energy calibration checks shall be carried out at regular intervals. The repeatability of such checks shall be better than 2%.

6 PROCEDURE

6.1 Setting up the apparatus

Switch on the instrument at least one hour prior to any testing to allow the electronic to temperature-equilibrate.

Select a nitrogen flow rate of 50 ml/min.

Note: *It is advisable to protect the instrument from air draughts, exposure to direct sunlight and/or sharp changes in temperature, pressure or electric supply during measurements.*

6.2 Loading the test specimen and calibration specimen into the pan

Do not handle pan with bare hands, use either tweezers or gloves. This should apply also to test material, however, if sampling textile samples is too difficult with gloves, touch the test specimens only with clean hands.

Select aluminum pans of the appropriate volume, ensuring that they are clean and of equal mass.

Use two identical pans, one for the test specimen and one empty as reference specimen.

Use a specimen mass of 5 mg to 8 mg for the analysis. The accuracy of weighing shall be to the nearest 0.01 mg.

Load the weighed test specimen or calibration specimen into a pan and seal it with its lid using a press.

Ensure that the bottom of the pan is flat, if this is not the case discard it and start again. Good contact between the pans and the specimen holders is crucial to obtaining good data.

Visually inspect the closed pan to check if any piece of fabric or yarn is stuck outside, in this case discard the pan and start again.

Make 3 pin holes in the upper lid to allow the flow of the inert atmosphere inside the pan.

Note: *some yarn samples have a high volume, which can pose an extra difficulty in sealing the pan with the lid without leaving pieces of yarn outside.*

6.3 Prepare the reference pan

Do not handle pan with bare hands, use either tweezers or gloves.

Take an aluminum pan, of the same material and weigh of the ones used for the test specimen, and seal it with its lid using a press.

Ensure that the bottom of the pan is flat, if this is not the case discard the pan and start again. Good contact between the pan and the specimen holder is crucial to obtaining good data.

Make 3 pin holes in the upper lid to allow the flow of the inert atmosphere inside the pan.

6.4 Insertion of pans in the instruments

Use tweezers or any other suitable tool to place the reference pan and test specimen or calibration specimen pan in the specimen holders, ensuring that there is good contact between the test specimen and the pan and between the pan and holder. Close the cover of the specimen older assembly.

Before starting the analysis of the test specimen, insert as input data its mass, expressed in the mass unit requested by the instrument.

6.5 Temperature-scanning measurement

Perform and record a thermal cycle at a rate of 5 °C/min starting at 150 °C up to 300 °C.

Bring the apparatus back to room temperature and take out the pan containing the test specimen.

Examine the pan to determine if any deformation of it or specimen overflow have occurred.

If the cell becomes contaminated from specimen overflow, clean the cell in accordance with the manufacturer’s instructions and confirm that the calibration still valid using at least one temperature and enthalpy reference standard.

Load another test specimen pan and start the following analysis.

7 CALCULATION AND EXPRESSION OF RESULTS

7.1 Linear integration using fixed ranges of temperature

The integration is a very important and critical step for the quantification.

The area of the melting peaks of both PTT (at about 225 °C) and PET (at about 250 °C) shall be integrated.

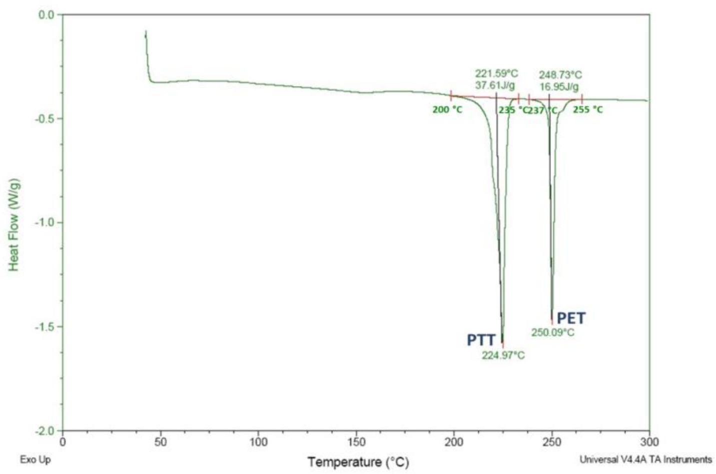
In certain cases the PTT and PET melting peaks could show shoulders, the whole peak including the possible shoulders shall be considered and integrated as one.

Integration shall be performed drawing a linear baseline between fixed ranges of temperature.

Zoom one thermogram of the test specimen under quantification so that the peaks under evaluation cover at least 25% of full scale.

The start and end point of the integrations (in °C) must be chosen appropriately and kept constant for all the thermograms in the same set of analyses. This means that the 14 thermograms needed to quantify each test specimen (11 analyses for the calibration curve plus three replicates for the test specimen) shall be integrated using the same fixed initial and final temperatures for the PTT and PET melting peaks. The start and end point of the integrations can be kept constant either selecting the "fixed range" feature of the DSC software, when available, or doing it manually on each thermogram.

An example can be seen below. In this case the fixed ranges 200 – 235 °C and 237 – 255 °C have been selected for the integration of PTT and PET respectively and have to be kept constant in the integration of the same set of analyses.



Be advised that the selected fixed initial and final temperatures to integrate the PTT and PET melting peaks could have to be changed among different blind samples in order to optimise the integrations according to the different shape of the peaks.

7.2 Build the calibration curve

To quantify each test specimen, calculate the linear regression slopes of the two calibration curves obtained integrating either PTT or PET melting peaks.

Using linear regression, interpolate on the correspondent calibration graph the concentration in mass percentage of either PTT or PET.

The concentration of PTT or PET in the sample (expressed in mass percentage) is calculated as follows:

$$\%\ \text{polymer} = \frac{polymer\ enthalpy\ of\ fusion\ (Area\ of\ polymer\ melting\ peak)\left(\frac{J}{g}\right)}{slope\ of\ the\ linear\ calibration\ curve}$$

Note: the data reporting Excel sheet contains all the calculations to provide the final result in terms of mass percentage of PTT of the six blind samples.

You need to insert:

- *the PTT and PET mass used to prepare the calibration points, expressed in mg;*
- *the PTT and PET enthalpy of fusions, expressed in J/g, obtained by integration;*
- *the linear regression slopes of the calibration curves, based on PTT or PET melting peaks, which are automatically calculated by the software.*

Note: please report any deviations to the method protocol in the provided Excel sheet.

Participant's comments

	Sample A
Lab01	
Lab02	The PET peak occurs in the PTT peak. I take the choice to focus on the PTT peak which is clearer.
Lab03	
Lab04	In this case (Sample A) areas of melting peaks of both fibres slightly overleaped.
Lab05	
Lab06	room temperature: 25°C air humidity: 55% pure PTT and PET measured at 8-Aug-13 temperature range for calibration curve points: PTT: 200-232 / PET: 232-256, temperature range for fabric: PTT: 200-232, PET: 232-256 the PET signal shows 2 peaks (not pure) the sample was hard to integrate, because there was no optimal baseline
Lab07	
Lab08	The fabric samples showed two peaks corresponding to PET. The two peaks were integrated separately so that the entirety of the peaks was integrated. The complete temperature range is stated above. The individual ranges used were; 230.8 - 240.1 240.1 - 252.65
Lab09	
Lab10	
Lab11	PTT - sharp big peak; PET Fabric - one sharp small peak+one broad small peak
Lab12	
Lab13	DSC heat cycle: Load at RT, Heat @30ºC/min to 140ºC, Heat @ 5ºC/min to 285ºC. No holes in pan lids.
Lab14	
Lab15	analysis made after lack of power and equipment technician intervention

	Sample B
Lab01	
Lab02	
Lab03	
Lab04	
Lab05	
Lab06	room temperature: 25°C air humidity: 55% 2 repetitions at the next day necessary: pure PTT and a PTT/PET-mixture good sample, good baseline separation
Lab07	
Lab08	
Lab09	
Lab10	
Lab11	Fabric: PTT - sharp peak, small shoulder on the left side; PET - small shoulder on the right side
Lab12	I have taken 20%, 40%, 55%, 70% and 85% PET instead of 20%,40%,55%,70% and 85% PTT
Lab13	DSC heat cycle: Load at RT, Heat @30ºC/min to 140ºC, Heat @ 5ºC/min to 285ºC. No holes in pan lids.
Lab14	
Lab15	analysis made after lack of power and equipment technician intervention

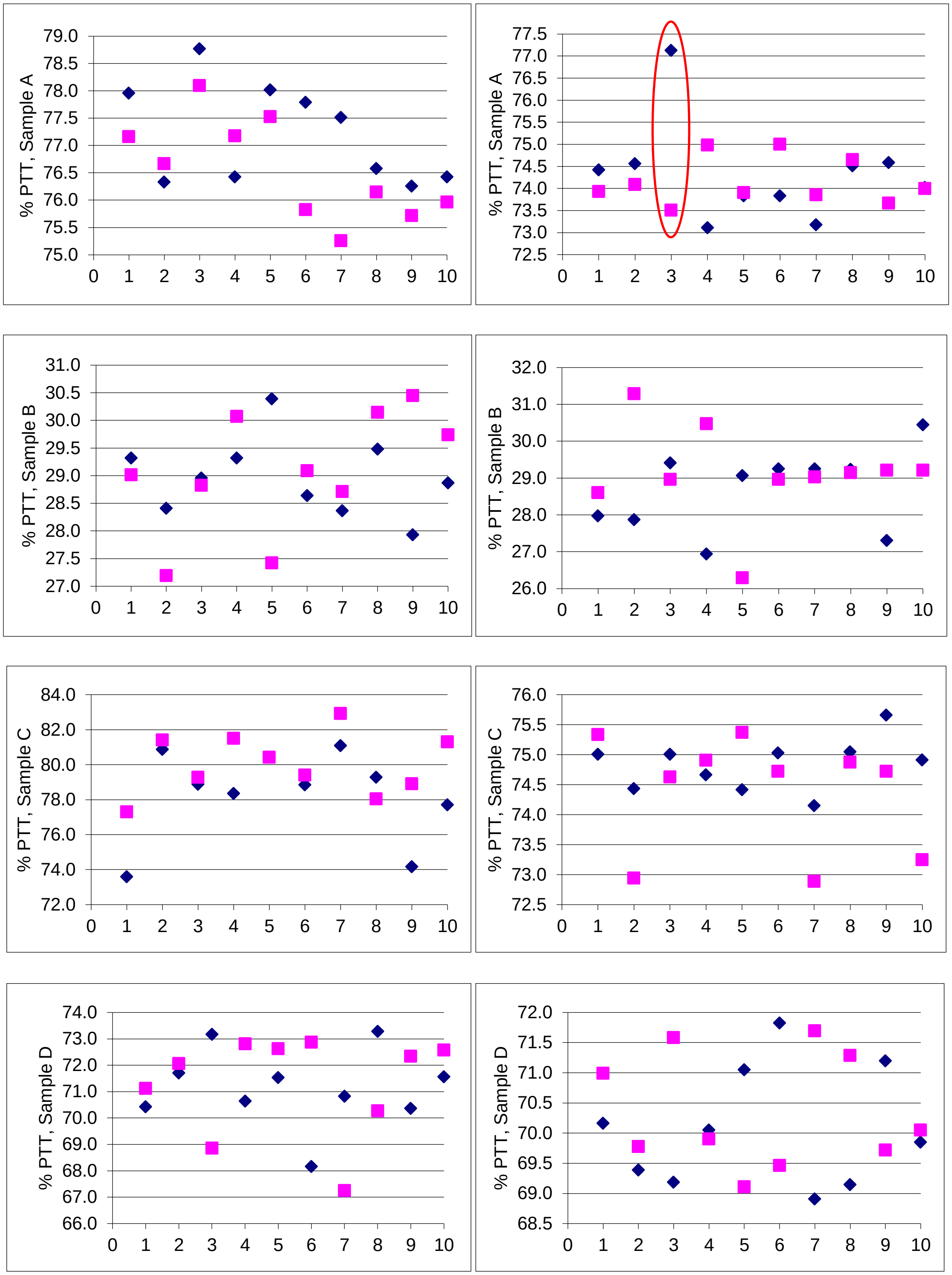
	Sample C
Lab01	No ha sido posible deshilachar el tejido para cortar los cuadraditos sin error.
Lab02	
Lab03	
Lab04	
Lab05	Pure PET fibres were contaminated by the PTT fibres, hence for the analysis of pure PET samples were prepared 5 samples, found in this group of 3 samples of pure PET, and these have been developed.
Lab06	room temperature: 25°C air humidity: 55% good sample, good baseline separation
Lab07	
Lab08	Reference 5 (85%) repeated as the calibration curve R2 was less than 0.99 as a result of this reference point. The initial enthalpy for the anomalous result is PTT - 56.31 J/g, PET - 7.90 J/g
Lab09	
Lab10	
Lab11	Fabric: PTT - sharp peak; PET - sharp peak
Lab12	
Lab13	DSC heat cycle: Load at RT, Heat @30ºC/min to 140ºC, Heat @ 5ºC/min to 285ºC. No holes in pan lids.
Lab14	
Lab15	analysis made after lack of power and equipment technician intervention

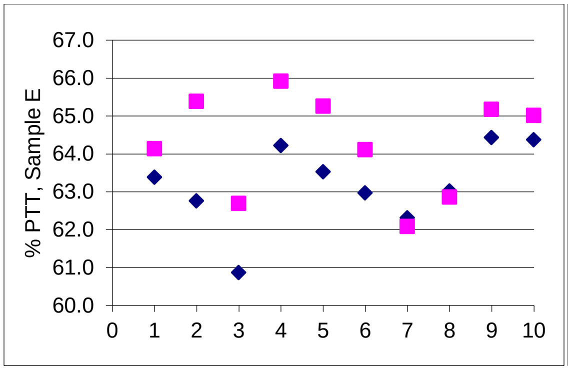
	Sample D
Lab01	
Lab02	
Lab03	
Lab04	
Lab05	
Lab06	room temperature: 25°C air humidity: 55% temperature range for calibration curve points: PTT: 200-232 / PET: 232-265 temperature range for fabric: PTT: 200-235, PET: 235-262 measurements over two days (next day PTT 80%/PET and the fabrics)
Lab07	
Lab08	Reference 5 (85%) repeated as the calibration curve R2 was less than 0.99 as a result of this reference point. The initial enthalpy for the anomalous result is PTT - 44.63 J/g, PET - 19.90 J/g
Lab09	
Lab10	
Lab11	
Lab12	
Lab13	DSC heat cycle: Load at RT, Heat @30ºC/min to 140ºC, Heat @ 5ºC/min to 285ºC. No holes in pan lids.
Lab14	
Lab15	analysis made after lack of power and equipment technician intervention

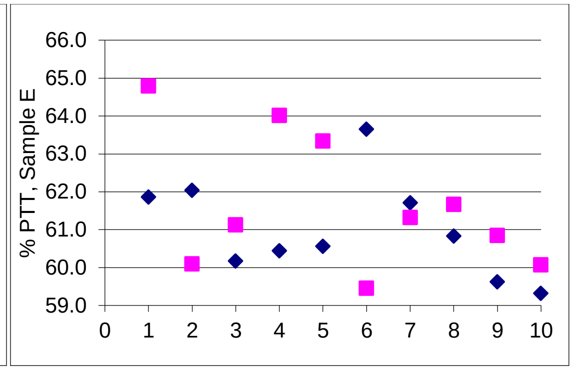
	Sample E
Lab01	
Lab02	
Lab03	
Lab04	
Lab05	
Lab06	room temperature: 22°C air humidity: 46% temperature range for calibration curve points: PTT: 200-232 / PET: 232-265 temperature range for fabric: PTT: 200-232, PET: 232-262
Lab07	Broad peak for pure PET between 241oC and 259 ºC. Double peak, followed by a broad peak, for PET in fabric between 245oC and 259 ºC.
Lab08	Reference 1 (20%) repeated as the calibration curve R2 was less than 0.99 as a result of this reference point. The initial enthalpy for the anomalous result is PTT - 12.34 J/g, PET - 44.82 J/g
Lab09	
Lab10	
Lab11	PTT - sharp peak; PET - sharp peak, small left shoulder
Lab12	it looks like there are to different threads in the pure sample PTT and pure sample PET
Lab13	DSC heat cycle: Load at RT, Heat @30ºC/min to 140ºC, Heat @ 5ºC/min to 285ºC. No holes in pan lids. PTT and PET samples manually separated from spare fabric because "Pure PTT" was contaminated with PET.
Lab14	
Lab15	analysis made after lack of power and equipment technician intervention

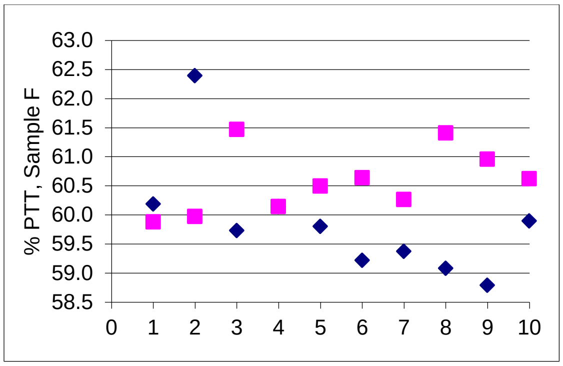
	Sample F
Lab01	
Lab02	
Lab03	
Lab04	In this case (Sample A) areas of melting peaks of both fibres slightly overlapped.
Lab05	
Lab06	room temperature: 21°C air humidity: 50% temperature range for calibration curve points: PTT: 200-232 / PET: 232-259 temperature range for fabric: PTT: 200-232, PET: 232-259 the PET-curve shows 2 peaks, the fabric shows 3 peaks
Lab07	Two peaks showed for pure PET. The two peaks (broad, broad) appeared between 231 and 258 ºC. Three peaks showed for PET in fabric. The three peaks (sharp, broad, sharp) appeared between 231 and 258 ºC
Lab08	
Lab09	
Lab10	
Lab11	PET - 3 peaks!
Lab12	On Sample LC12-Sample F45 Fabric, there is a melting peak at 253, which I have subtracted from the PET result.
Lab13	DSC heat cycle: Load at RT, Heat @30ºC/min to 140ºC, Heat @ 5ºC/min to 285ºC. No holes in pan lids. "Pure PTT" and "Pure PET" fibres provided were obviously not obtained from Fabric F. Fabric F PET fibres are both light and dark. PET fibres provided are only dark.
Lab14	
Lab15	analysis made after lack of power and equipment technician intervention

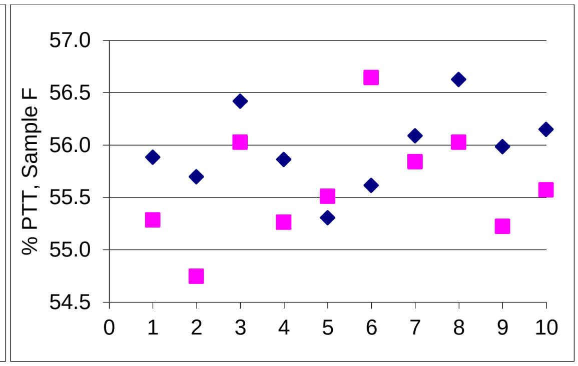
Graphic representation of the homogeneity study, PTT quantification *via* calibration curve on PTT (left) and PET (right) peaks.




% PTT, Sample E
67.0
66.0
65.0
64.0
63.0
62.0
61.0
60.0
0 1 2 3 4 5 6 7 8 9 10


% PTT, Sample E
66.0
65.0
64.0
63.0
62.0
61.0
60.0
59.0
0 1 2 3 4 5 6 7 8 9 10


% PTT, Sample F
63.0
62.5
62.0
61.5
61.0
60.5
60.0
59.5
59.0
58.5
0 1 2 3 4 5 6 7 8 9 10


% PTT, Sample F
57.0
56.5
56.0
55.5
55.0
54.5
0 1 2 3 4 5 6 7 8 9 10

PTT quantification *via* calibration curve based on PTT peak

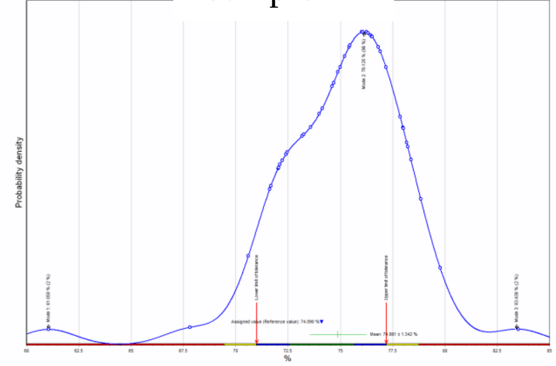
Table A, B and C: original values, cell means and cell standard deviation, respectively.

Table A	Sample code					
Lab code	A	B	C	D	E	F
Lab 1	72.46	28.32	79.82	72.28	54.57	59.94
	73.17	29.81	79.40	72.00	56.59	58.90
	73.24	30.53	79.53	72.86	54.83	58.94
Lab 2	79.77	30.01	80.50	72.56	64.52	61.22
	83.51	30.04	78.96	70.47	61.38	61.78
	77.18	30.20	78.60	67.05	64.88	56.52
Lab 3	78.00	29.40	74.17	70.40	64.28	57.52
	77.99	29.24	72.81	69.59	62.62	58.40
	78.84	29.97	77.88	71.34	65.05	55.87
Lab 4	70.60	34.98	77.37	73.50	64.13	58.03
	76.32	34.58	77.02	76.12	67.42	62.49
	72.40	35.29	77.18	74.82	65.50	61.66
Lab 5	76.07	28.40	75.39	71.74	63.29	59.19
	76.52	28.75	77.63	70.67	63.80	59.26
	76.02	28.33	77.37	69.79	63.48	59.78
Lab 6	74.98	28.83	75.89	70.54	64.53	60.56
	75.20	28.65	77.93	69.89	62.92	60.13
	76.49	29.41	76.01	70.94	63.66	57.73
Lab 7	73.57	29.18	77.25	71.75	72.46	59.18
	72.22	28.94	75.56	71.14	68.49	58.74
	72.04	29.57	77.38	69.16	70.30	57.12
Lab 8	61.05	28.77	71.61	69.12	60.69	54.98
	76.07	31.19	71.18	71.78	62.62	64.71
	76.80	28.45	69.08	74.78	71.09	55.33
Lab 9	78.22	39.66	78.45	74.93	69.43	65.62
	75.46	38.12	79.82	74.71	66.73	61.81
	67.80	37.68	78.66	75.34	71.01	65.21
Lab 10	74.61	29.32	76.81	71.51	63.43	59.68
	73.99	30.63	77.46	71.78	62.44	59.15
	74.67	29.07	75.77	74.48	64.22	59.85
Lab 11	78.17	24.81	76.74	70.23	63.72	59.14
	78.37	27.96	70.67	70.74	62.15	59.34
	77.86	26.94	74.31	71.45	65.80	55.84
Lab 12	72.02	28.66	72.27	70.52	63.79	59.63
	71.68	29.11	73.13	70.19	64.19	63.31
	72.11	28.14	71.89	68.77	64.04	61.69
Lab 13	76.24	29.35	77.57	72.21	63.47	59.33
	76.24	27.95	76.94	70.85	63.05	59.37
	76.44	28.01	77.25	69.97	63.93	58.45
Lab 14	74.88	29.62	76.81	73.22	63.04	59.07
	76.08	29.52	76.52	72.77	64.47	60.34
	75.42	28.63	78.08	73.92	63.05	56.18
Lab 15	71.62	27.23	71.68	69.54	64.34	57.89
	74.13	26.41	71.99	74.00	62.16	62.33
	76.90	27.26	72.57	73.08	61.93	59.76

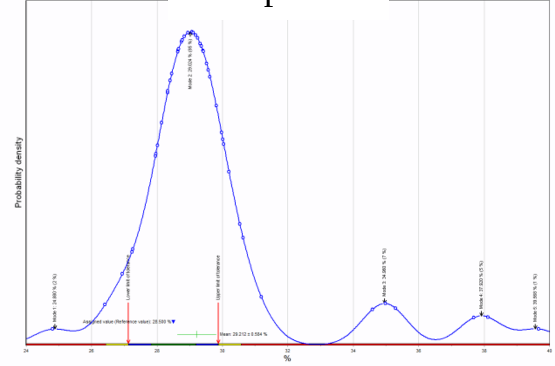
Table B	Sample code					
Lab code	**A**	**B**	**C**	**D**	**E**	**F**
Lab 1	72.96	29.55	79.58	72.38	55.33 (h**,G*)	59.26
Lab 2	80.15 (h*)	30.08	79.35	70.03	63.59	59.84
Lab 3	78.28	29.54	74.95	70.44	63.98	57.26
Lab 4	73.11	34.95 (G**)	77.19	74.81 (h*)	65.68	60.73
Lab 5	76.20	28.49	76.80	70.73	63.52	59.41
Lab 6	75.56	28.96	76.61	70.46	63.70	59.47
Lab 7	72.61	29.23	76.73	70.68	70.41 (h*)	58.35
Lab 8	71.31	29.47	70.62 (h*)	71.89	64.80	58.34
Lab 9	73.83	38.48 (h**,G**)	78.97	74.99 (h*)	69.06	64.21 (h**,G*)
Lab 10	74.42	29.67	76.68	72.59	63.36	59.56
Lab 11	78.13	26.57	73.91	70.81	63.89	58.11
Lab 12	71.94	28.64	72.43	69.83	64.01	61.54
Lab 13	76.31	28.44	77.25	71.01	63.48	59.05
Lab 14	75.46	29.26	77.14	73.30	63.52	58.53
Lab 15	74.22	26.97	72.08	72.21	62.81	59.99

Table C	Sample code					
Lab code	**A**	**B**	**C**	**D**	**E**	**F**
Lab 1	0.43	1.13	0.22	0.44	1.10	0.59
Lab 2	3.18	0.10	1.01	2.78 (k*)	1.93	2.89
Lab 3	0.49	0.38	2.62 (k*)	0.88	1.24	1.28
Lab 4	2.92	0.36	0.18	1.31	1.65	2.37
Lab 5	0.28	0.23	1.23	0.98	0.26	0.32
Lab 6	0.82	0.40	1.14	0.53	0.81	1.53
Lab 7	0.84	0.32	1.02	1.35	1.99	1.08
Lab 8	8.89 (k**,C**)	1.49 (k*)	1.35	2.83 (k*)	5.53 (k**,C**)	5.51 (k**,C**)
Lab 9	5.39 (k*,C**)	1.04	0.74	0.32	2.16	2.09
Lab 10	0.38	0.84	0.85	1.64	0.89	0.37
Lab 11	0.25	1.60 (k*)	3.05 (k**,C*)	0.61	1.83	1.97
Lab 12	0.23	0.49	0.64	0.93	0.20	1.84
Lab 13	0.12	0.79	0.32	1.13	0.44	0.52
Lab 14	0.60	0.55	0.83	0.58	0.82	2.13
Lab 15	2.64	0.48	0.45	2.35	1.33	2.23

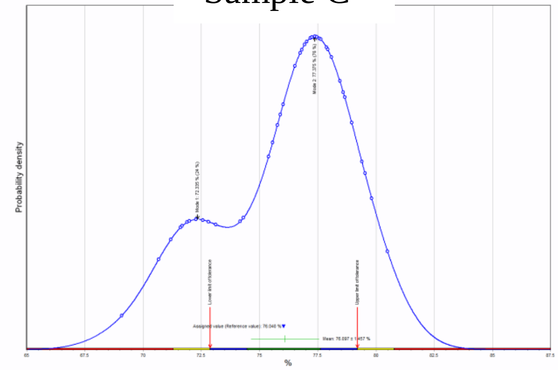
Sample A



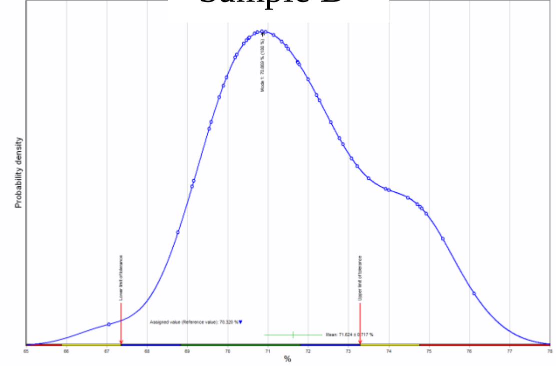
Sample B



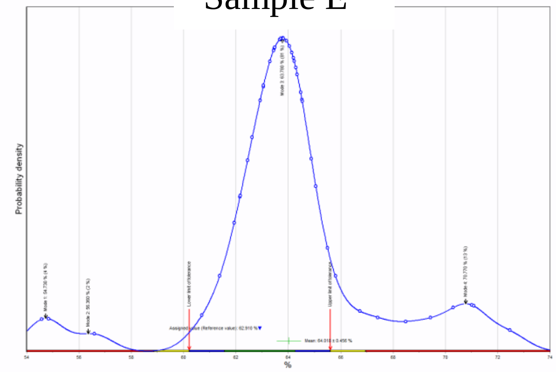
Sample C



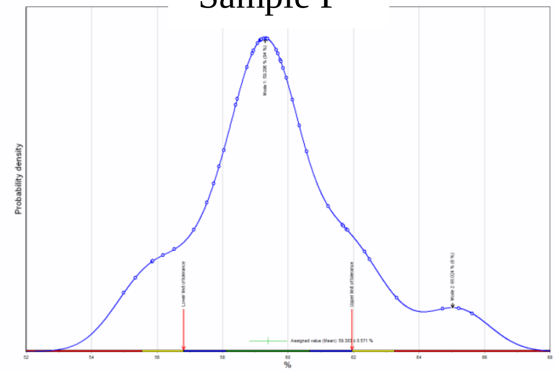
Sample D

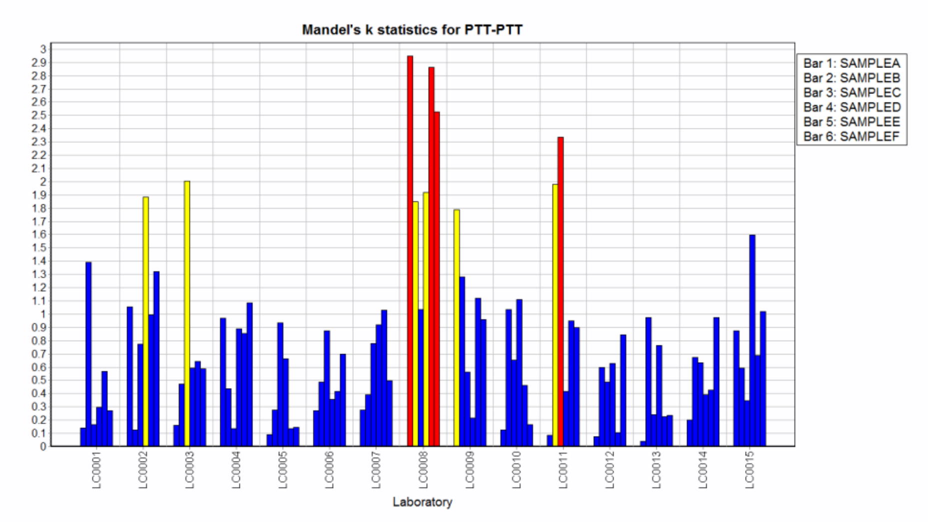


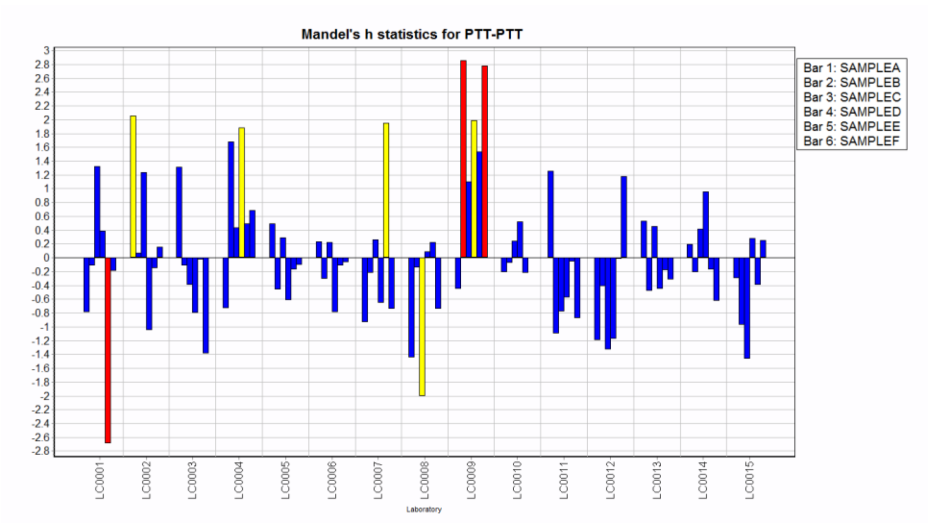
Sample E

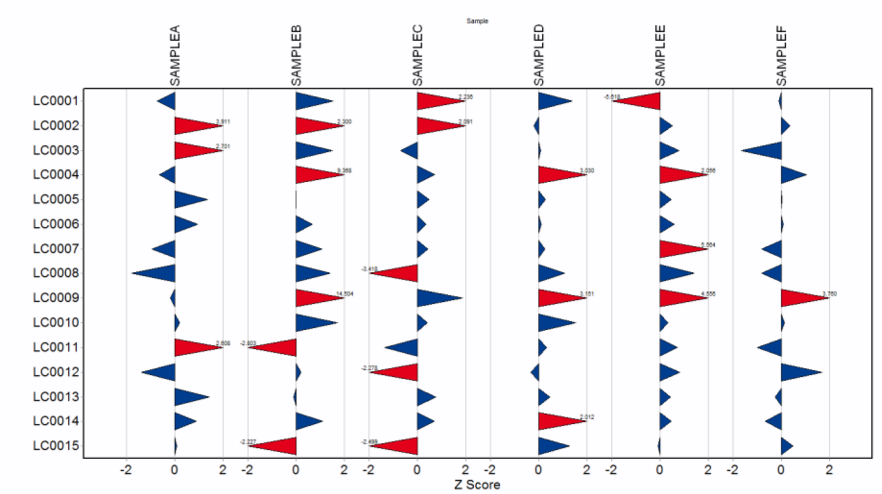


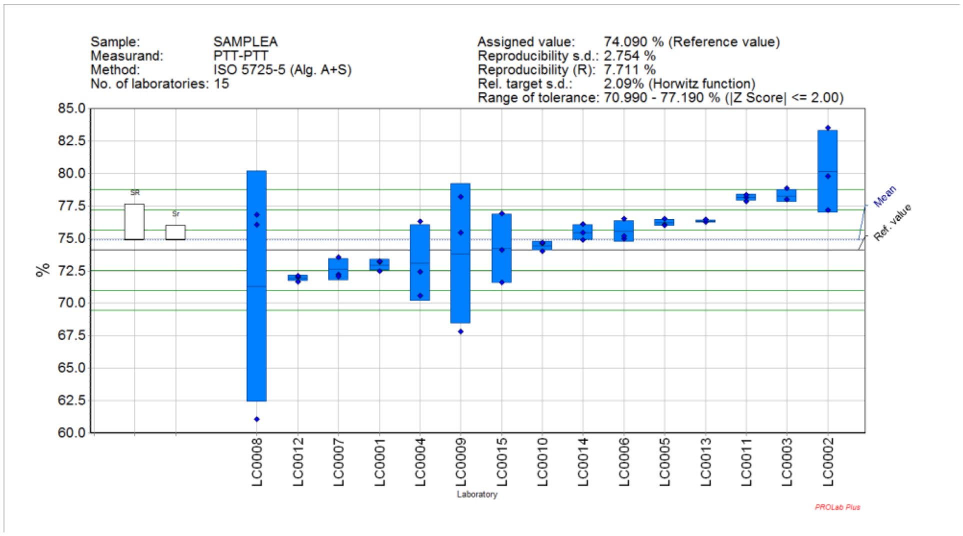
Sample F



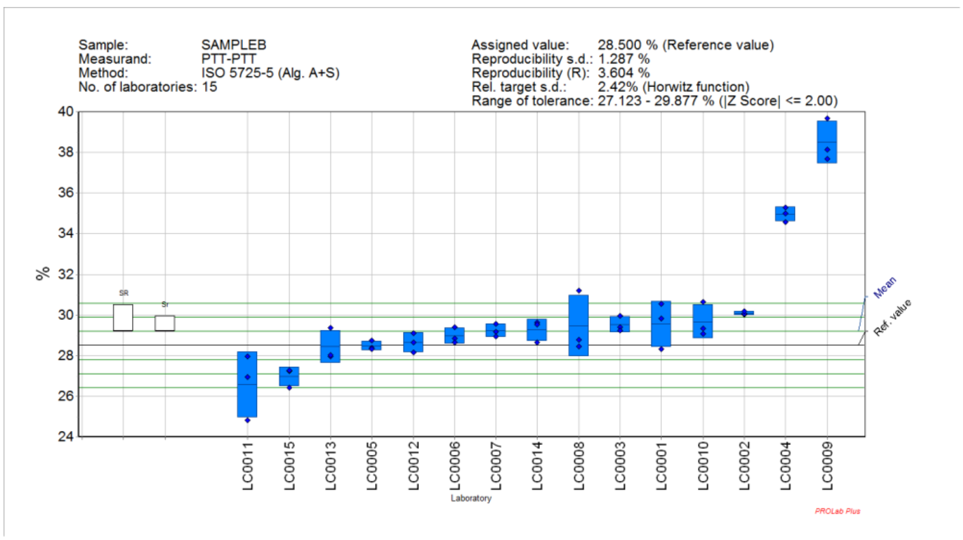

Mandel's k statistics for PTT-PTT
Bar 1: SAMPLEA
Bar 2: SAMPLEB
Bar 3: SAMPLEC
Bar 4: SAMPLED
Bar 5: SAMPLEE
Bar 6: SAMPLEF
Laboratory


Mandel's h statistics for PTT-PTT
Bar 1: SAMPLEA
Bar 2: SAMPLEB
Bar 3: SAMPLEC
Bar 4: SAMPLED
Bar 5: SAMPLEE
Bar 6: SAMPLEF
Laboratory

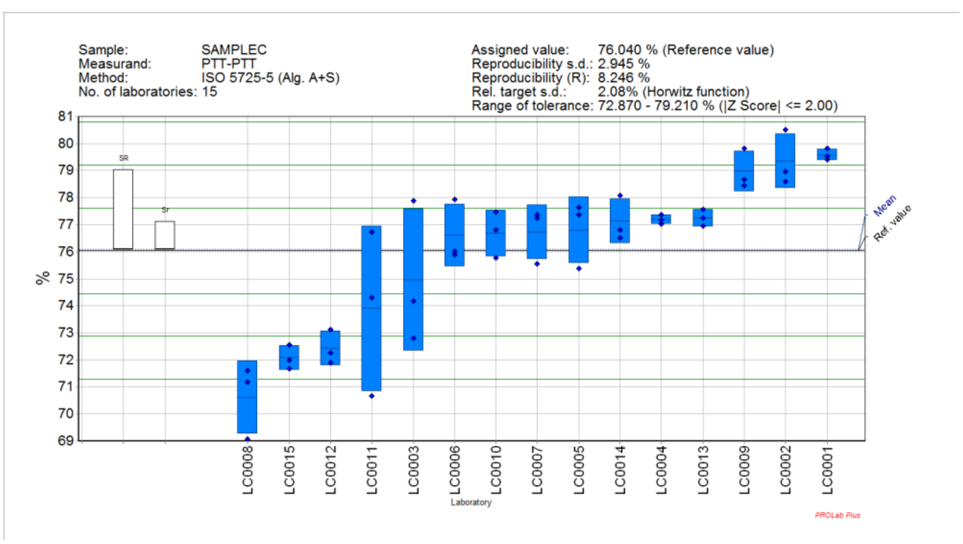

Sample
SAMPLEA
SAMPLEB
SAMPLEC
SAMPLED
SAMPLEE
SAMPLEF
Z Score
PROLab Plus



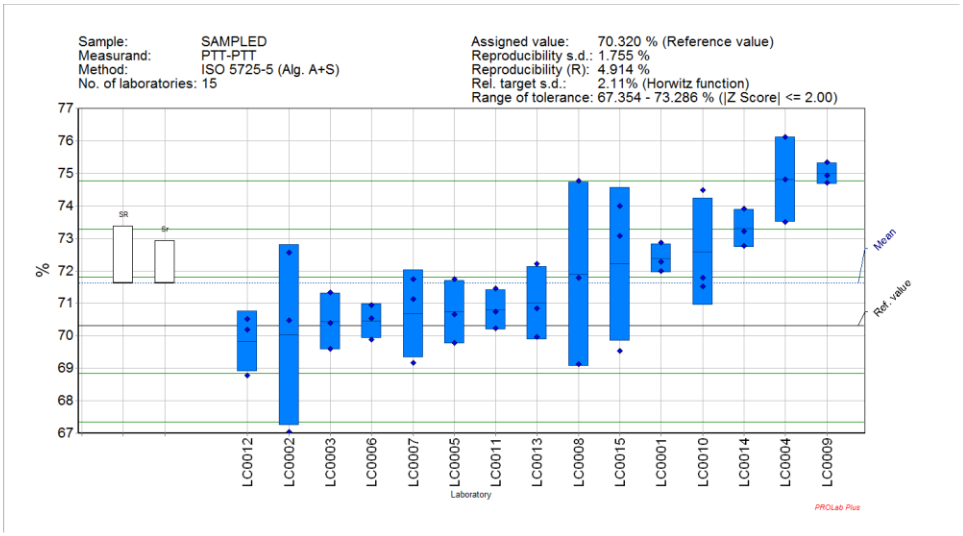
Sample: SAMPLEA
Measurand: PTT-PTT
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 74.090 % (Reference value)
Reproducibility s.d.: 2.754 %
Reproducibility (R): 7.711 %
Rel. target s.d.: 2.09% (Horwitz function)
Range of tolerance: 70.990 - 77.190 % (|Z Score| <= 2.00)
%
SR
Sr
Mean
Ref. value
LC0008 LC0012 LC0007 LC0001 LC0004 LC0009 LC0015 LC0010 LC0014 LC0006 LC0005 LC0013 LC0011 LC0003 LC0002
Laboratory
PROLab Plus



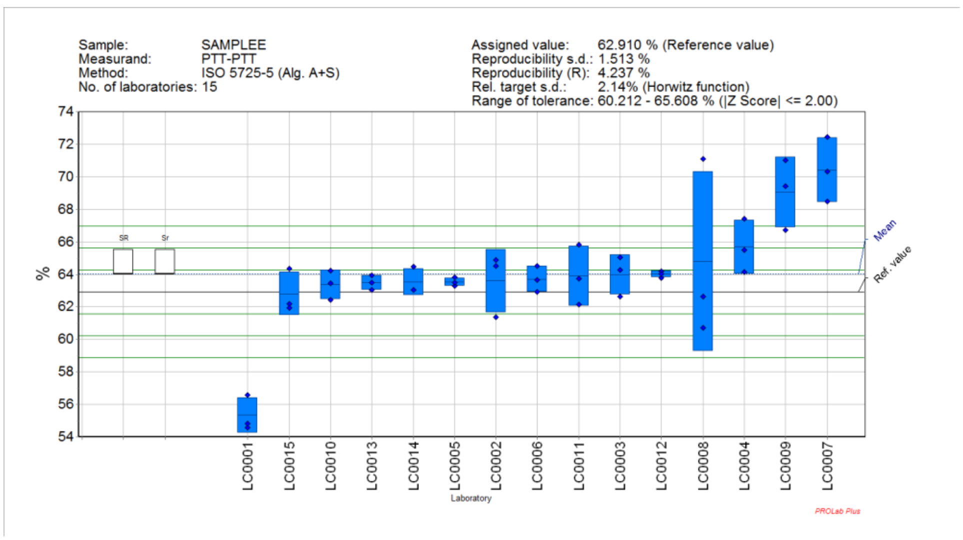
Sample: SAMPLEB
Measurand: PTT-PTT
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 28.500 % (Reference value)
Reproducibility s.d.: 1.287 %
Reproducibility (R): 3.604 %
Rel. target s.d.: 2.42% (Horwitz function)
Range of tolerance: 27.123 - 29.877 % (|Z Score| <= 2.00)
%
SR
Sr
Mean
Ref. value
LC0011 LC0015 LC0013 LC0005 LC0012 LC0006 LC0007 LC0014 LC0008 LC0003 LC0001 LC0010 LC0002 LC0004 LC0009
Laboratory
PROLab Plus



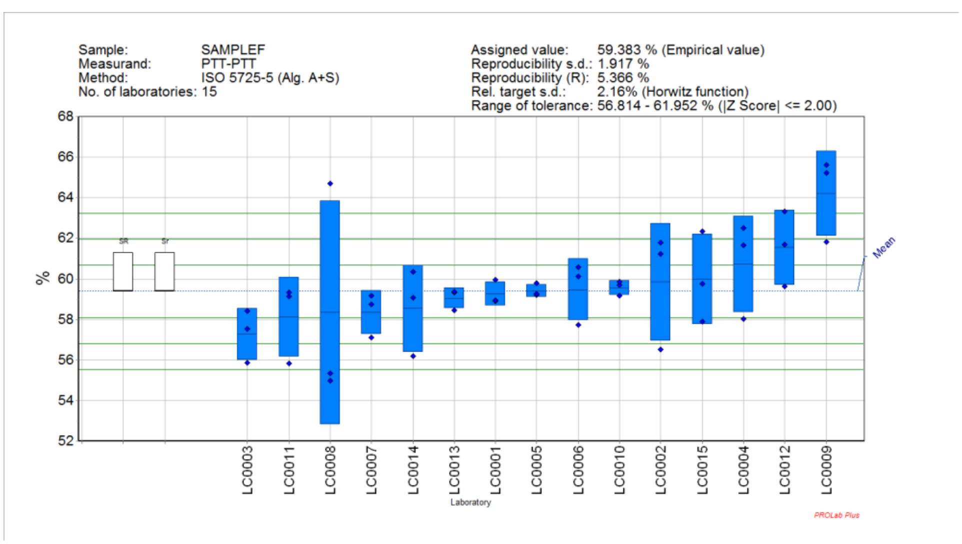
Sample: SAMPLEC
Measurand: PTT-PTT
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 76.040 % (Reference value)
Reproducibility s.d.: 2.945 %
Reproducibility (R): 8.246 %
Rel. target s.d.: 2.08% (Horwitz function)
Range of tolerance: 72.870 - 79.210 % (|Z Score| <= 2.00)
%
SR
Sr
Mean
Ref. value
LC0008 LC0015 LC0012 LC0011 LC0003 LC0006 LC0010 LC0007 LC0005 LC0014 LC0004 LC0013 LC0009 LC0002 LC0001
Laboratory
PROLab Plus



Sample: SAMPLED
Measurand: PTT-PTT
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 70.320 % (Reference value)
Reproducibility s.d.: 1.755 %
Reproducibility (R): 4.914 %
Rel. target s.d.: 2.11% (Horwitz function)
Range of tolerance: 67.354 - 73.286 % (|Z Score| <= 2.00)
%
SR
Sr
Mean
Ref. value
LC0012 LC0002 LC0003 LC0006 LC0007 LC0005 LC0011 LC0013 LC0008 LC0015 LC0001 LC0010 LC0014 LC0004 LC0009
Laboratory
PROLab Plus



Sample: SAMPLEE
Measurand: PTT-PTT
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 62.910 % (Reference value)
Reproducibility s.d.: 1.513 %
Reproducibility (R): 4.237 %
Rel. target s.d.: 2.14% (Horwitz function)
Range of tolerance: 60.212 - 65.608 % (|Z Score| <= 2.00)
%
SR
Sr
Mean
Ref. value
LC0001 LC0015 LC0010 LC0013 LC0014 LC0005 LC0002 LC0006 LC0011 LC0003 LC0012 LC0008 LC0004 LC0009 LC0007
Laboratory
PROLab Plus



Sample: SAMPLEF
Measurand: PTT-PTT
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 59.383 % (Empirical value)
Reproducibility s.d.: 1.917 %
Reproducibility (R): 5.366 %
Rel. target s.d.: 2.16% (Horwitz function)
Range of tolerance: 56.814 - 61.952 % (|Z Score| <= 2.00)
%
SR
Sr
Mean
LC0003 LC0011 LC0008 LC0007 LC0014 LC0013 LC0001 LC0005 LC0006 LC0010 LC0002 LC0015 LC0004 LC0012 LC0009
Laboratory
PROLab Plus

PTT quantification *via* calibration curve based on PET peak

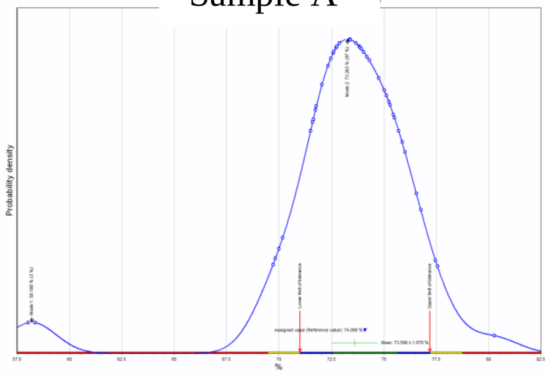
Table A, B and C: original values, cell means and cell standard deviation, respectively.

Table A	Sample code					
Lab code	**A**	**B**	**C**	**D**	**E**	**F**
Lab 1	72.76	23.34	76.93	70.73	53.88	57.51
	77.47	28.32	76.72	70.15	55.10	57.39
	75.46	24.81	78.38	68.78	53.68	56.94
Lab 2	69.99	29.06	73.08	67.59	64.92	54.03
	70.17	25.75	73.88	69.13	60.56	53.70
	71.74	26.74	73.86	70.62	59.13	58.16
Lab 3	72.88	27.82	75.01	68.54	65.41	56.78
	72.46	28.40	74.81	68.94	64.34	57.33
	74.76	30.50	73.12	68.57	65.43	57.70
Lab 4	77.56	20.27	73.98	68.25	60.63	58.82
	73.36	24.71	73.59	66.96	58.35	53.84
	75.71	23.90	73.83	67.60	58.06	55.02
Lab 5	72.33	27.48	75.07	68.50	61.70	56.19
	72.73	27.48	75.24	69.58	61.40	56.15
	73.39	26.88	75.26	69.89	60.48	56.34
Lab 6	72.58	29.02	75.95	69.75	62.43	57.33
	73.82	29.49	74.74	69.81	61.77	57.53
	72.33	32.84	75.51	69.63	62.38	57.02
Lab 7	71.63	30.88	76.37	71.02	63.08	53.59
	74.30	30.88	78.47	70.56	61.42	53.67
	69.82	29.35	75.81	71.77	60.45	57.02
Lab 8	80.27	26.67	76.15	71.02	66.05	61.70
	75.87	24.46	76.91	71.53	62.89	57.17
	75.02	32.04	77.76	71.06	64.42	61.76
Lab 9	58.36	53.82	74.01	60.46	62.57	51.38
	58.02	53.02	74.44	64.67	63.03	57.81
	69.71	51.80	72.89	64.71	61.73	51.77
Lab 10	71.78	30.43	75.77	67.79	60.03	54.80
	71.60	25.68	75.83	67.60	60.57	54.56
	72.61	29.37	76.76	66.71	60.54	55.37
Lab 11	74.20	40.93	71.50	68.25	60.70	57.57
	74.04	37.61	71.30	68.97	59.64	56.69
	73.86	36.75	70.72	69.12	59.76	57.12
Lab 12	76.76	33.52	74.80	70.25	62.06	57.85
	76.55	31.70	75.12	70.63	62.83	54.38
	76.01	28.75	76.90	70.66	61.55	54.09
Lab 13	75.29	30.08	75.44	69.10	61.40	54.72
	75.51	29.13	75.62	69.48	60.90	55.05
	75.12	29.29	75.92	69.46	60.42	56.10
Lab 14	73.40	28.09	76.09	68.57	61.59	57.40
	73.66	27.84	75.65	68.93	61.87	57.10
	73.93	28.49	75.83	69.76	61.85	60.66
Lab 15	75.23	34.76	76.14	70.71	62.28	55.33
	72.05	34.37	77.72	66.92	61.11	54.80
	71.50	36.35	75.94	67.56	60.60	55.24

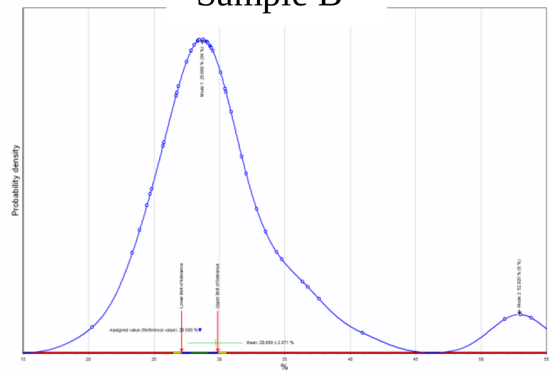
Table B	Sample code					
Lab code	**A**	**B**	**C**	**D**	**E**	**F**
Lab 1	75.23	25.49	77.34	69.89	54.22 (h**,G*)	57.28
Lab 2	70.63	27.18	73.61	69.11	61.54	55.30
Lab 3	73.37	28.91	74.31	68.68	65.06	57.27
Lab 4	75.54	22.96	73.80	67.60	59.01	55.89
Lab 5	72.82	27.28	75.19	69.32	61.19	56.23
Lab 6	72.91	30.45	75.40	69.73	62.19	57.29
Lab 7	71.92	30.37	76.88	71.12	61.65	54.76
Lab 8	77.05	27.72	76.94	71.20	64.45	60.21 (h**)
Lab 9	62.03 (h**)	52.88 (h**,G**)	73.78	63.28 (h**,G**)	62.44	53.65
Lab 10	72.00	28.49	76.12	67.37	60.38	54.91
Lab 11	74.03	38.43	71.17 (h**)	68.78	60.03	57.13
Lab 12	76.44	31.32	75.61	70.51	62.15	55.44
Lab 13	75.31	29.50	75.66	69.35	60.91	55.29
Lab 14	73.66	28.14	75.86	69.09	61.77	58.39
Lab 15	72.93	35.16	76.60	68.40	61.33	55.12

Table C	Sample code					
Lab code	**A**	**B**	**C**	**D**	**E**	**F**
Lab 1	2.36	2.56	0.90	1.00	0.77	0.30
Lab 2	0.96	1.70	0.46	1.52	3.02 (k**,C**)	2.49
Lab 3	1.22	1.41	1.04	0.22	0.62	0.46
Lab 4	2.10	2.36	0.20	0.65	1.41	2.60
Lab 5	0.54	0.35	0.10	0.73	0.64	0.10
Lab 6	0.80	2.08	0.61	0.09	0.37	0.26
Lab 7	2.25	0.88	1.40 (k*)	0.61	1.33	1.96
Lab 8	2.82	3.90 (k*)	0.81	0.28	1.58	2.63
Lab 9	6.65 (k**,C**)	1.02	0.80	2.44 (k**,C*)	0.66	3.60 (k**)
Lab 10	0.54	2.49	0.56	0.58	0.30	0.42
Lab 11	0.17	2.21	0.41	0.47	0.58	0.44
Lab 12	0.39	2.41	1.13	0.23	0.64	2.09
Lab 13	0.20	0.51	0.24	0.21	0.49	0.72
Lab 14	0.27	0.33	0.22	0.61	0.16	1.97
Lab 15	2.01	1.05	0.98	2.03 (k*,C*)	0.86	0.28

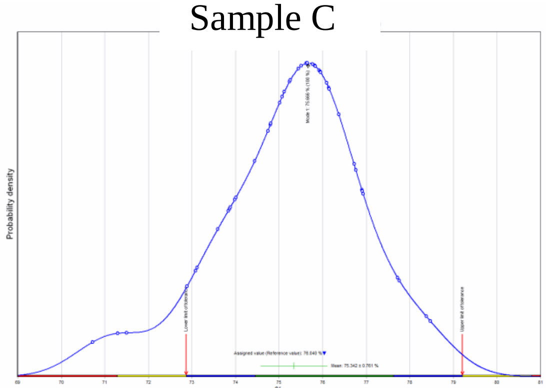
Sample A



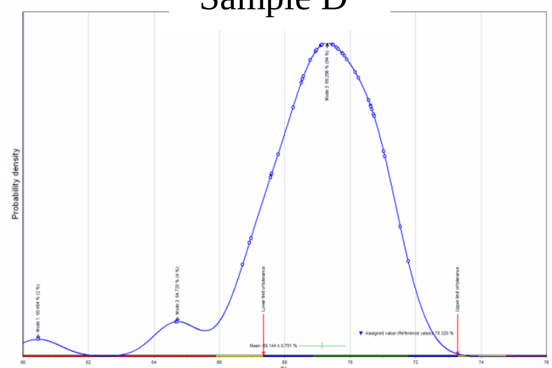
Sample B



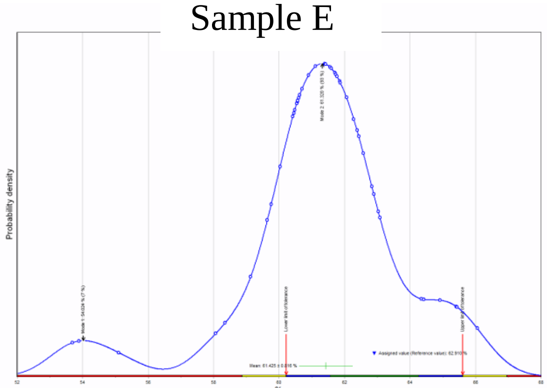
Sample C



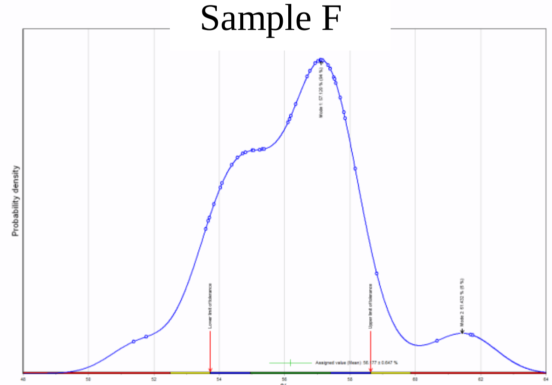
Sample D

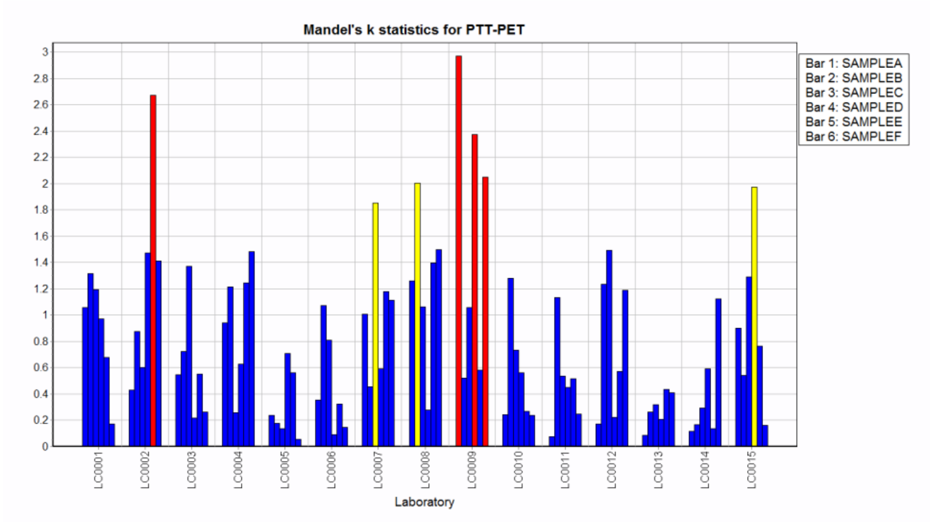


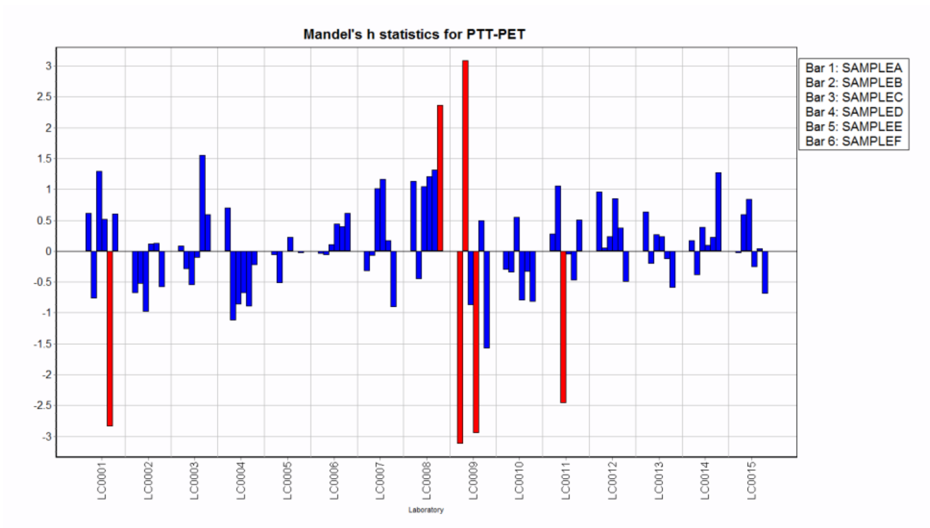
Sample E

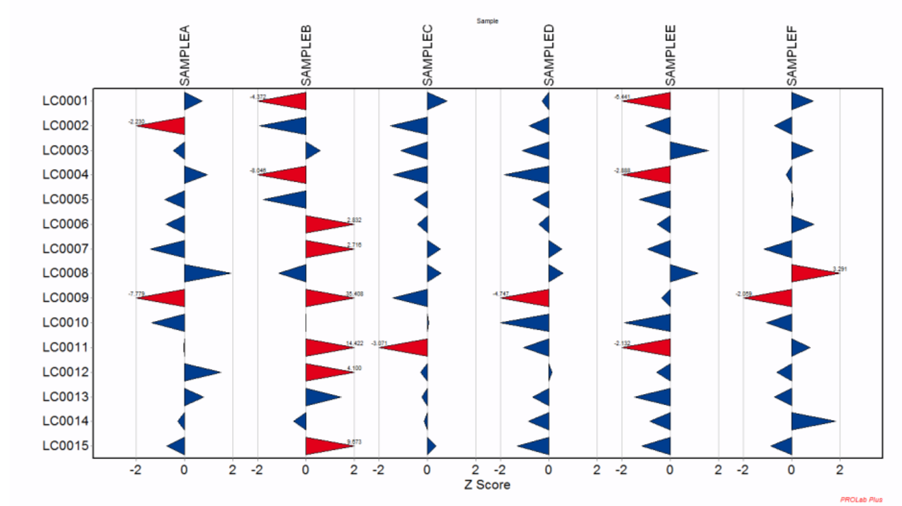


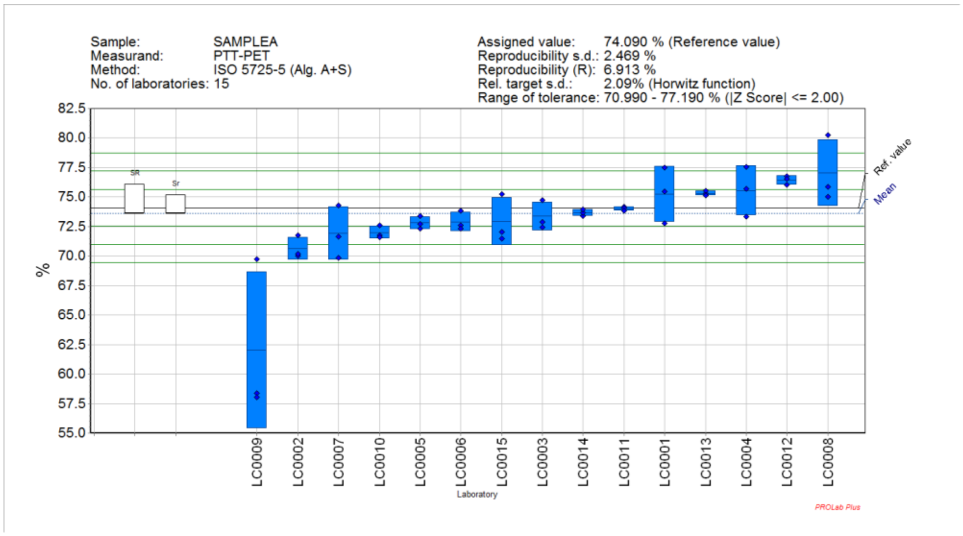
Sample F

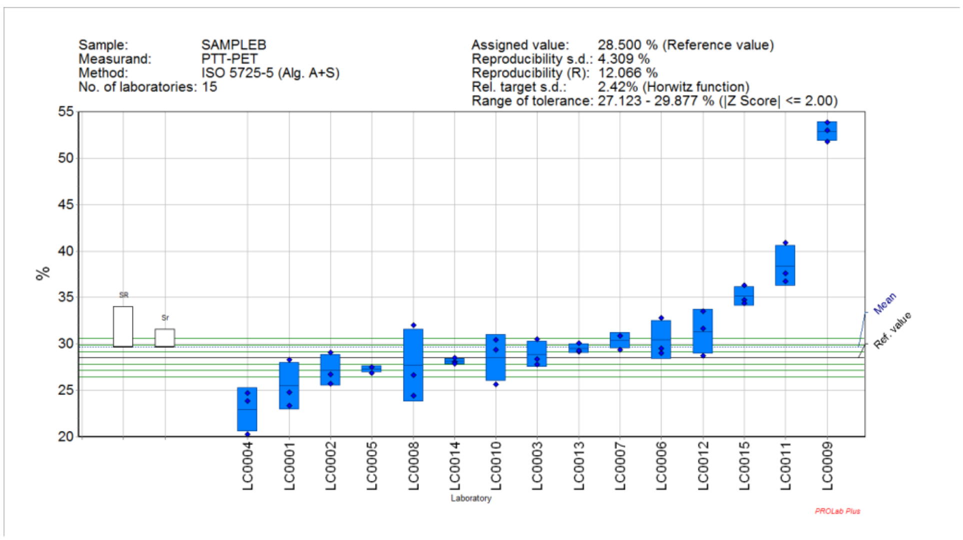


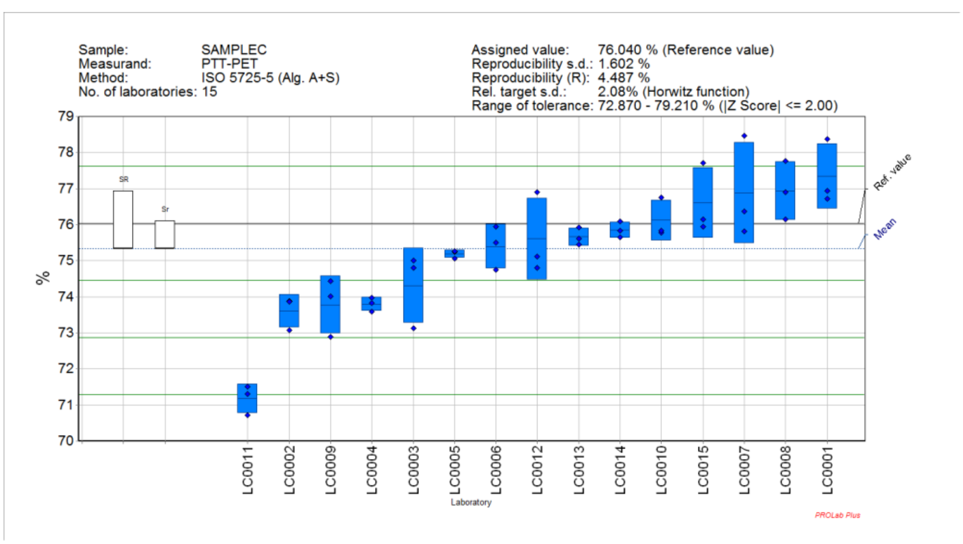

Mandel's k statistics for PTT-PET
Bar 1: SAMPLEA
Bar 2: SAMPLEB
Bar 3: SAMPLEC
Bar 4: SAMPLED
Bar 5: SAMPLEE
Bar 6: SAMPLEF
Laboratory

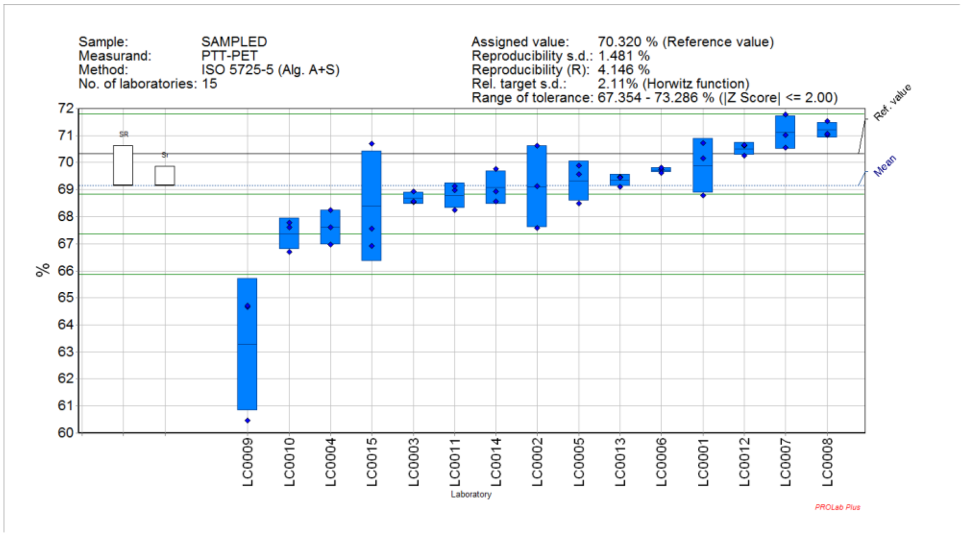

Mandel's h statistics for PTT-PET
Bar 1: SAMPLEA
Bar 2: SAMPLEB
Bar 3: SAMPLEC
Bar 4: SAMPLED
Bar 5: SAMPLEE
Bar 6: SAMPLEF
Laboratory


Sample
SAMPLEA
SAMPLEB
SAMPLEC
SAMPLED
SAMPLEE
SAMPLEF
Z Score
PROLab Plus

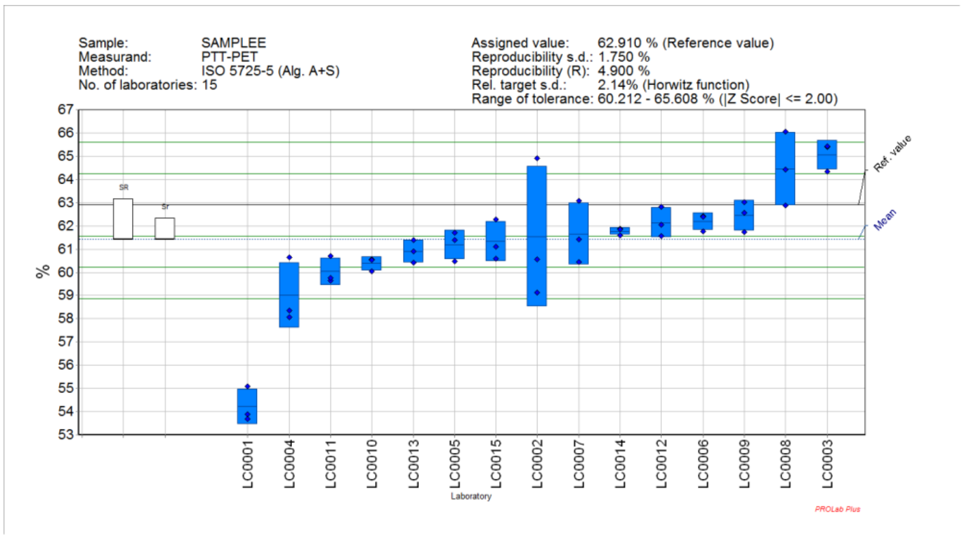

Sample: SAMPLEA
Measurand: PTT-PET
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 74.090 % (Reference value)
Reproducibility s.d.: 2.469 %
Reproducibility (R): 6.913 %
Rel. target s.d.: 2.09% (Horwitz function)
Range of tolerance: 70.990 - 77.190 % (|Z Score| <= 2.00)
%
Laboratory
LC0009 LC0002 LC0007 LC0010 LC0005 LC0006 LC0015 LC0003 LC0014 LC0011 LC0001 LC0013 LC0004 LC0012 LC0008
Ref. value
Mean
PROLab Plus


Sample: SAMPLEB
Measurand: PTT-PET
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 28.500 % (Reference value)
Reproducibility s.d.: 4.309 %
Reproducibility (R): 12.066 %
Rel. target s.d.: 2.42% (Horwitz function)
Range of tolerance: 27.123 - 29.877 % (|Z Score| <= 2.00)
%
Laboratory
LC0004 LC0001 LC0002 LC0005 LC0008 LC0014 LC0010 LC0003 LC0013 LC0007 LC0006 LC0012 LC0015 LC0011 LC0009
Mean
Ref. value
PROLab Plus

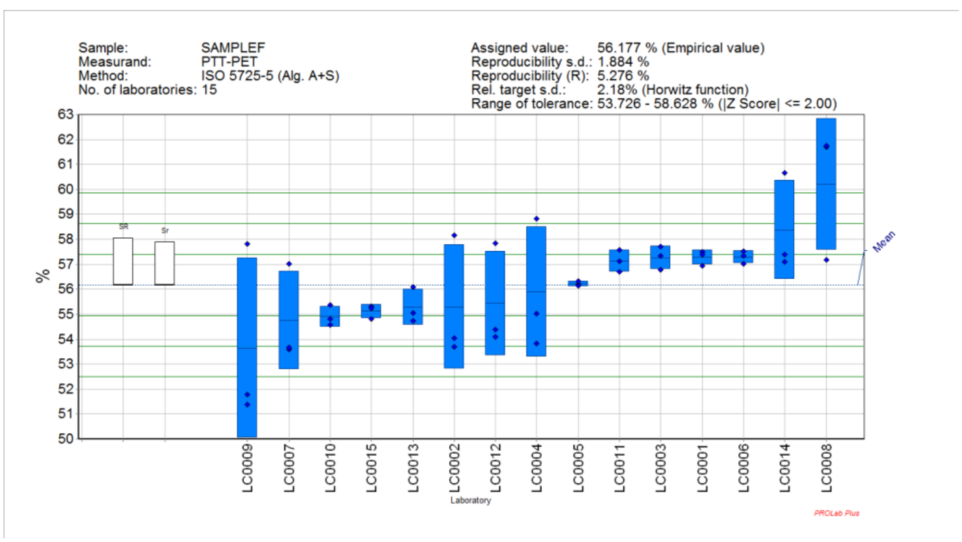

Sample: SAMPLEC
Measurand: PTT-PET
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 76.040 % (Reference value)
Reproducibility s.d.: 1.602 %
Reproducibility (R): 4.487 %
Rel. target s.d.: 2.08% (Horwitz function)
Range of tolerance: 72.870 - 79.210 % (|Z Score| <= 2.00)
%
Laboratory
LC0011 LC0002 LC0009 LC0004 LC0003 LC0005 LC0006 LC0012 LC0013 LC0014 LC0010 LC0015 LC0007 LC0008 LC0001
Ref. value
Mean
PROLab Plus



Sample: SAMPLED
Measurand: PTT-PET
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 70.320 % (Reference value)
Reproducibility s.d.: 1.481 %
Reproducibility (R): 4.146 %
Rel. target s.d.: 2.11% (Horwitz function)
Range of tolerance: 67.354 - 73.286 % (|Z Score| <= 2.00)
%
SR
Sr
Ref. value
Mean
LC0009 LC0010 LC0004 LC0015 LC0003 LC0011 LC0014 LC0002 LC0005 LC0013 LC0006 LC0001 LC0012 LC0007 LC0008
Laboratory
PROLab Plus



Sample: SAMPLEE
Measurand: PTT-PET
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 62.910 % (Reference value)
Reproducibility s.d.: 1.750 %
Reproducibility (R): 4.900 %
Rel. target s.d.: 2.14% (Horwitz function)
Range of tolerance: 60.212 - 65.608 % (|Z Score| <= 2.00)
%
SR
Sr
Ref. value
Mean
LC0001 LC0004 LC0011 LC0010 LC0013 LC0005 LC0015 LC0002 LC0007 LC0014 LC0012 LC0006 LC0009 LC0008 LC0003
Laboratory
PROLab Plus



Sample: SAMPLEF
Measurand: PTT-PET
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 56.177 % (Empirical value)
Reproducibility s.d.: 1.884 %
Reproducibility (R): 5.276 %
Rel. target s.d.: 2.18% (Horwitz function)
Range of tolerance: 53.726 - 58.628 % (|Z Score| <= 2.00)
%
SR
Sr
Mean
LC0009 LC0007 LC0010 LC0015 LC0013 LC0002 LC0012 LC0004 LC0005 LC0011 LC0003 LC0001 LC0006 LC0014 LC0008
Laboratory
PROLab Plus

PTT quantification *via* single point calibration based on PTT peak

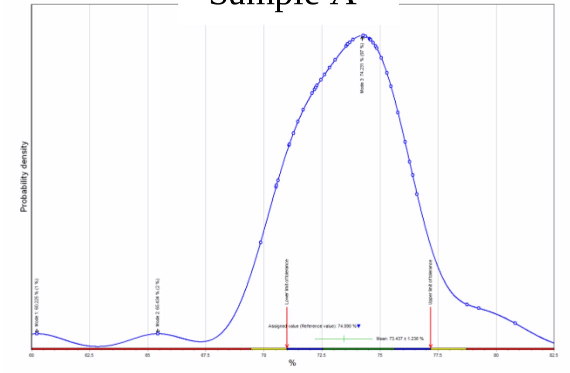
Table A, B and C: original values, cell means and cell standard deviation, respectively.

Table A	Sample code					
Lab code	**A**	**B**	**C**	**D**	**E**	**F**
Lab 1	71.47	28.41	77.52	70.78	62.68	57.78
	72.18	29.90	77.06	70.52	64.64	56.78
	72.24	30.63	77.20	71.35	62.93	56.82
Lab 2	76.60	30.84	80.26	79.26	65.14	63.08
	80.82	30.87	78.72	76.98	61.98	63.65
	73.70	31.04	78.36	73.24	65.52	58.24
Lab 3	73.62	28.72	75.76	76.75	61.84	55.84
	73.60	28.57	74.38	75.87	60.25	56.70
	74.59	29.28	79.55	77.79	62.58	54.24
Lab 4	70.53	29.49	77.09	78.14	64.39	57.92
	78.76	29.12	76.74	81.79	67.70	62.37
	73.07	29.77	76.90	79.97	65.77	61.54
Lab 5	74.86	28.92	75.84	71.61	62.90	58.55
	75.30	29.28	78.10	70.55	63.41	58.61
	74.81	28.84	77.84	69.67	63.09	59.12
Lab 6	72.08	28.65	75.69	70.58	65.00	59.47
	72.29	28.48	77.72	69.93	63.38	59.05
	73.54	29.23	75.81	70.98	64.13	56.69
Lab 7	72.61	29.07	77.39	70.67	73.16	59.42
	71.27	28.83	75.69	70.07	69.15	58.97
	71.10	29.46	77.52	68.11	70.97	57.34
Lab 8	60.23	27.79	68.61	67.97	60.44	55.87
	75.06	30.12	68.20	70.58	62.37	65.76
	75.78	27.48	66.18	73.54	70.81	56.22
Lab 9	75.48	40.40	75.32	74.47	68.19	65.57
	72.83	38.83	76.64	74.25	65.54	61.77
	65.43	38.39	75.52	74.87	69.75	65.17
Lab 10	70.55	29.29	75.81	75.08	61.14	58.78
	69.86	30.60	76.45	75.34	60.18	58.26
	70.61	29.04	74.78	77.91	61.89	58.95
Lab 11	76.08	31.83	80.65	71.11	64.12	59.83
	76.28	35.51	74.27	71.62	62.54	60.04
	75.78	34.33	78.10	72.34	66.21	56.49
Lab 12	74.61	28.25	72.94	69.55	65.28	60.89
	74.27	28.69	73.80	69.23	65.69	64.66
	74.71	27.74	72.55	67.83	65.53	62.99
Lab 13	74.37	29.47	76.80	71.27	63.07	58.67
	74.37	28.07	76.18	69.92	62.66	58.72
	74.56	28.13	76.48	69.06	63.53	57.80
Lab 14	71.09	28.98	76.04	71.78	63.32	57.81
	72.46	28.88	75.75	71.34	64.76	59.06
	71.70	28.01	77.29	72.46	63.34	54.99
Lab 15	73.83	27.37	73.57	68.00	63.19	55.98
	76.41	26.55	73.89	72.37	61.09	60.27
	79.26	27.40	74.49	71.47	60.87	57.79

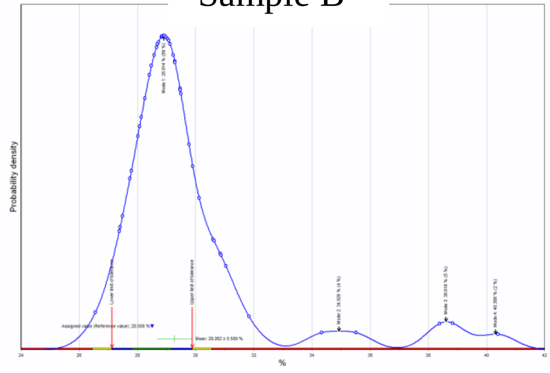
Table B	Sample code					
Lab code	**A**	**B**	**C**	**D**	**E**	**F**
Lab 1	71.96	29.65	77.26	70.88	63.42	57.13
Lab 2	77.04	30.92	79.11	76.49	64.21	61.65
Lab 3	73.93	28.86	76.56	76.80	61.56	55.59
Lab 4	74.12	29.46	76.91	79.97 (h*)	65.95	60.61
Lab 5	74.99	29.02	77.26	70.61	63.13	58.76
Lab 6	72.63	28.79	76.41	70.50	64.17	58.40
Lab 7	71.66	29.12	76.87	69.62	71.09 (h**,G*)	58.58
Lab 8	70.36	28.46	67.66 (h**,G**)	70.70	64.54	59.28
Lab 9	71.25	39.21 (h**,G**)	75.83	74.53	67.83	64.17 (h*)
Lab 10	70.34	29.64	75.68	76.11	61.07	58.66
Lab 11	76.05	33.89 (G**)	77.67	71.69	64.29	58.79
Lab 12	74.53	28.23	73.10	68.87	65.50	62.85
Lab 13	74.43	28.55	76.49	70.08	63.09	58.40
Lab 14	71.75	28.63	76.36	71.86	63.81	57.29
Lab 15	76.50	27.10	73.98	70.61	61.72	58.02

Table C	Sample code					
Lab code	**A**	**B**	**C**	**D**	**E**	**F**
Lab 1	0.43	1.13	0.24	0.43	1.06	0.57
Lab 2	3.58	0.11	1.01	3.04 (k*)	1.95	2.98
Lab 3	0.56	0.37	2.68 (k*)	0.96	1.19	1.24
Lab 4	4.21	0.33	0.18	1.83	1.66	2.37
Lab 5	0.27	0.23	1.23	0.97	0.26	0.32
Lab 6	0.79	0.39	1.14	0.53	0.81	1.50
Lab 7	0.82	0.32	1.02	1.33	2.01	1.09
Lab 8	8.78 (k**,C**)	1.45 (k*)	1.30	2.79 (k*)	5.51 (k**,C**)	5.61 (k**,C**)
Lab 9	5.21 (C*)	1.06	0.71	0.32	2.13	2.09
Lab 10	0.42	0.84	0.84	1.57	0.86	0.36
Lab 11	0.25	1.88 (k**)	3.22 (k**,C*)	0.62	1.84	1.99
Lab 12	0.23	0.48	0.64	0.92	0.21	1.89
Lab 13	0.11	0.79	0.31	1.11	0.44	0.52
Lab 14	0.68	0.53	0.82	0.57	0.82	2.09
Lab 15	2.72	0.48	0.47	2.31	1.28	2.15

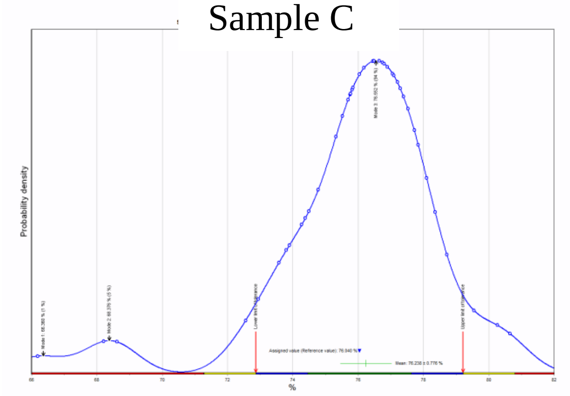
Sample A



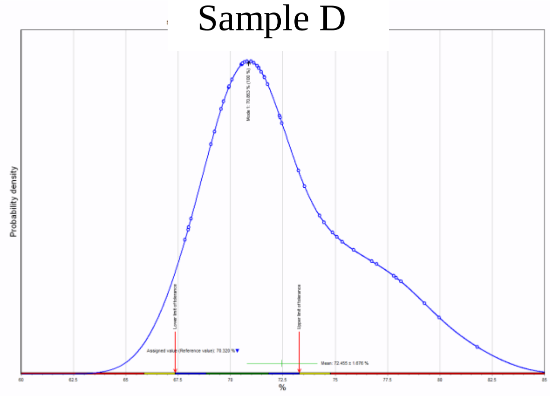
Sample B



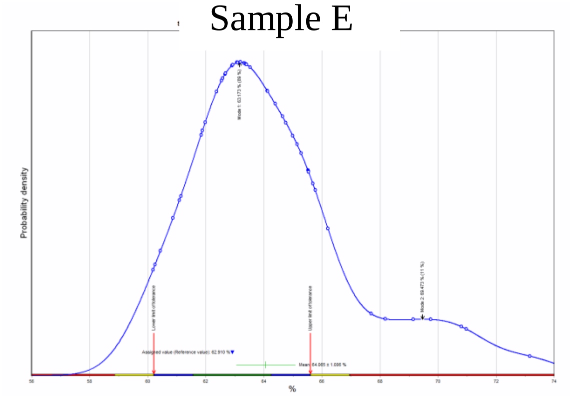
Sample C



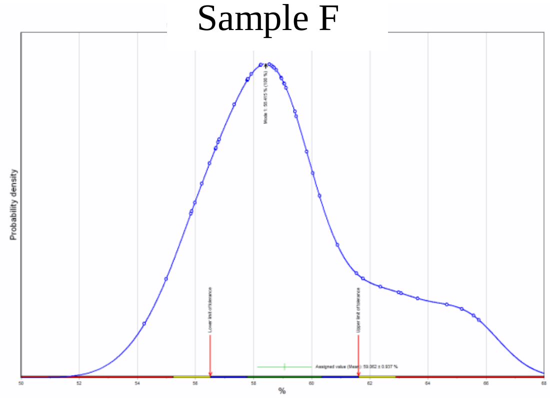
Sample D

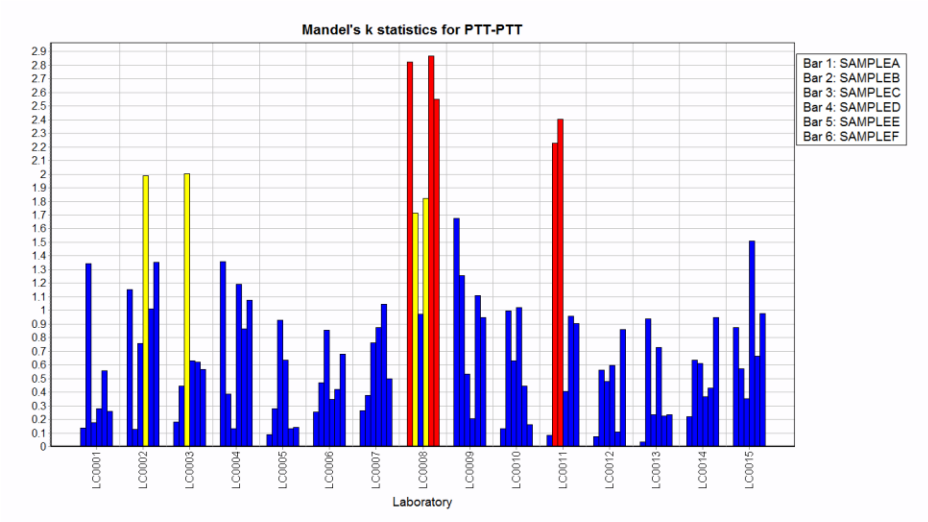


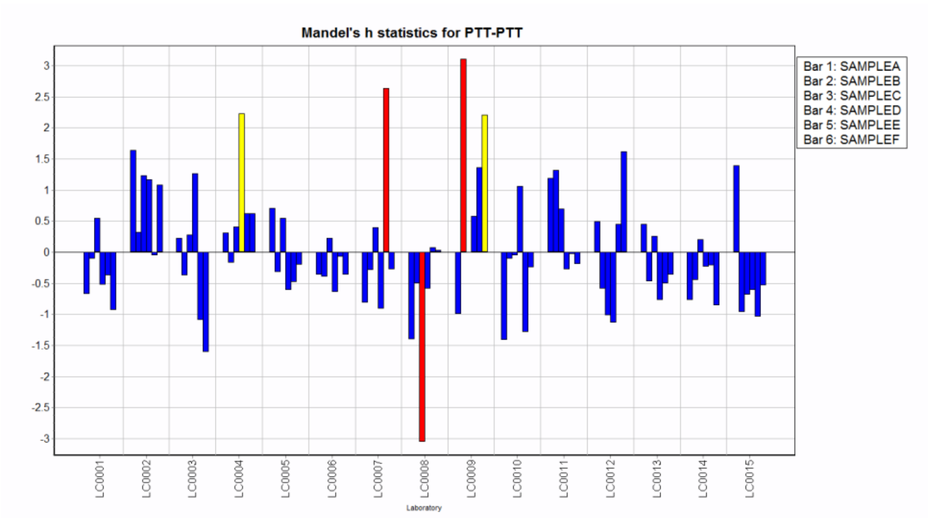
Sample E

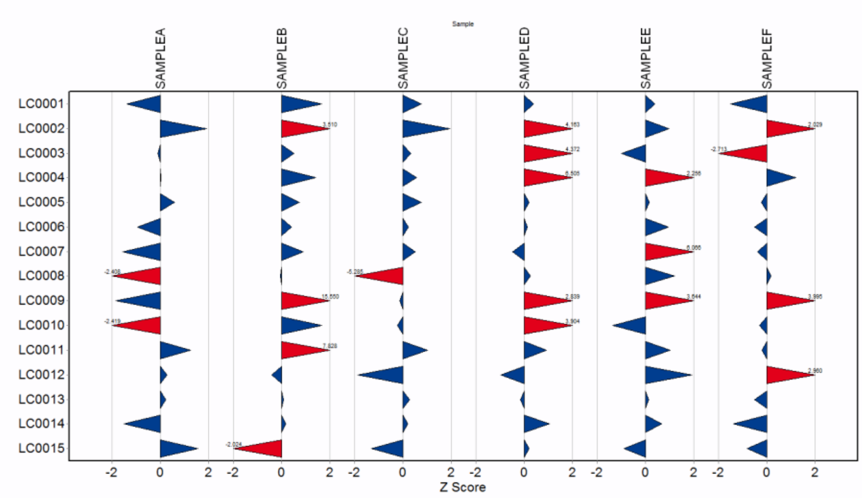


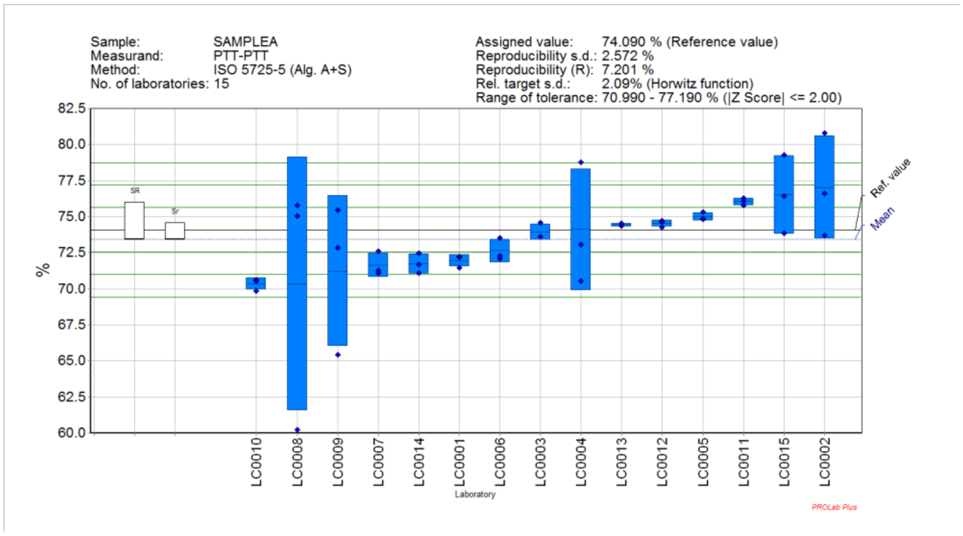
Sample F

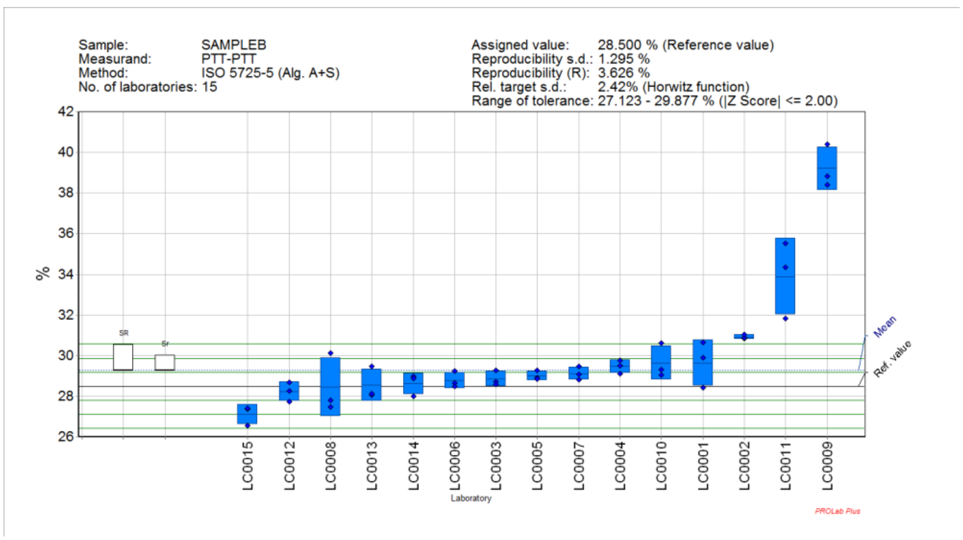


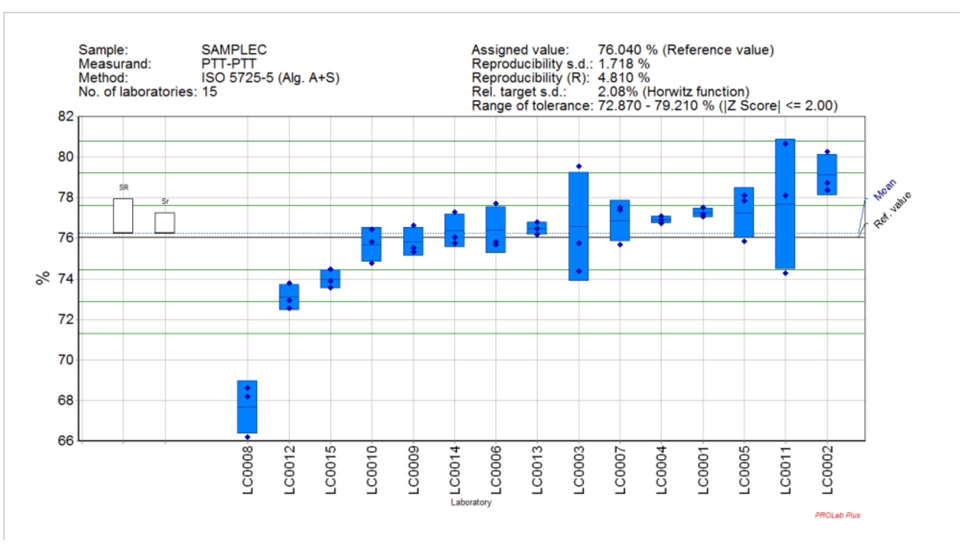

Mandel's k statistics for PTT-PTT
Bar 1: SAMPLEA
Bar 2: SAMPLEB
Bar 3: SAMPLEC
Bar 4: SAMPLED
Bar 5: SAMPLEE
Bar 6: SAMPLEF
LC0001
LC0002
LC0003
LC0004
LC0005
LC0006
LC0007
LC0008
LC0009
LC0010
LC0011
LC0012
LC0013
LC0014
LC0015
Laboratory

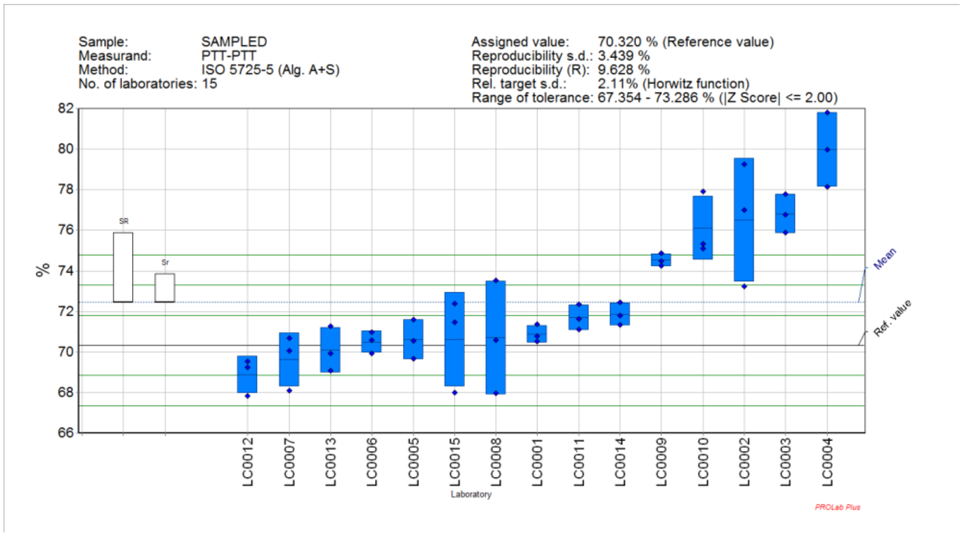

Mandel's h statistics for PTT-PTT
Bar 1: SAMPLEA
Bar 2: SAMPLEB
Bar 3: SAMPLEC
Bar 4: SAMPLED
Bar 5: SAMPLEE
Bar 6: SAMPLEF
LC0001
LC0002
LC0003
LC0004
LC0005
LC0006
LC0007
LC0008
LC0009
LC0010
LC0011
LC0012
LC0013
LC0014
LC0015
Laboratory

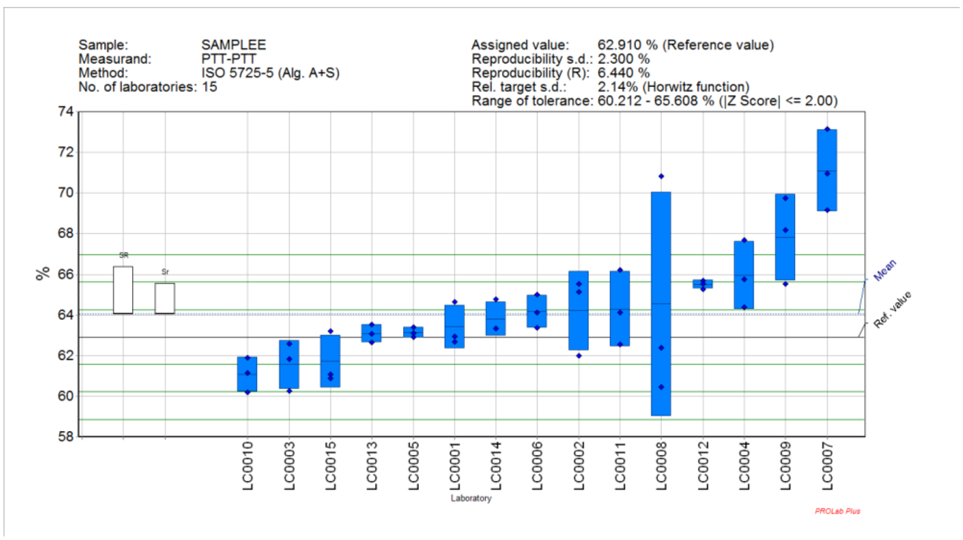

Sample
SAMPLEA
SAMPLEB
SAMPLEC
SAMPLED
SAMPLEE
SAMPLEF
LC0001
LC0002
LC0003
LC0004
LC0005
LC0006
LC0007
LC0008
LC0009
LC0010
LC0011
LC0012
LC0013
LC0014
LC0015
Z Score
PROLab Plus

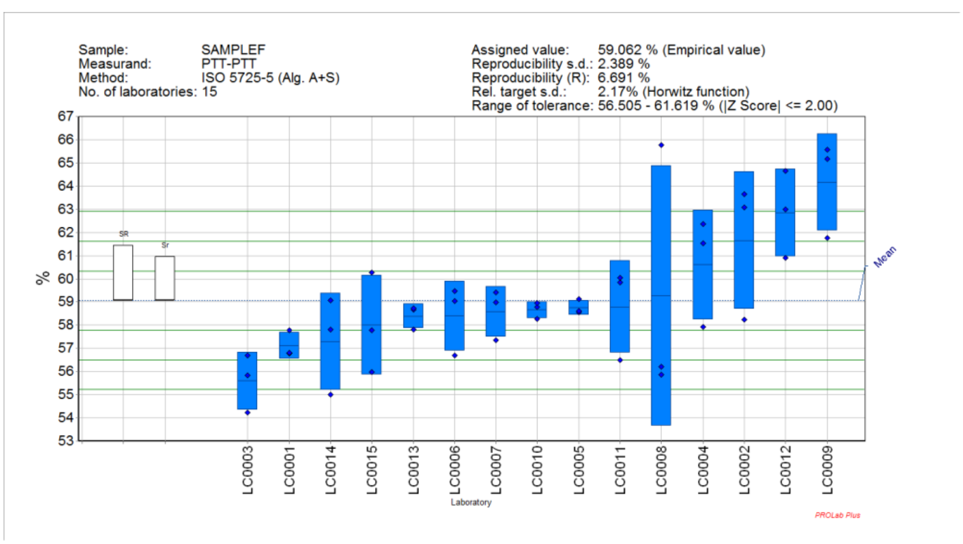

Sample: SAMPLEA
Measurand: PTT-PTT
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 74.090 % (Reference value)
Reproducibility s.d.: 2.572 %
Reproducibility (R): 7.201 %
Rel. target s.d.: 2.09% (Horwitz function)
Range of tolerance: 70.990 - 77.190 % (|Z Score| <= 2.00)
%
Laboratory
LC0010 LC0008 LC0009 LC0007 LC0014 LC0001 LC0006 LC0003 LC0004 LC0013 LC0012 LC0005 LC0011 LC0015 LC0002
Ref. value
Mean
PROLab Plus


Sample: SAMPLEB
Measurand: PTT-PTT
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 28.500 % (Reference value)
Reproducibility s.d.: 1.295 %
Reproducibility (R): 3.626 %
Rel. target s.d.: 2.42% (Horwitz function)
Range of tolerance: 27.123 - 29.877 % (|Z Score| <= 2.00)
%
Laboratory
LC0015 LC0012 LC0008 LC0013 LC0014 LC0006 LC0003 LC0005 LC0007 LC0004 LC0010 LC0001 LC0002 LC0011 LC0009
Mean
Ref. value
PROLab Plus


Sample: SAMPLEC
Measurand: PTT-PTT
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 76.040 % (Reference value)
Reproducibility s.d.: 1.718 %
Reproducibility (R): 4.810 %
Rel. target s.d.: 2.08% (Horwitz function)
Range of tolerance: 72.870 - 79.210 % (|Z Score| <= 2.00)
%
Laboratory
LC0008 LC0012 LC0015 LC0010 LC0009 LC0014 LC0006 LC0013 LC0003 LC0007 LC0004 LC0001 LC0005 LC0011 LC0002
Mean
Ref. value
PROLab Plus


Sample: SAMPLED
Measurand: PTT-PTT
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 70.320 % (Reference value)
Reproducibility s.d.: 3.439 %
Reproducibility (R): 9.628 %
Rel. target s.d.: 2.11% (Horwitz function)
Range of tolerance: 67.354 - 73.286 % (|Z Score| <= 2.00)
%
Laboratory
LC0012
LC0007
LC0013
LC0006
LC0005
LC0015
LC0008
LC0001
LC0011
LC0014
LC0009
LC0010
LC0002
LC0003
LC0004
Mean
Ref. value
PROLab Plus


Sample: SAMPLEE
Measurand: PTT-PTT
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 62.910 % (Reference value)
Reproducibility s.d.: 2.300 %
Reproducibility (R): 6.440 %
Rel. target s.d.: 2.14% (Horwitz function)
Range of tolerance: 60.212 - 65.608 % (|Z Score| <= 2.00)
%
Laboratory
LC0010
LC0003
LC0015
LC0013
LC0005
LC0001
LC0014
LC0006
LC0002
LC0011
LC0008
LC0012
LC0004
LC0009
LC0007
Mean
Ref. value
PROLab Plus


Sample: SAMPLEF
Measurand: PTT-PTT
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 59.062 % (Empirical value)
Reproducibility s.d.: 2.389 %
Reproducibility (R): 6.691 %
Rel. target s.d.: 2.17% (Horwitz function)
Range of tolerance: 56.505 - 61.619 % (|Z Score| <= 2.00)
%
Laboratory
LC0003
LC0001
LC0014
LC0015
LC0013
LC0006
LC0007
LC0010
LC0005
LC0011
LC0008
LC0004
LC0002
LC0012
LC0009
Mean
PROLab Plus

PTT quantification *via* single point calibration based on PET peak

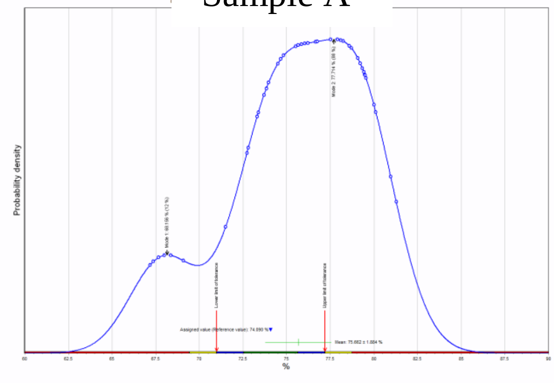
Table A, B and C: original values, cell means and cell standard deviation, respectively.

Table A	Sample code					
Lab code	**A**	**B**	**C**	**D**	**E**	**F**
Lab 1	68.37	25.09	76.59	70.33	63.38	58.00
	73.84	29.96	76.37	69.74	64.55	57.88
	71.51	26.54	78.06	68.35	63.18	57.43
Lab 2	67.17	27.85	73.63	67.12	63.81	53.44
	67.37	24.48	74.42	68.68	59.32	53.10
	69.08	25.48	74.40	70.19	57.84	57.62
Lab 3	79.54	28.53	75.16	68.98	65.30	61.08
	79.22	29.10	74.96	69.37	64.23	61.57
	80.96	31.19	73.28	69.01	65.32	61.90
Lab 4	77.52	20.32	74.65	68.70	59.83	60.43
	73.31	24.75	74.27	67.43	57.51	55.65
	75.66	23.94	74.51	68.07	57.21	56.78
Lab 5	72.73	28.79	75.48	69.65	61.53	56.30
	73.38	28.20	75.50	69.96	60.61	56.26
	72.81	28.60	75.43	69.40	61.32	56.45
Lab 6	79.42	29.98	74.86	70.17	61.05	62.62
	78.25	33.31	75.62	70.00	61.67	62.80
	78.71	30.93	75.51	70.09	61.48	62.35
Lab 7	79.07	30.64	77.00	71.61	70.51	61.11
	81.28	30.64	79.04	71.17	68.99	61.19
	77.53	29.10	76.46	72.35	68.10	64.47
Lab 8	80.00	27.40	77.03	74.13	64.30	65.12
	75.53	34.69	77.88	73.70	65.76	60.99
	74.67	30.54	77.06	73.84	65.80	65.17
Lab 9	67.99	52.33	74.89	65.50	63.08	55.89
	67.68	51.49	75.30	69.47	63.53	62.11
	77.91	50.23	73.81	69.51	62.24	56.27
Lab 10	79.48	31.18	76.12	67.06	61.37	61.51
	79.34	26.15	76.18	66.86	61.89	61.31
	80.11	30.05	77.10	65.95	61.86	62.00
Lab 11	74.83	41.38	71.53	68.60	60.28	57.91
	74.67	38.08	71.34	69.31	59.21	57.03
	74.49	37.23	70.76	69.46	59.34	57.46
Lab 12	76.76	31.54	75.39	70.85	61.95	57.74
	76.23	29.66	75.70	70.89	60.63	54.27
	76.65	26.63	77.44	70.74	61.25	53.97
Lab 13	76.04	29.16	75.69	69.10	60.33	55.13
	75.66	29.32	75.99	69.08	59.85	55.46
	75.84	29.53	75.73	68.96	60.34	56.50
Lab 14	73.70	28.40	75.53	69.29	62.46	63.12
	73.97	29.04	75.71	70.11	62.44	62.87
	73.70	28.69	75.74	69.45	62.36	65.95
Lab 15	78.60	34.31	77.07	66.19	60.77	55.67
	78.11	36.29	75.24	66.84	60.25	55.14
	79.49	35.10	75.92	67.69	60.99	55.58

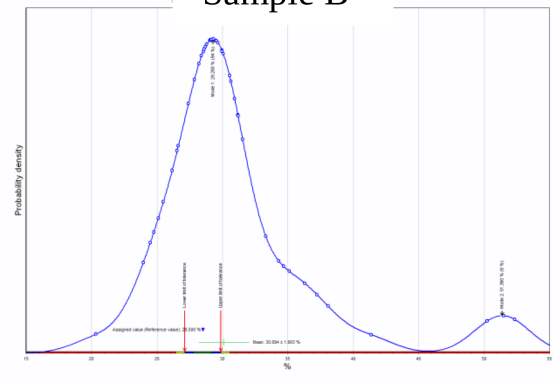
Table B	Sample code					
Lab code	**A**	**B**	**C**	**D**	**E**	**F**
Lab 1	71.24 (h*)	27.20	77.01	69.48	63.70	57.77
Lab 2	67.87	25.94	74.15	68.66	60.32	54.72
Lab 3	79.91	29.60	74.47	69.12	64.95	61.51
Lab 4	75.50	23.00	74.48	68.07	58.19	57.62
Lab 5	72.97	28.53	75.47	69.67	61.15	56.34
Lab 6	78.79	31.40	75.33	70.09	61.40	62.59
Lab 7	79.29	30.12	77.50	71.71	69.20 (h**)	62.26
Lab 8	76.73	30.87	77.32	73.89 (h**)	65.29	63.76
Lab 9	71.20	51.35 (h**,G**)	74.67	68.16	62.95	58.09
Lab 10	79.64	29.13	76.47	66.62	61.71	61.61
Lab 11	74.67	38.90	71.21 (h**,G**)	69.12	59.61	57.46
Lab 12	76.55	29.28	76.17	70.83	61.28	55.33
Lab 13	75.85	29.34	75.80	69.05	60.17	55.70
Lab 14	73.79	28.71	75.66	69.62	62.42	63.98
Lab 15	78.73	35.23	76.07	66.91	60.67	55.46

Table C	Sample code					
Lab code	**A**	**B**	**C**	**D**	**E**	**F**
Lab 1	2.74	2.50	0.92	1.01	0.74	0.30
Lab 2	1.05	1.73	0.45	1.54 (k*,C*)	3.11 (k**,C**)	2.52
Lab 3	0.92	1.40	1.03	0.22	0.63	0.42
Lab 4	2.11	2.36	0.19	0.63	1.44	2.50
Lab 5	0.36	0.30	0.04	0.28	0.48	0.10
Lab 6	0.59	1.71	0.41	0.09	0.32	0.23
Lab 7	1.89	0.89	1.36 (k*)	0.60	1.22	1.92
Lab 8	2.86	3.66 (k*)	0.48	0.22	0.86	2.40
Lab 9	5.82 (k**,C**)	1.05	0.77	2.30 (k**,C**)	0.65	3.49 (k**)
Lab 10	0.41	2.64	0.55	0.59	0.29	0.36
Lab 11	0.17	2.19	0.40	0.46	0.58	0.44
Lab 12	0.28	2.48	1.11	0.08	0.66	2.10
Lab 13	0.19	0.19	0.16	0.07	0.28	0.71
Lab 14	0.16	0.32	0.11	0.43	0.05	1.71
Lab 15	0.70	1.00	0.93	0.76	0.38	0.28

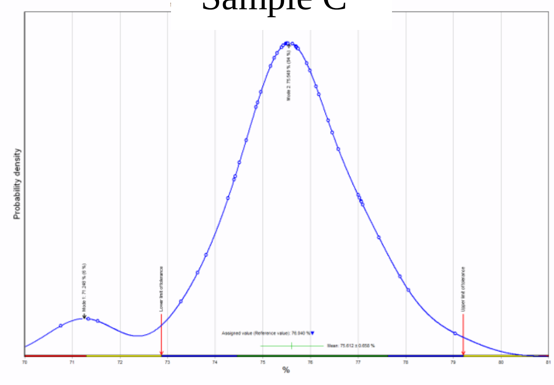
Sample A



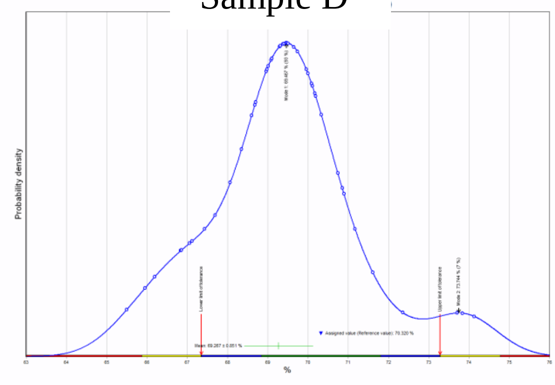
Sample B



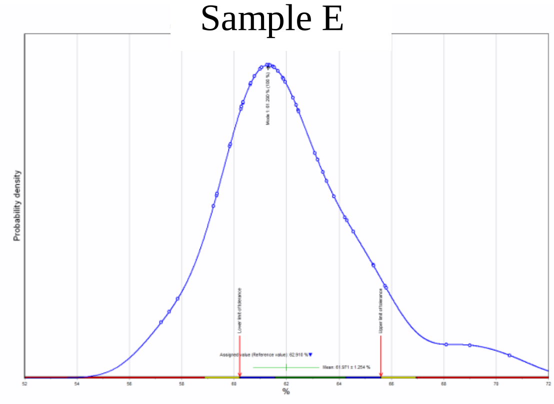
Sample C



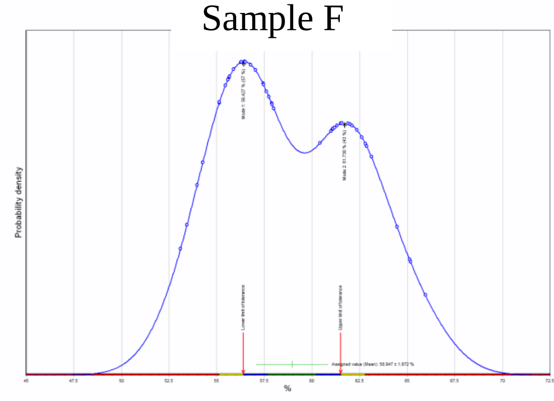
Sample D

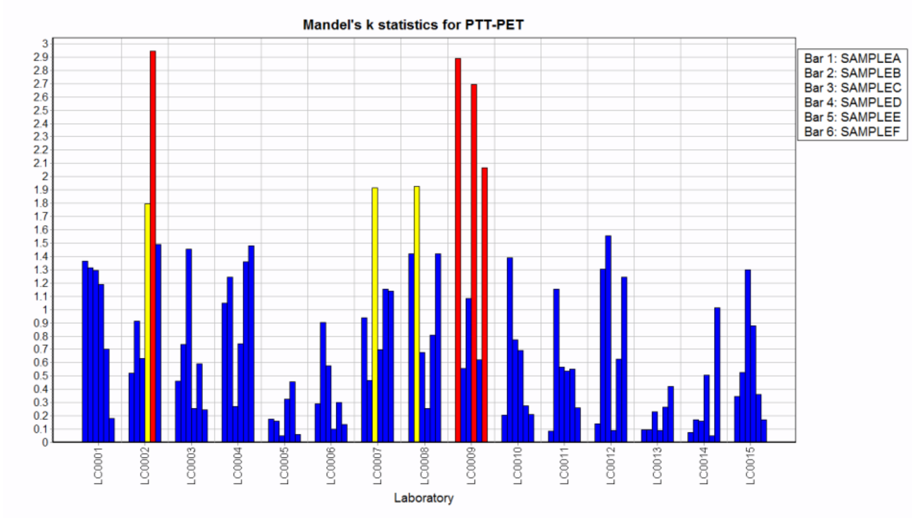


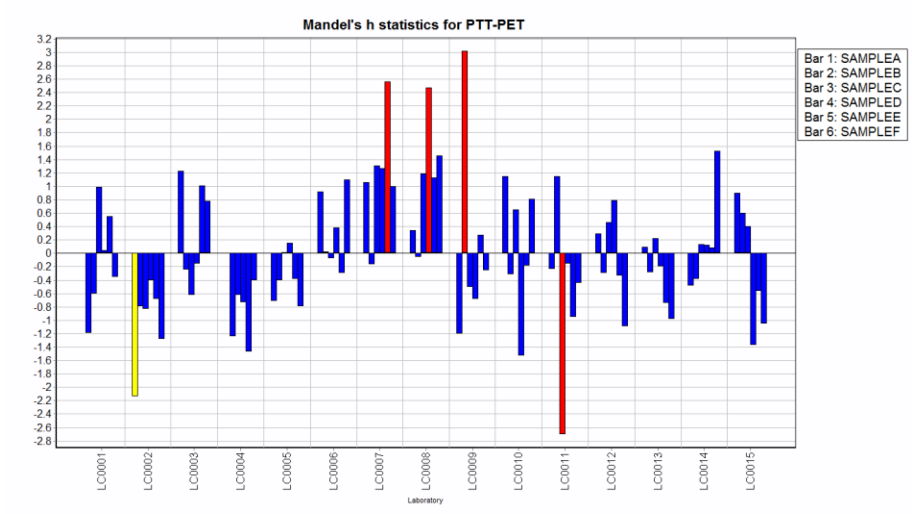
Sample E

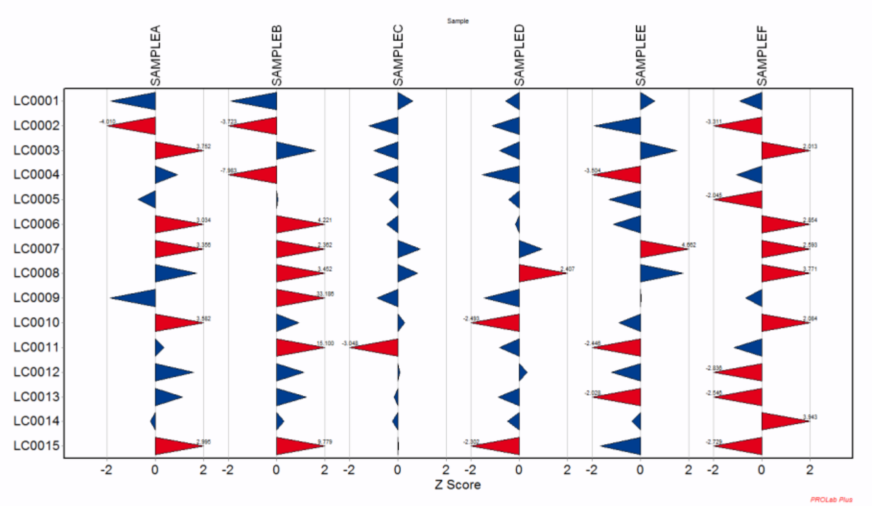


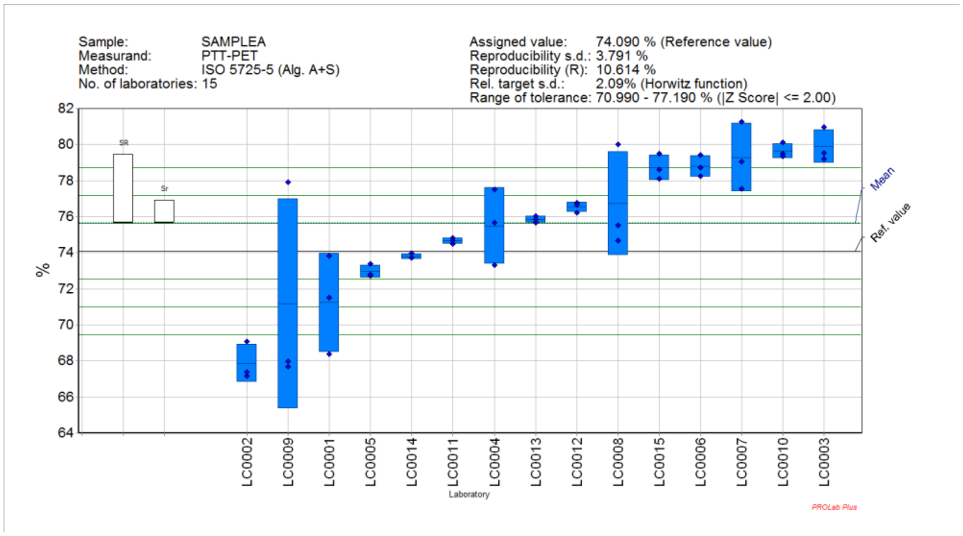
Sample F

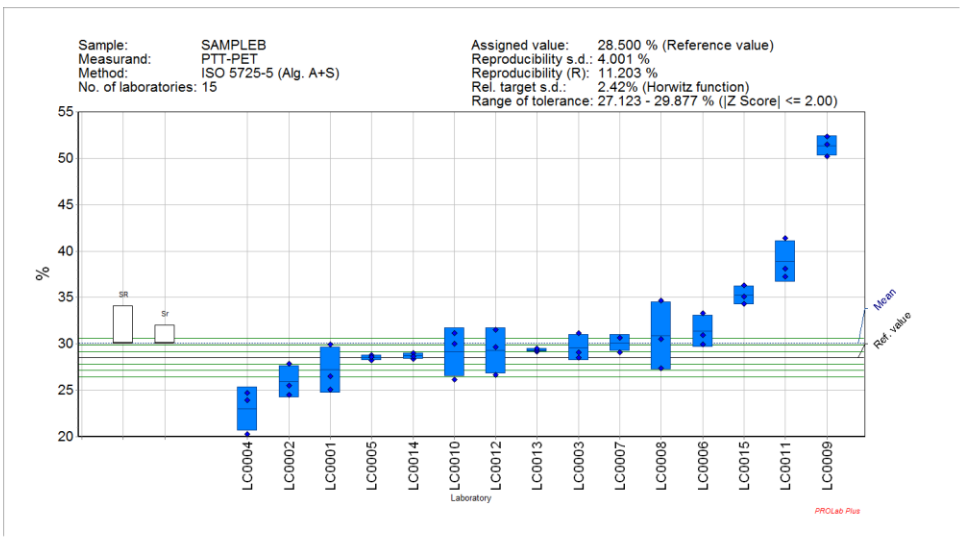


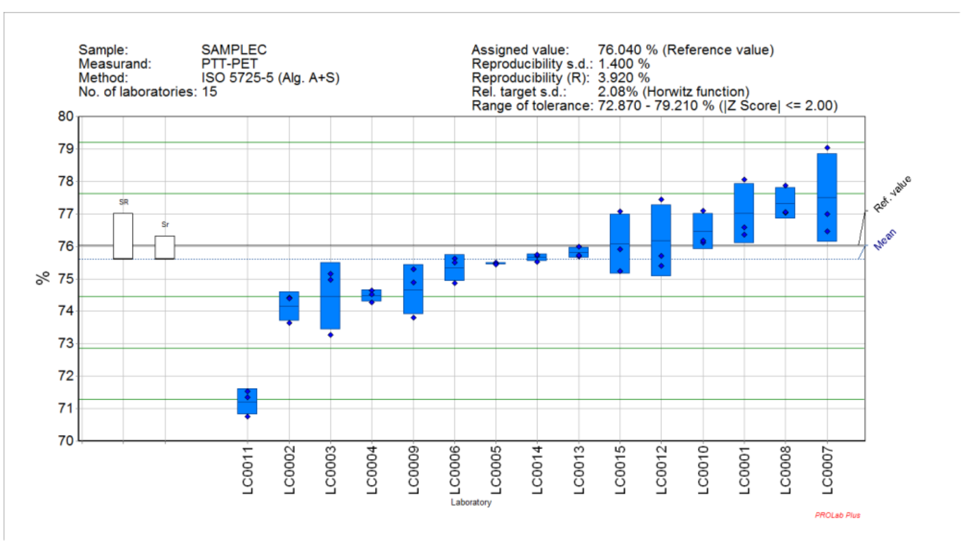

Mandel's k statistics for PTT-PET
Bar 1: SAMPLEA
Bar 2: SAMPLEB
Bar 3: SAMPLEC
Bar 4: SAMPLED
Bar 5: SAMPLEE
Bar 6: SAMPLEF
LC0001
LC0002
LC0003
LC0004
LC0005
LC0006
LC0007
LC0008
LC0009
LC0010
LC0011
LC0012
LC0013
LC0014
LC0015
Laboratory

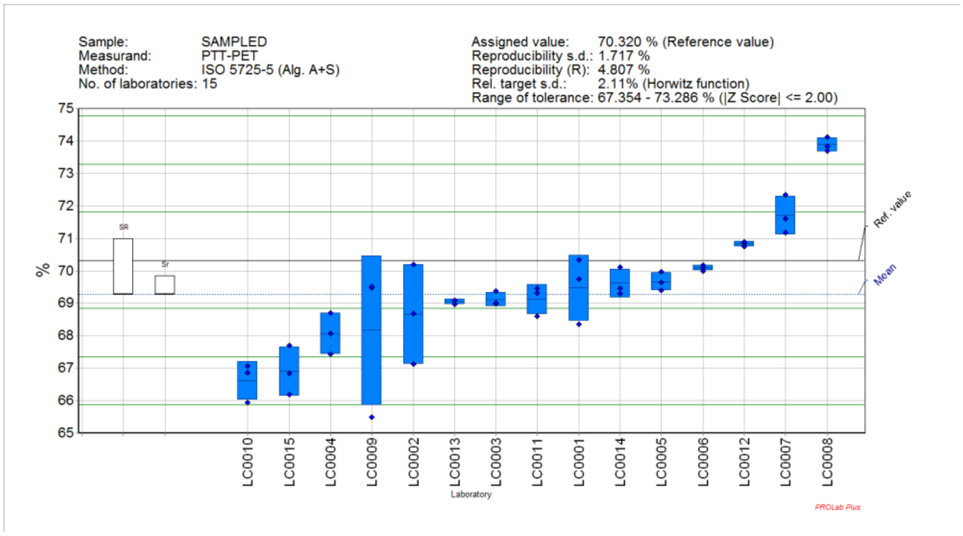

Mandel's h statistics for PTT-PET
Bar 1: SAMPLEA
Bar 2: SAMPLEB
Bar 3: SAMPLEC
Bar 4: SAMPLED
Bar 5: SAMPLEE
Bar 6: SAMPLEF
LC0001
LC0002
LC0003
LC0004
LC0005
LC0006
LC0007
LC0008
LC0009
LC0010
LC0011
LC0012
LC0013
LC0014
LC0015
Laboratory

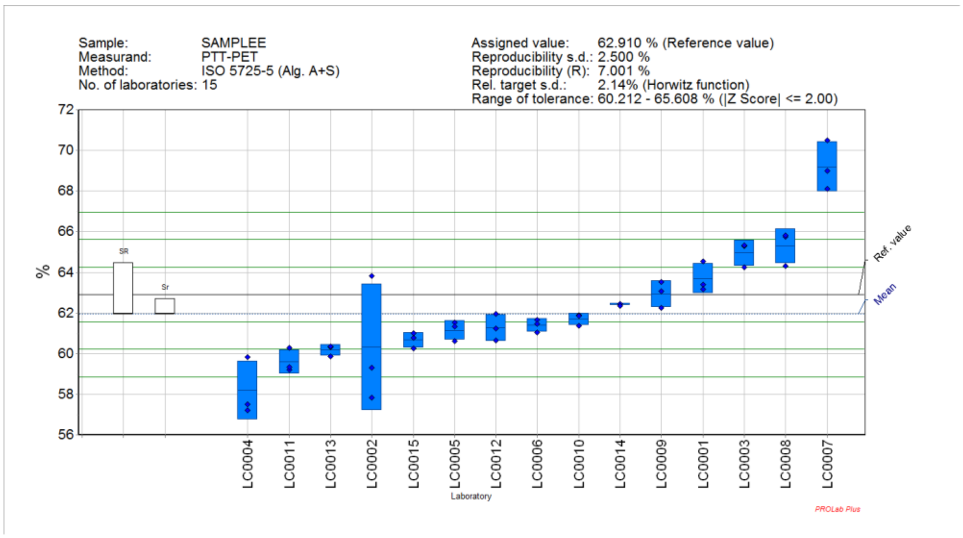

Sample
SAMPLEA
SAMPLEB
SAMPLEC
SAMPLED
SAMPLEE
SAMPLEF
LC0001
LC0002
LC0003
LC0004
LC0005
LC0006
LC0007
LC0008
LC0009
LC0010
LC0011
LC0012
LC0013
LC0014
LC0015
Z Score
PROLab Plus

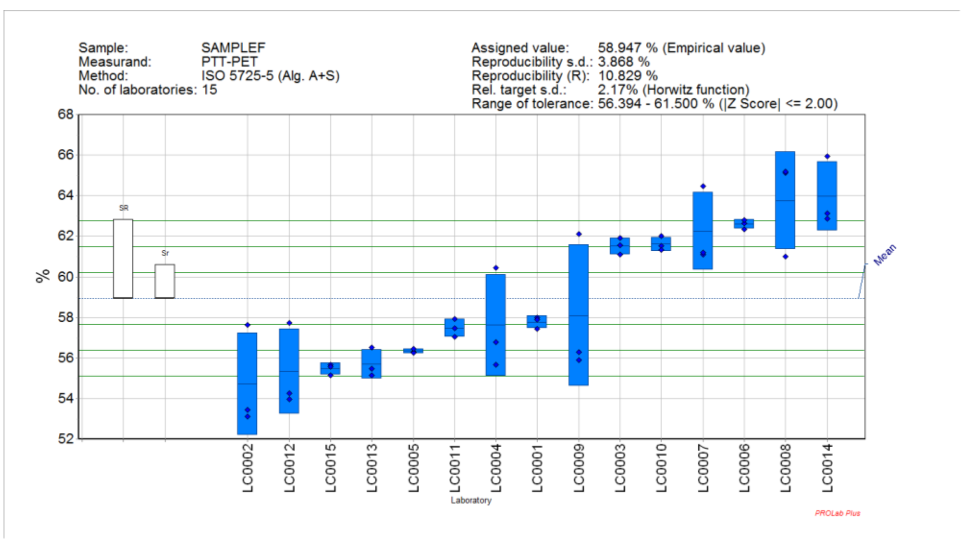

Sample: SAMPLEA
Measurand: PTT-PET
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 74.090 % (Reference value)
Reproducibility s.d.: 3.791 %
Reproducibility (R): 10.614 %
Rel. target s.d.: 2.09% (Horwitz function)
Range of tolerance: 70.990 - 77.190 % (|Z Score| <= 2.00)
%
SR
Sr
Mean
Ref. value
LC0002 LC0009 LC0001 LC0005 LC0014 LC0011 LC0004 LC0013 LC0012 LC0008 LC0015 LC0006 LC0007 LC0010 LC0003
Laboratory
PROLab Plus


Sample: SAMPLEB
Measurand: PTT-PET
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 28.500 % (Reference value)
Reproducibility s.d.: 4.001 %
Reproducibility (R): 11.203 %
Rel. target s.d.: 2.42% (Horwitz function)
Range of tolerance: 27.123 - 29.877 % (|Z Score| <= 2.00)
%
SR
Sr
Mean
Ref. value
LC0004 LC0002 LC0001 LC0005 LC0014 LC0010 LC0012 LC0013 LC0003 LC0007 LC0008 LC0006 LC0015 LC0011 LC0009
Laboratory
PROLab Plus


Sample: SAMPLEC
Measurand: PTT-PET
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 76.040 % (Reference value)
Reproducibility s.d.: 1.400 %
Reproducibility (R): 3.920 %
Rel. target s.d.: 2.08% (Horwitz function)
Range of tolerance: 72.870 - 79.210 % (|Z Score| <= 2.00)
%
SR
Sr
Ref. value
Mean
LC0011 LC0002 LC0003 LC0004 LC0009 LC0006 LC0005 LC0014 LC0013 LC0015 LC0012 LC0010 LC0001 LC0008 LC0007
Laboratory
PROLab Plus


Sample: SAMPLED
Measurand: PTT-PET
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 70.320 % (Reference value)
Reproducibility s.d.: 1.717 %
Reproducibility (R): 4.807 %
Rel. target s.d.: 2.11% (Horwitz function)
Range of tolerance: 67.354 - 73.286 % (|Z Score| <= 2.00)
%
SR
Sr
Ref. value
Mean
LC0010 LC0015 LC0004 LC0009 LC0002 LC0013 LC0003 LC0011 LC0001 LC0014 LC0005 LC0006 LC0012 LC0007 LC0008
Laboratory
PROLab Plus


Sample: SAMPLEE
Measurand: PTT-PET
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 62.910 % (Reference value)
Reproducibility s.d.: 2.500 %
Reproducibility (R): 7.001 %
Rel. target s.d.: 2.14% (Horwitz function)
Range of tolerance: 60.212 - 65.608 % (|Z Score| <= 2.00)
%
SR
Sr
Ref. value
Mean
LC0004 LC0011 LC0013 LC0002 LC0015 LC0005 LC0012 LC0006 LC0010 LC0014 LC0009 LC0001 LC0003 LC0008 LC0007
Laboratory
PROLab Plus


Sample: SAMPLEF
Measurand: PTT-PET
Method: ISO 5725-5 (Alg. A+S)
No. of laboratories: 15
Assigned value: 58.947 % (Empirical value)
Reproducibility s.d.: 3.868 %
Reproducibility (R): 10.829 %
Rel. target s.d.: 2.17% (Horwitz function)
Range of tolerance: 56.394 - 61.500 % (|Z Score| <= 2.00)
%
SR
Sr
Mean
LC0002 LC0012 LC0015 LC0013 LC0005 LC0011 LC0004 LC0001 LC0009 LC0003 LC0010 LC0007 LC0006 LC0008 LC0014
Laboratory
PROLab Plus

Annex VIII

Method proposed for EU Regulation 1007/2011

Method for the quantification of textile binary mixtures made of polytrimethylene terephthalate and polyethylene terephthalate by DSC

1 SCOPE

This standard describes a method for the quantification of textile binary mixtures made of polytrimethylene terephthalate and polyethylene terephthalate via DSC analysis.

2 FIELD OF APPLICATION

This method is applicable to textile binary mixtures of:

1. polyethylene terephthalate (PET)

with

2. polytrimethylene terephthalate (PTT)

3 PRINCIPLE

The method uses Differential Scanning Calorimetry (DSC) analysis for the quantification of textile binary mixtures PTT/PET. The quantification is based on the integration of the melting peak of PTT (about 225 °C) obtained during the heating cycle. The calibration curve is built using both pure PTT and PET manually separated from the binary mixture to be quantified.

4 APPARATUS AND MATERIALS

All specimens and pans shall be handled using clean gloves or tweezers. However, if sampling textile samples is too difficult using gloves, touch the test specimens only with clean hands.

4.1 Differential Scanning Calorimeter (DSC), the main features of which are as follows:

a) the capability to generate constant heating rate of 5 °C/min;
b) the capability to maintain the test temperature constant to within ± 0.5 °C for at least 5 min;
c) the capability to carry out step heating or any other heating mode;
d) a gas-flow rate of 50 ml/min, controllable to ± 10%;
e) temperature signals with 0.1 °C resolution and noise below 0.5 °C;
f) a recording device which is capable of recording the DSC curve, and of integrating the area between the curve and the visual baseline with an error of less than 2%;
g) a specimen holder assembly which has one or more holders for pans;

4.2 Aluminum pans, for test specimens and reference specimens, all made of the same material and of equal mass. The pans shall be physically and chemically inert under the measurement conditions to both the test specimen and the atmosphere. The pans shall be able to be fitted with lids and sealed.

4.3 Analytical balance, capable of measuring the specimen mass with an accuracy of ± 0.01 mg

4.4 Standard reference materials:

a) At least one of reference material, which has a suitable melting point in or near the temperature range to be examined, shall be used for the temperature calibration of the instrument.
b) At least one standard reference material, which has a suitable heat capacity and a melting point in or near the temperature range to be examined, shall be used for the energy or thermal-power calibration of the instrument.

4.5 Gas supply, nitrogen, analytical grade

5 TEST PROCEDURE

5.1 Manual separation

Manual separation of the binary mixture is performed to get a small quantity of pure PTT and PET needed to build up the calibration curve.

5.2 Sampling

Use the specimens (both fabric and yarns) without pre-treating or drying them.
The test specimen shall be representative of the sample being examined and shall be prepared and handle with care, if possible with gloves or tweezers; if not possible, handle the samples only with clean hands.

After having weighed the first test specimen try to prepare the other test specimens with a similar weight.

5.2.1 Sampling of fabrics

Cut small squares (as little as possible) from the fabric sample (cuts must be parallel to the sample warp or weft). The squares must be cut at least one centimeter away from the selvedges (selvages).

Weigh and record the weight of one or more squares in order to reach the desired test specimen weight (5-8 mg).

Load the weighed test specimen into an aluminum pan as described in 5.4.2.

5.2.2 Sampling of yarns

Weigh and record the weight of one or more yarns in order to reach the desired test specimen weight (5-8 mg).

Using your fingers roll those yarns up until you obtain a small ball that has to be closed in a DSC pan.

Load the weighed test specimen into an aluminum pan as described in 5.4.2.

5.2.3 Sampling of calibration samples

5.2.3.1 Sampling of calibration sample 100 % PTT or 100 % PET

To prepare the test specimen containing 100 % PTT or 100 % PET, weigh and record the weight of one or more yarns of pure PTT or PET in order to reach the desired test specimen weight (5-8 mg).
Using your fingers roll those yarns up until you obtain a small ball that has to be closed in a DSC pan.
Load the weighed test specimen into an aluminum pan as described in 5.4.2.

5.2.3.2 Sampling of calibration samples containing both PTT and PET

Prepare five calibration samples, containing both PTT and PET, with mass percentage of PTT approximately of 20 %, 40 %, 55 %, 70 % and 85 %.
Weigh and record the weight of one or more yarns of pure PTT.
Successively, weigh and record the weight of one or more yarns of pure PET, so that the total mass of the calibration specimen (PTT + PET) is in the range 5-8 mg.
Using your fingers roll up both the weighed PTT and PET yarns together until you obtain a small ball that has to be closed in a DSC pan.
Load the weighed calibration specimen into an aluminum pan as described in 5.4.2.
Use the weight of pure PTT and PET to calculate the mass percentages of the calibration samples in terms of both PTT and PET percentages.

5.3 Calibration of the instrument

5.3.1 General

Calibrate the energy and temperature measurement devices of the calorimeter at least in accordance with the instrument manufacturer's recommendations.

> *Note: Calibration is affected by:*
>
> - *the type of calorimeter used;*
> - *the gas used and its flow rate;*
> - *the type of specimen pan used, its dimensions and its position in the specimen holder;*
> - *the mass of the test specimen;*
> - *the heating and cooling rates;*
> - *the type of cooling system used.*
>
> *It is therefore advisable to define the conditions of the actual determination as precisely as possible and carry out the calibration using the same conditions.*
>
> *It is advisable carry out calibrations regularly. It is considered good practice to check the temperature and energy measurement devices using standard reference materials which have melting points close to the temperature range used for the material being analysed.*

5.3.2 Temperature calibration

Before starting analyses, carry out the temperature calibration as follows:

- use at least one standard reference material, if possible use two or more standard reference materials;

- determine the transition temperatures for the standard reference materials under the same conditions as those to be used for the test specimen (temperature program 150 °C – 5 °C/min – 300 °C; nitrogen flow rate, 50 ml/min). The transition temperatures of the standard reference materials are defined as the intercept of the extrapolated baseline and the tangent to the leading flank of the transition peak at the point of maximum gradient (i.e. the extrapolated onset temperature);
- determine the temperature calibration function by comparison of the nominal values with the recorded values, unless it can be obtained automatically by an associated computer system by feeding in the nominal and recorded values

Temperature calibration shall be performed each time the test conditions are changed. Temperature calibration checks shall be carried out at regular intervals. The repeatability of such checks shall be better than 2%.

5.3.3 Energy or thermal-power calibration

Before starting analyses, carry out the energy or thermal-power calibration as follows:

- use at least one standard reference material, if possible use two or more standard reference materials;
- examine the standard materials under the same conditions as those which will be used for the test specimen (temperature program 150 °C – 5 °C/min – 300 °C; nitrogen flow rate, 50 ml/min);
- record the plot of E versus temperature for the heat of transition or heat capacity;
- determine the energy or thermal-power calibration function by comparison of the nominal values with the recorded values, unless it can be obtained automatically by an associated computer system by feeding in the nominal and recorded values.

Energy calibration checks shall be carried out at regular intervals. The repeatability of such checks shall be better than 2%.

5.4 Analytical procedure

5.4.1 Setting up the apparatus

Switch on the instrument at least one hour prior to any testing to allow the electronic to temperature-equilibrate.

Select a nitrogen flow rate of 50 ml/min.

Note: It is advisable to protect the instrument from air draughts, exposure to direct sunlight and/or sharp changes in temperature, pressure or electric supply during measurements.

5.4.2 Loading the test specimen and calibration specimen into the pan

Do not handle pan with bare hands, use either tweezers or gloves. This should apply also to test material, however, if sampling textile samples is too difficult with gloves; touch the test specimens only with clean hands.

Select aluminum pans of the appropriate volume, ensuring that they are clean and of equal mass.Use two identical pans, one for the test specimen and one empty as reference specimen.

Use a specimen mass of 5 mg to 8 mg for the analysis. The accuracy of weighing shall be to the nearest 0.01 mg.

Load the weighed test specimen or calibration specimen into a pan and seal it with its lid using a press.

Ensure that the bottom of the pan is flat, if this is not the case discard it and start again. Good contact between the pans and the specimen holders is crucial to obtaining good data.

Visually inspect the closed pan to check if any piece of fabric or yarn is stuck outside, in this case discard the pan and start again.
Make 3 pin holes in the upper lid to allow the flow of the inert atmosphere inside the pan.

Note: *some yarn samples have a high volume, which can pose an extra difficulty in sealing the pan with the lid without leaving pieces of yarn outside.*

5.4.3 Prepare the reference pan

Do not handle pan with bare hands, use either tweezers or gloves.

Take an aluminum pan, of the same material and weigh of the ones used for the test specimen, and seal it with its lid using a press.

Ensure that the bottom of the pan is flat, if this is not the case discard the pan and start again. Good contact between the pan and the specimen holder is crucial to obtaining good data.

Make 3 pin holes in the upper lid to allow the flow of the inert atmosphere inside the pan.

5.4.4 Insertion of pans in the instruments

Use tweezers or any other suitable tool to place the reference pan and test specimen or calibration specimen pan in the specimen holders, ensuring that there is good contact between the test specimen and the pan and between the pan and holder. Close the cover of the specimen older assembly.

Before starting the analysis of the test specimen, insert as input data its mass, expressed in the mass unit requested by the instrument.

5.4.5 Temperature-scanning measurement

Perform and record a thermal cycle at a rate of 5 °C/min starting at 150 °C up to 300 °C. Bring the apparatus back to room temperature and take out the pan containing the test specimen.

Examine the pan to determine if any deformation of it or specimen overflow have occurred.

If the cell becomes contaminated from specimen overflow, clean the cell in accordance with the manufacturer’s instructions and confirm that the calibration still valid using at least one temperature and enthalpy reference standard.

Load another test specimen pan and start the following analysis.

6 CALCULATION AND EXPRESSION OF RESULTS

6.1 Integration and calculation of results

The integration is a very important and critical step for the quantification.

The area of the melting peak of PTT (at about 225 °C) shall be integrated.

In certain cases the PTT melting peak could show shoulders, the whole peak including the possible "shoulders" shall be considered and integrated as one.

In order to optimize the integration, zoom one thermogram of the test specimen under quantification so that the peak under evaluation covers at least 25% of full scale.

Integration shall be performed connecting, with a linear line, the baseline before and after the PTT melting peak.

To quantify each test specimen, calculate the linear regression slope of the calibration curve obtained integrating the PTT melting peaks of the calibration samples.

Using linear regression forced through the origin, interpolate on the correspondent calibration graph the concentration in mass percentage of PTT.

The concentration of PTT in the sample (expressed in mass percentage) is calculated as follows:

$$\%\ PTT = \frac{PTT\ enthalphy\ of\ fusion\ (area\ of\ PTT\ melting\ peak)}{slope\ of\ the\ linear\ calibration\ curve}$$

7 PRECISION

On a homogeneous mixture of textile materials, the confidence limits of results obtained by this method are not greater than ± 1 for a confidence level of 95 %.

European Commission
EUR 26564 EN – Joint Research Centre – Institute for Health and Consumer Protection

Title: Fibre Labelling. Polytrimethylene terephthalate - PTT - DuPont. Final Report.

Author(s): P. Piccinini, C. Senaldi, J. F. Alberto Lopes

Luxembourg: Publications Office of the European Union

2014 – 334 pp. – 21.0 x 29.7 cm

EUR – Scientific and Technical Research series – ISSN 1831-9424 (online)

ISBN 978-92-79-36513-3 (PDF)

doi: 10.2788/34915

Abstract

In 2011 DG Enterprise and Industry requested the European Commission's DG-JRC to technical evaluate a petition submitted by E. I. du Pont de Nemours and Company (DuPont). This petition requested the creation of a new generic fibre name under the Directive 2008/121/EC on textile names, now repealed by the EU Regulation 1007/2011. This would allow distinguishing between their fibre, polytrimethylene terephthalate (PTT) and, in particular, polyethylene terephthalate (PET) and polybutylene terephthalate (PBT), the two most common types of polyesters. Although the three polyesters are very similar in terms of chemical composition, according to DuPont, PTT fibres have a set of improved properties that justify the petition, such as for example durability, resilience, bleach resistance and comfort-stretch properties. As identification and quantification methods are required in order to allow market surveillance of textile products, the JRC was responsible for the verification of the test methods proposed by the applicant and for the development and validation of the new required ones.

Regarding identification, optical microscopy cannot differentiate between the three types of polyesters, as they are all man-made fibres with very similar structure. Also chemical solubility methods cannot be used for this purpose. On the contrary, Fourier Transform Infrared Spectroscopy can distinguish between PTT, PET and PBT. This distinction can be achieved also using Differential Scanning Calorimetry (DSC), but only on the basis of their crystallisation peaks, since the melting peaks of PTT and PBT occur at the same temperature.

The mechanical properties of PTT were studied. As most PTT fibres cannot reach 50% elongation without breaking, tests were carried out at 25% elongation. In these conditions, PTT showed an elastic recovery and a permanent deformation ranging from 65.7 to 78.1% and from 5.4 to 8.8 %, respectively. On the basis of such results, PTT cannot be considered an elastic fibre.

Regarding quantification, the usual pre-treatment protocol described in the EU Regulation 1007/2011 is applicable to PTT fibres. The correction factor *b* for mass loss during pre-treatment for PTT was established as 0%. The experimental value for the *agreed allowance* of PTT was determined (0.34%). However, for consistency with the values already adopted for polyester and elastomultiester, the value 1.50% was agreed by the members of the European Network of National Experts on Textile Labelling (ENNETL). PTT is completely soluble in method 14. The following correction factors *d* for PTT (mass loss due to dissolution methods) were determined: 1.00 for methods 2, 3, 7 and 11; 1.01 for methods 1, 4, 5, 9 and 10; 1.02 for method 13; 1.03 for methods 6, 8 and 16. Method 15 is not applicable to binary mixtures containing PTT. Several binary and ternary blends containing PTT were quantified using both manual separation method and chemical dissolution ones. The JRC developed a new DSC method that was proved to be adequate and accurate for the quantification of PTT in blends with PET. The method uses calibration curves prepared with yarns manually separated from the sample under analysis, thus ensuring a common thermal history. Different types of integration as well as multipoint and single point calibration curves based on PTT or PET melting peaks were evaluated. The JRC organised the validation of the optimised DSC method at European level, as a balanced uniform–level experiment with six levels and 15 laboratories. Statistical analysis showed that the best results were obtained using multipoint calibration curves based on the integration of PTT melting peak with a linear integration. The method was successfully validated and showed good accuracy, in terms of both trueness and precision, as proved by the following parameters: bias values (0.06 -1.30%), confidence limits at 95 % probability level (0.60 - 1.07%) and HORRAT values (0.5 – 2).

Results were presented in two meetings of the European Network of National Experts on Textile Labelling, held in Ispra, Italy, on 30th November 2012 and 4th October 2013. The definition proposed by DuPont for PTT ("*fibre formed of linear macromolecules comprising at least 85% (by mass) in the chain of an ester of 1,3-propane diol and terephthalic acid)* was consistent with the evaluation carried out. As regards the proposed name of the fibre (*triexta*) there was no consensus among the experts belonging to ENNETL.

LB-NA-26564-EN-N

As the Commission's in-house science service, the Joint Research Centre's mission is to provide EU policies with independent, evidence-based scientific and technical support throughout the whole policy cycle. Working in close cooperation with policy Directorates-General, the JRC addresses key societal challenges while stimulating innovation through developing new methods, tools and standards, and sharing its know-how with the Member States, the scientific community and international partners.

Publications Office

DOI 10.2788/34915
ISBN 978-92-79-36513-3