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QUOVADIS Project
Organization of Validation Exercises

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Symbols and abbreviations

Throughout this report the symbols, abbreviations and conventions are adopted:

Conventions

- subscript *i* represents the laboratory ($i = 1,2, ..., p$);
- subscript *j* represents the level ($j = 1, 2, ..., q$);
- subscript *t* represents the sample ($t = 1, 2, ..., g$);
- subscript *k* represents the test result ($k = 1, 2, ..., n$);
- unless otherwise stated letters after a dot are equivalent to a subscript (ex. *B.ij* is the same of B_{ij}).

Symbols

The following symbols are used:

y_{ijtk} each measurement result

z_{ijtk} the residual (value – model) associated to each results

m_j general mean of the level *j* (*m.j*)

B_{ij} laboratory component of bias under repeatability conditions in a particular laboratory $i = 1, ..., p$ at a particular level $j = 1, ..., q$

H_{ijt} term taking into account the variation between bottles (homogeneity)

SS_{Lj} sums of squares for laboratory effects *(SS.L)*

SS_{Hj} sums of squares for between-bottle effects *(SS.H)*

SS_{rj} sums of squares for repeatability *(SS.r)*

σ_{Lj} standard deviation for laboratory effects *(s.Lj)*

σ_{Hj} standard deviation for between-bottle effects *(s.Hj)*

σ_{rj} standard deviation for repeatability *(s.rj)*

σ_{Rj} standard deviation for reproducibility *(s.Rj)*

ni indicates the degrees of freedom (ν)

K, K' and K" are the factors for each level

When not at subscript *h* and *k* indicate Mandel's statistics.

C value of Cochran's test

G value of Grubbs' test

Abbreviations

ANOVA	analysis of variances
AQC	analytical quality control
CBA	cost-benefit analysis
CEN	European Committee for Standardisation
d.m.	dry matter
EC	European Commission
EU	European Union
IES	Institute for Environment and Sustainability
IC	ion chromatography
ICP	inductively coupled plasma
IRMM	Institute for Reference Materials and Measurements
ISO	International Standardisation Organisation
JRC	Joint Research Centre
LOD	limit of detection
NMS	new member state
NCV	net calorific value
PE	polyethylene
QA	quality assurance
QC	quality control
QM	quality management
QR	QUOVADIS material for ruggedness testing
QV	QUOVADIS material for validation studies
r:	repeatability as defined by ISO 5725
R:	within-lab reproducibility as defined by ISO 5725
RDF	refuse-derived fuels
SRF	solid recovered fuel
WEEE	waste electronic and electrical equipment
WP	work package

Summary

Waste-to-energy Solid Recovered Fuels (SRF) are prepared from non-hazardous waste. Their use is regulated under EU legislation and requires specifications for commercial or regulatory purposes. SRFs are seen as important contributors to a sustainable EU waste management. Directive 2001/77/EC includes in its scope the production of electricity from biomass, being defined as the biodegradable fraction of products, waste and residues from agriculture, forestry and related industries, as well as the biodegradable fraction of industrial and municipal waste. In this context the European Commission (EC) gave a mandate to the European Committee for Standardisation (CEN) to develop and validate Technical Specification (TS) concerning SRF for energy recovery and to transform these TS into European Standards. To meet these requests, a holistic validation programme covering quality management and the validation exercises for the pre-standards of CEN's Technical Committee on Solid Recovered Fuels (CEN TC 343) was designed and carried out by various members of CEN TC 343, interested Non-Governmental Organisations (NGOs) and the European Commission's Joint Research Centre (JRC).

This report gives an overview about the validation program called QUOVADIS (from QUality Management Organisation, VAlidation of Standards, Developments and Inquiries for Solid-Recovered Fuels). In particular the results stemming from the validation intercomparisons, which were carried out according to the requirements of the ISO Standard 5725 are shown. Upon statistical treatment the respective performance characteristics in terms of repeatability and reproducibility could be obtained.

1 Overview on the QUOVADIS Project

1.1 Introduction to the use of SRF

Waste incineration practices are currently being diversified and optimised in terms of the efficiency of the recovery of the energy embedded in the waste. One of these tendencies is the conversion of non-hazardous waste into an more adequate or better upgraded form for utilisation in an efficient combustion process (Chiaramonti *et al.*, 2007; Moustakas *et al.*, 2005), e.g. characterised in terms of homogeneity, being an essential parameter for the control of the combustion process. The European Directive 2000/76/EC on the incineration of waste (WID) is an indicative of the gaining recognition of this process. However, strict standards for the use of waste are essential due to the variability and presence of potentially harmful species in its composition. This is also explicitly in recital (7) of this Directive. In addition, standards for the characterisation of SRF are important, if they are to be established as tradeable goods to be used in commercial transactions.

In Europe, in the last ten years, energy policy targets and waste management legislation gave an impetus to the usage of waste derived fuels based on non hazardous wastes. These fuels, having an average content of 50 - 60% on biogenics, may contribute considerably to the reduction of CO_2 emission and the doubling of the share of renewable energy (Odenberger and Johnsson, 2007). Moreover, due to the permanent need for cost reduction, industry is interested in less expensive homogenous substitute fuels of a specified quality. The recent increase of costs of fossil energy carriers has emphasized this driving force.

At present, the main end-users of SRF are the cement and lime industry. However, the market chances in the potential bigger market of the power generation sector are increasing also due to concerns about climate change and energy security. The standardisation effort undertaken is an effect of this, as SRF standards are a useful tool to convert non-hazardous waste into a “product” for the changing energy market. Furthermore, the waste management sectors of the New Member States and Acceding Countries are characterised by an increase of residual wastes quantities within the municipal solid wastes. At the moment these countries are still characterised by a large disparity between landfilling, which is the major disposal option for all categories of waste, and incineration (Streimikiene and Klevas, 2007; Patlitzianas *et al.*, 2007). This applies to many of the old Member States such as the UK, Spain, Portugal, Italy, Ireland, Greece and Finland. In addition to this, the strong public opposition against waste incineration emphasises the need to implement SRF as waste management option.

In this framework the European Commission’s Joint Research Centre together with CEN and various stakeholders launched the QUOVADIS Project dealing with quality management organisation, validation of standards, developments and inquiries for solid-recovered fuels.

1.2 SRF in the European context – Some background information

In Europe, the recovery of energy from waste has been adopted by the European Commission as one of the sustainable waste management options, with the scope to decrease the amount of non-hazardous waste going to landfill. In this context, the use of so-called Solid Recovered Fuels (SRF) and their development as suitable alternatives for classical fuels such as coal or lignite has become an interesting option. SRF are prepared from non-hazardous waste and composed of a variety of materials of which some, although recyclable in theory, may have become available in forms that made their recycling an environmentally an unsound option (De Vriesa *et al.*, 2007). It is obtained usually shredding municipal solid waste (MSW) or steam pressure treating in an autoclave. Consequently, SRF consists largely of organic and polymeric components of municipal waste such as plastics and biodegradable waste. SRF processing facilities are normally located near a source of MSW, while an optional combustion facility is normally close to the processing facility, it may also be located at a remote location. The SRF has been introduced to distinguish it from classical refuse-derived fuels (RDF) – as a matter of fact to qualify for the SRF label, RDF must comply with a series of environmental and process-relevant standards such as minimum requirements concerning contents of some critical trace elements (mercury, thallium, cadmium), corroding capacity and net caloric value.

One may argue that incineration with energy recovery of the raw non-hazardous waste would suffice to divert waste from landfill. However, this requires the construction of dedicated incinerators, having besides a direct environmental impact also a poor acceptance in the public. Thus, co-incineration offers an additional viable waste management option. However, in order to be acceptable and usable as a replacement for classical fuels, for instance in power generation, it is of utmost importance to achieve a good quality in terms of homogeneity, energy efficiency and environmental parameters. Compared to untreated non-hazardous waste, SRF has less moisture content, a higher calorific value and a more homogenous form (frequently as pellets). As a result SRF burns more efficiently than untreated non-hazardous waste.

The use of SRF is regulated under EU legislation, and requires specifications for commercial and regulatory purposes. Thus, Directive 2001/77/EC () includes in its scope the production of electricity from biomass (Orecchini and Bocci, 2007; Athanasiou *et al.*, 2007), being defined as the biodegradable fraction of products, wastes and residues from agriculture, forestry and related industries, as well as the biodegradable fraction of industrial and municipal waste (Zamorano *et al.*, 2007; Autret *et al.*, 2007; Consonni *et al.*, 2005).

Standards for measurement and testing are fundamental instruments for the successful implementation of environmental legislation. However, the development of these tools requires a joint effort of pre-normative and co-normative research. In this context, the European Commission's Directorate-General Joint Research Centre plays an important role in supporting European Standardisation work.

Thus, the European Commission (EC) gave upon input from the JRC a specific mandate (M325) to CEN, the European standardisation body, to develop and validate Technical Specification (TS) for SRF, and then to transform these Technical Specifications into full European Standards (EN) in collaboration with the JRC. To meet these requirements, a thorough programme of validation covering (a) examination of the implementation of quality-management to the whole production process of SRF, and (b) validation exercises based on Round Robins for single Technical Specifications agreed in the various working groups of CEN TC 343 was setup under the umbrella of the QUOVADIS-Project. The Technical Specifications to be validated so as to guarantee the quality of the produced SRF, can be grouped as follows:

- Sampling: statistical considerations, preparation of a laboratory sample, reduction of a laboratory sample arriving at the lab to a test sample used to execute a specific measurement;

- Tests for chemical properties such as major and minor constituents (Cl, F, Br, S, N, C, H), heavy metals and trace elements, (As, Cd, Hg, Pb, Co, Cr, Cu, Mn, Ni, Sb, V, Zn, Al, K, Na, P, Si, Ca);

- Physical properties, such as moisture- and ash-content, volatiles and parameters such as lower heating value, grain-size/particle-size distribution;

- Biological parameters (biodegradable fraction).

The results from the validation tests are a requirement to finalise the European Standards, which will then be implemented via European Legislation. Another aspect of QUOVADIS is the gathering of information about production and use of SRF by the New EU-Member States (Nilsson *et al.*, 2006).

1.3 Project description

QUOVADIS-Project aims to deliver the methodological/performance characteristics of CEN standards for SRF, taking account of the proposed procedures for QM and classification, by assessing the various contributors of uncertainty. A synthetic scheme of the structure is set out here:

- WP 1 – Co-ordination and Management
- WP 2 – A holistic approach towards quality management and classification
- WP 3 – Organisation of validation exercises
- WP 4 – Sampling
- WP 5 – Physical parameters
- WP 6 – Chemical parameters
- WP 7 – Biological parameters
- WP 8 – Data collection
- WP 9 – Dissemination

The main aim of QUOVADIS is the validation of the draft technical specification prepared by the European Standardisation Body CEN within its Technical Committee 343. As validation is the main aim of QUOVADIS, a very important step is the production of the testing materials to be used for ruggedness testing and laboratory intercomparisons. Another important objective of QUOVADIS is the collection of information on waste-management and market-potential for SRF in the New Member States of the EU.

1.3.1 Description of the main activities

1.3.1.1 A holistic approach towards quality management and classification

The main aim of this activity is to validate the draft standard for specification and classification and Quality Management of SRF that CEN is currently finalising. Such validation is necessary to demonstrate that the future standards are fit-for-purpose.
Other related objectives are:

- to develop an European database on quality of SRF according to the classification system introduced;

- to develop QM guidelines and model manuals to facilitate the later application in plants producing SRF.

To fulfil these main objectives, four tasks have been conceived:

- Assessment of current status on quality assurance of SRFs and methods for Cost Benefit Analysis (CBA)
- Establishment of a European Database on the quality of SRF and validation of the draft standard on classification
- Production of QM Guidelines and model-manuals for the application of QM systems
- Implementation and validation of the draft standard on QM

1.3.1.2 Validation exercises – production of testing materials, validation intercomparisons and statistical evaluation

This activity has the following objectives:

- identification of representative SRF for the production of test materials;
- production and characterisation of test materials for validation studies;
- to perform ruggedness testing and validation exercises according to ISO-Standard 5725;
- to perform a statistical evaluation of validation intercomparisons (performance characteristics of methods).

This activity is divided into four tasks:

- Identification of representative SRFs for production of test materials including gathering of existing analytical data
- Characterisation and dispatch of five EU-representative SRF-test materials for validation studies and experiments in compliance with the respective ISO-Standards
- Design of ruggedness testing and validation exercises
- Statistical evaluation of validation results and determination of method performance characteristics from the intercomparison data

1.3.1.3 Sampling of SRFs

This part of the project has the following main objectives:

- drafting of a reference document for the validation of sampling;
- validation of the draft standards for the sampling of SRFs;

The respective work comprises the following tasks

- The design of a reference document for the validation for sampling of SRF
- Ruggedness testing of sampling procedures down to the step of the production of a representative sample with the required sample size using collaborative field trials
- Validation exercise of sampling procedures down to the step of the production of a representative sample with the required sample size using collaborative field trials

1.3.1.4 Physical and chemical parameters of SRF

For this activity the overall objective is the evaluation of standards' robustness for the measurement and testing of physical properties. To this end, the following tasks were defined:

- Ruggedness testing of procedures for physical properties
- Set-up of the respective intercomparisons
- Validation of the sample-reduction procedure from laboratory sample to test portion comparing different milling techniques and considering different final dimensions

1.3.1.5 Biological Parameters

Main objective is the determination of biomass content of SRF in relationship CO_2-Trading and the Biomass Action plan established by the European Commission. This work package is divided into the following tasks:

- Carry out determination of the content of biodegradable material using the manual sorting and the selective dissolving methods.
- Work out information on the ruggedness of procedures for biological parameters
- Design a draft reference document for determining the biodegradable fraction of SRF

1.3.1.6 Waste management and solid-recovered fuel potential in the New Member States of the EU

The main scope is to collect information about waste-management and the market-potential for SRF in the NMS, taking account of relevant waste-streams, waste-management strategies and policy, and relevant organisations in NMS. The following activities are envisaged:

- collecting information on current and expected waste-management strategies in NMS and analysing how implementation of standards for SRF might affect those strategies to maximise the use of SRF;
- gathering and updating information about current practices in waste-management in NMS;

- determining data about the waste-streams that are relevant for SRF-production in NMS. Considering how NMS could influence the perspective of the-production and use of SRF in the enlarged EU;

- foster the application of the new standards so that SRF will contribute to lower-cost energy production, and reduce specific investments in infrastructure, e.g. by substituting co-combustion for incineration.

The work is focused on an overview of relevant waste streams and waste-management strategies in the NMS compared to Europe-15, with regard to:

- laws, standards and time table for application of EU legislation;

- overview of waste-management policies in Central Eastern Europe in the context of the production and utilisation of SRF;

- overview of waste-management policies in Europe-25;

- study comparing future directions of waste management strategies in Europe-25;

Consequently, this activity is divided into two tasks:

- Information gathering on waste management in the NMS

- Assess the potential of SRF in the New EU accession MS

1.4 Role of a set of validated standards for SRFs

Waste-to-fuel-production for a later energy conversion is one option of waste management when done properly and not in competition with environmentally sound material recovery options. The potential of waste-to-fuel production to reduce pressure on landfill is generally acknowledged. Solid recovered fuels represent a significant potential storable source of indigenous energy and therefore contribute to the security of energy supply for the EU (Lund, 2007; Caspary *et al.*, 2007).

Standards and specifications help promote their use while ensuring a level of environmental protection at least equivalent to the traditional energy sources they are replacing, subject to criteria and definitions of the Waste Framework Directive. In this context, solid recovered fuels (SRFs) have a potential application for the substitution of traditional fossil energy carriers such as hard coal or lignite. Generally, SRFs can be considered "clean" fuels, from an emissions perspective if their content of heavy metals is below certain levels. As a general rule heavy metal concentrations therefore need to be kept as low as possible.

Not all refuse-derived fuels can be seen automatically as SRFs. Here the upcoming CEN standards have a role to clarify the situation. To be defined as a SRF, the fuel shall comply with certain minimum requirements concerning their homogeneity as well as their net calorific value (NCV).

An optimal waste management strategy has to consider strongly local requirements and particularities (Lopez Gonzales *et al.*, 2007; Terrados, 2007). Hence, the availability of a possibly broad range of different technological and systems solutions besides sheer material re-cycling (for example waste prevention and reuse) may enable a better waste management. In this context it has to be stressed that the volume of waste plastics which cannot be sensibly recycled as a material represents 15% of imported solid fuels into EU-15.

Public confidence into thermal waste treatment is still a critical issue. On one hand, steps towards an integrated quality management for SRF-producers, including a proper documentation of incoming waste streams, are of paramount importance to gain confidence. Generally, transparency and guaranteeing a high level of environmental protection is seen as a key element in this discussion. In any assessment of environmental impacts it is important to consider overall fossil fuel energy balances and associated climate change effects. Thus, it was stated that Solid recovered fuels produce lower greenhouse gas emissions than conventional fuels and therefore contribute to the important climate change objectives of the EU (Bomb *et al.*, 2007; Clift, 2007).

Standardisation work as accomplished in the QUOVADIS-Project can contribute to increase public confidence as well as market opportunities. Concerning the latter point, the discussion, if and when a waste-derived material can be qualified as a product, remains controversial. If materials as SRFs are subject to trading, i.e. the exchange of goods against money, they are de facto handled as such.

Unfortunately, this has a series of legal implications, which are not yet fully clarified at least in the EU. However as a matter of fact there is a lot of waste handled as a product under the waste regime and there are relevant environmental protection reasons for this.

In addition to that there are still large discrepancies in the environmental legislation in the various Member States and Acceding Countries (Streimikiene *et al.*, 2007), which should be overcome. For instance, Bulgaria and Poland reported on difficulties in environmental legislation unification in respect to waste-to-fuel production. The need of informing the potential customers and the society in the New Member States and Acceding Countries on the subject of waste-to-fuel production is apparent.

Many synergies are possible between renewable biomass resources and other waste in the production of SRF, (e.g. paper/plastic mixtures from municipal waste, as in the Sub-coal process in the Netherlands (Ptasinski *et al.*, 2007)).

2 Preparation of the validation exercises

The validation exercises organized in the context of the QUOVADIS Project were necessary in order to establish the so-called performance characteristics of the various standards for measurement and testing of chemical, physical and biological properties of solid recovered fuels (SRF). To this end, QUOVADIS incorporated a own work package (WP 3) with the following tasks

- identification and sampling of representative SRF for the production of test materials
- production and characterisation of test materials for validation studies;
- to perform ruggedness testing and validation exercises according to ISO-Standard 5725;
- to perform a statistical evaluation of validation intercomparisons (performance characteristics of methods).

2.1 Identification and sampling of representative SRF for the production of test materials

In order to cover the needs for representative SRF samples for the ruggedness testing and validation a set of five different SRF materials had to be identified. Many different qualities of SRF are produced within the European Union, some in small quantities, some in very large quantities, some for very specific purposes and some for more general applications, some used internationally and some only at regional level. Therefore, the selection of the five representative SRF to be used as basis material for the validation of the TS defined by the CEN TC 343 was limited to materials satisfying the following principles:

- The SRF must comply at least with one of the classes as defined in the TS established by CEN TC WG2 for NCV, Cl and Hg content;
- Due to the small number of SRF materials to be selected compared to those available, only widely produced (industrial production must exist) and/or used SRF (i.e. SRF which meet the requirements of the main final users) should be taken into account;
- The selected SRF needed to be produced and sampled in different EU member states, so that five countries could be involved, thus widening the interest at European level for the project and helping the diffusion of the awareness about SRF;
- Data about the composition, behaviour and production processes must be available;
- The five SRF should represent a good panel of usual physical aspects of SRF;
- Availability and possibilities of delivery to the JRC has been considered;

It should be noted that, with just five selected types of SRF materials, it was impossible to cover the five classes for NCV, five classes for chlorine content and five classes for the mercury content. This implies that some classes for some parameters will not be represented in the SRF selected, even if such SRF materials might exist in practice. In fact, the restrictions due to the limited human, technical and financial possibilities of the QUOVADIS project had to be taken into account. The decision on this point was to try, to cover as far as possible different classes, when complying as far as possible with the selection principles defined above. Based on the above considerations, the resulting set was composed by the SRF materials described here below.

2.1.1 Sample type A – SRF produced from shredded tyres

This kind of SRF is used in most of the European countries, mainly in cement plants. It has a high calorific value compared to other, so that it is a good test material for the high NCV classes. The selected country for the sampling of this SRF was France and the samples were collected at the Norvalo Centre de Valorisation des Pneumatiques at Dompierre-Bequincourt, near Amiens (Fig. 1-2).

After plant inspection, five 60 litres PE barrels have been filled with the tyre pieces (which are composite materials containing of iron, rubber and cotton textile) from the open air stocks of semi-processed material. In order to preserve representativeness, samples were collected in different parts of the stocks.

Figure 1: Shredded end-of-life tyres

Figure 2: Manual sampling of SRF

2.1.2 Sample type B – SRF produced from demolition wood

This kind of SRF is widely used in the Nordic countries, Germany and in other European countries at a smaller scale. It is used mainly in district heating and power plants, but can also be used in cement and lime plants. The selected country for the sampling of this SRF was Finland, where the material was collected at the Lassila & Tikanoja's plant in Kerava (about 30 km north from Helsinki) (Fig. 3-4).

This waste management plant processes different types of waste such as waste electrical and electronic equipment (WEEE), plastic waste (so-called "*light fraction*") as well as demolition wood. Twelve 60 litres PE barrels have been filled from the process output after plant inspection. Impressions from the sampling can be gained below.

Figure 3: SRF derived from demolition wood

Figure 4: Sampled demolition wood

Figure 5: SRF pre-treatment centre of SCORIBEL in Seneffe, Belgium

Figure 6: SRF produced by SCORIBEL

2.1.3 Sample type C – SRF produced from sewage sludge

SRF produced with dried waste water sewage sludge, filter cake and coal or lignite residues. This SRF usually presents a low NCV compared to other. It is mainly used in cement plants to substitute fuel (through its the calorific value) and to save raw materials (through its ash content). Use in power plants is also possible, but depends of the mineral contents. Some 100 kg of suitable sludge SRF were obtained from SCORIBEL in Seneffe, Belgium (Fig. 5 – 6). After collection this material was delivered to IRMM in Geel, Belgium, for storage and processing.

2.1.4 Sample type D – SRF produced from Municipal waste

This SRF is produced from the combustible fraction of municipal solid waste. Those SRF are produced in Germany, Finland, Sweden, Belgium, Italy, The Netherlands and probably also in other member states. The physical aspect of this SRF is small pieces of some centimetres. The sampling of this kind of material was carried out at the ECODECO plant near Milan, Italy, where 6 PE barrels of SRF (soft pellets) were sampled and shipped to the JRC for further processing. Impressions from the sampling site can be gained from the pictures below (Fig 7 – 8).

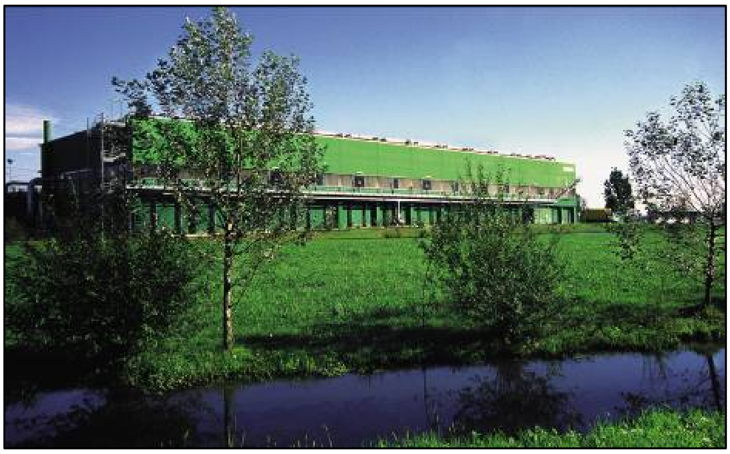


Figure 7: ECODECO SRF Processing plant, Milan, Italy

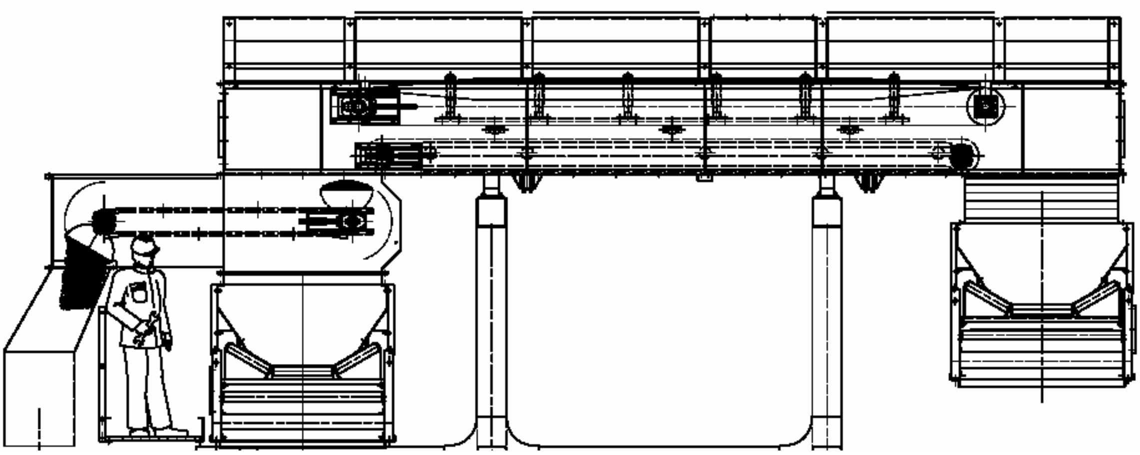


Figure 8: Scheme of automatic sampler used at ECODECO

2.1.5 Sample type E – SRF produced from Municipal waste (paper and plastic rich)

SRF produced from industrial and/or municipal non hazardous waste like a combination of plastic, cardboard and paper, transformed into pellets that ease the transport and manipulation of that SRF. Such pellets are mainly produced in Germany and in the Netherlands. The calorific value is medium, the chlorine content can be determined depending on the quality chosen (Fig. 9 – 11). This type of fuel was sampled at Remondis plant in Erfstadt, in the surroundings of Cologne, Germany. In this plant a highly advanced technology is employed to enhance the fuel quality at the end of the process; a detailed presentation of the process was given us before the sample collection. Five 60 litres barrel plus one 40 litres box of sample were collected according to the plant's QA/QC sampling procedures.

Figure 9: REMONDIS device for SRF production

Figure 10: Filling the barrels

Figure 11: SRF from municipal waste *"light fraction"*

2.1.6 Sample type F – Acid-digested SRF for sample preparation effect assessment.

Two PE barrels of SRF from the Pirelli IDEA GRANDA plant were sampled in Cuneo, Italy and transported to JRC IES for further processing (Fig. 12). From this material 5 L of acidic digest were produced following the draft TS for mineralization (Fig. 13). The digest was sent to JRC's Institute for Reference Materials and Measurements in Geel, Belgium (IRMM) for ampouling.

Figure 12: Sub-sample from the IDEA GRANDA plant

Figure 13: Production of Acidic Digest from SRF by open-vessel digestion under reflux condition

2.2 Production and characterisation of the test materials for validation studies

The processing of the sampled raw SRF into samples ready for distribution to the laboratories participating in the validation study, was co-ordinated at IRMM.

After an initial period of process optimisation, ruggedness test materials were prepared and homogeneity tests were carried out jointly by the WP partners. Homogeneity of the ruggedness test materials was assessed in terms of ash content (IVD, Stuttgart), calorific value (SLU, Umeå), and content of Al, S, Cl and Cr by XRF (IES-JRC-EC, Ispra). The chemical properties (Hg, XRF) were tested on 0.5, 1.0 and 1.5 mm nominal granulometries, the physical properties (ash, moisture, calorific value) were measured on 1.0 and 6.0 mm nominal granulometries. The expected trend of having more uniform analysis data with decreasing granulometry is confirmed. On the other hand, the effect of the additional processing to obtain a smaller granulometry does not seem to affect significantly the level of the measured analytes, except moisture. All QR-materials (QR-A, QR-A2, QR-B, QR-C, QR-E) met the agreed 'between-sample' homogeneity criterion (30 %) using a 1 mm sieve insert in the mill. This result was a prerequisite for processing the validation test materials along the same protocol. Based on these results, the final validation materials were produced (Fig. 14 – 19). An example of homogeneity testing results is shown in Fig. 20.

The materials QV-D and QV-E2 were judged potentially unstable during transport. Therefore, samples of these materials were submitted to a dedicated stability study. Based on the test results obtained [ash content (IVD, Stuttgart), calorific value (SLU, Umeå), elements by XRF (IES-JRC-EC, Ispra), biogenic fraction (INFA)] the materials could be declared stable during transport, at least with respect to the tested parameters.

Furthermore, 5 L of an acidic digest were produced as AQC samples for the validation exercise on the critical heavy metals. The automated flame-seal ampouling from a stirred solution assured the homogeneity and stability of this material (QV-F).

Thus, in total 6 classes of materials were prepared, in 11 processing batches (5 QR materials and 6 QV materials), and requiring 60 filling sequences. (On average 5 different sample sizes were prepared per batch). In total 3382 individually numbered samples were obtained, a considerable number of which were used in homogeneity and stability tests. Finally, 1982 samples were distributed to the 28 laboratories participating in the validation study. Average processing time for all materials from receipt of raw materials to dispatch of QRmaterials and from receipt of homogeneity test results to packing of validation test materials, was less than 8 weeks.

Figure 14: Equipment used for the processing of the SRFs

Figure 15: Ruggedness test materials produced

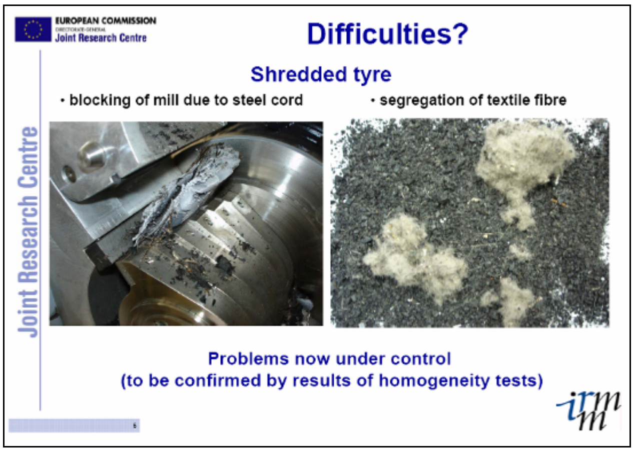


Figure 16: Problems encountered during production

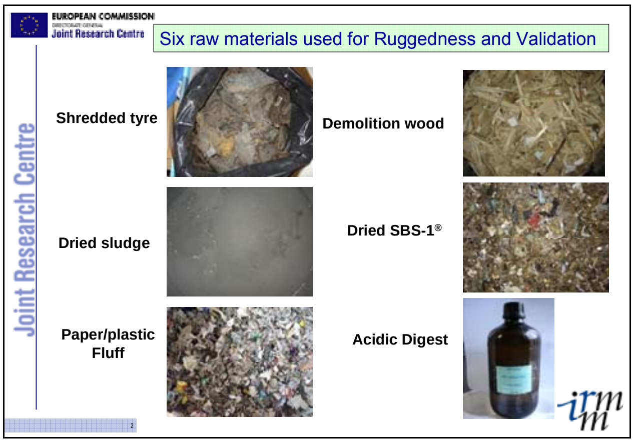
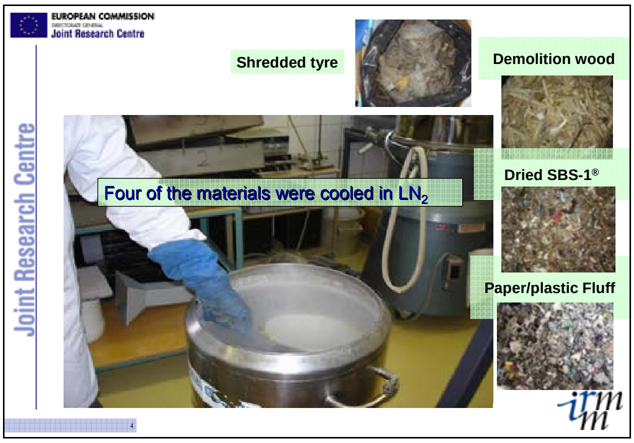


Figure 17: Overview on the materials used



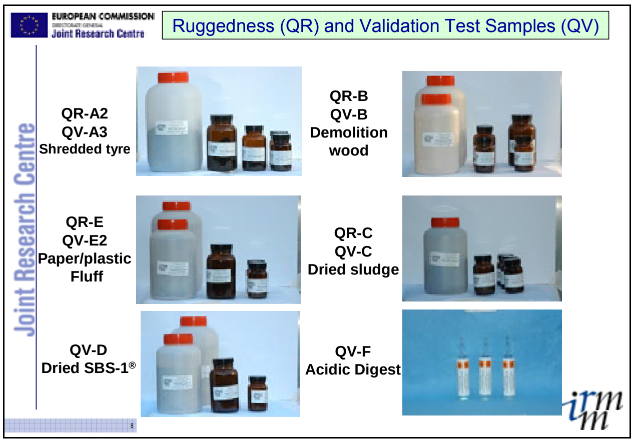


Figure 18: Nitrogen cooling of material prior to grinding operations

Figure 19: Ready materials

The homogeneity of the test materials was investigated in terms of different properties: ash content, Hg content, moisture, calorific value, and overall composition (by XRF). The chemical properties (Hg, XRF) were tested on 0.5, 1.0 and 1.5 mm nominal granulometries, the physical properties (ash, moisture, calorific value) were measured on 1.0 and 6.0 mm nominal granulometries. The expected trend of having more uniform analysis data with decreasing granulometry is confirmed. On the other hand, the effect of the additional processing to obtain a smaller granulometry does not seem to affect significantly the level of the measured analytes, except moisture.

The target level of between-bottle homogeneity (set at 30 %) has been met in the materials and for all properties evaluated. One can proceed to the processing of the validation study test samples. From the results of the homogeneity tests, the 1.0 mm nominal granulometry is sufficiently fine to obtain the required repeatability. An example of homogeneity testing results is shown in Fig. 20.

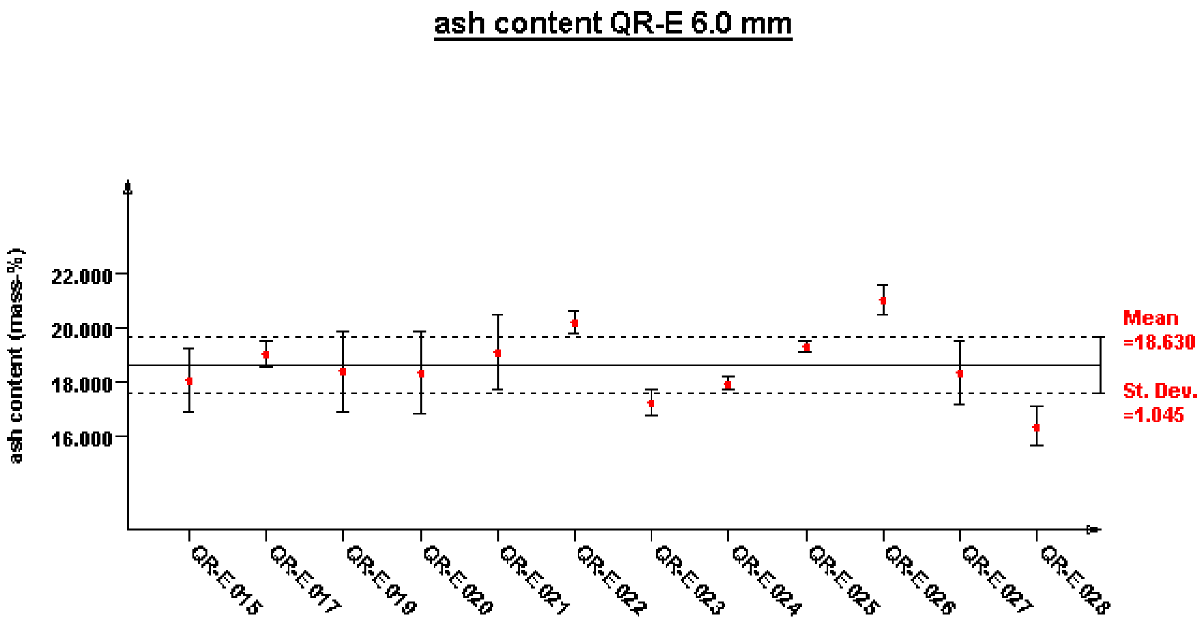


Figure 20: Ash content homogeneity test for sample type E

2.3 Statistical data treatment concept for the validation exercise based on ISO 5725

After the selection of the participants in the intercomparisons, validation samples have been delivered to the laboratories. A Microsoft® Excel® template has been prepared, in cooperation with CESI-Ricerca, in order to speed up final collection of data for the validation experiment. A specific database management system has been prepared in order to minimize the interferences on the dataset subsequent to manual data manipulation. The data received on the data recording template have been processed through a macro script and then imported automatically into the database. Once constituted and consolidated the dataset, the data have been pre-processed in order to discard the values below the respective limits of detection (LOD), as their use may bring to an artifact in the statistical treatment.

The statistical data analysis was based on the international standard ISO 5725-2 '*Accuracy (trueness and precision) of measurement methods and results – part 2: Basic method for determination of repeatability and reproducibility of a standard measurement method*' (ISO, 1994a), as generalized in chapter 5 of the ISO 5725-5 '*Accuracy (trueness and precision) of measurement methods and results – part 5: Alternative methods for the determination of precision of a standard measurement method*' (ISO, 1994b) . Data analysis was carried out by means of the statistical software package R, by a dedicated script developed on purpose. This specific approach chosen has the following paybacks:

a. The high flexibility allows to deal in an automatic way with missing replicates and outliers;

b. The procedure is in principle iterative. The presence of very deviant outliers can distort the view of the whole distribution. Multiple outliers can mask each other; by eliminating outliers, new outliers and stragglers may pop up. During each iteration outliers are eliminated and the statistical analysis is repeated to study the distributions in order to trace 'new' outliers or stragglers. This iterative procedure continues until no new outliers are found or, if the iterative approach brings the program to instability or to senseless results, then the number of iterations is limited by scrutiny of the data by the Mandel statistics. A scheme of the iterative procedure is shown in the flowchart below (Fig. 21).

c. The design for heterogeneous materials proposed in clause 5 of ISO 5725-5 yields information about the variability between samples that is not obtainable simply from the uniform level design described in IS0 5725-2. Therefore valuable information may be gathered by this approach, with an associated cost related to the fact that: the proposed design requires more samples to be tested.

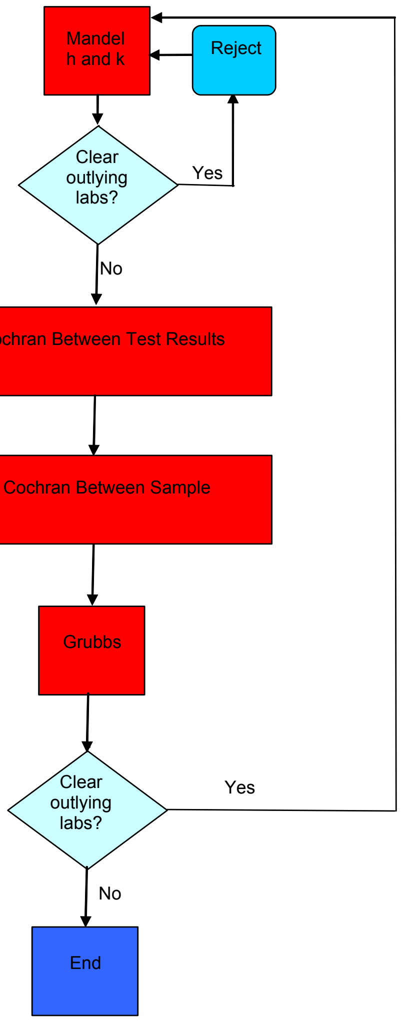


Figure 21: Iterative procedure for statistical data treatment

The assumptions made to chose the statistical design are that, with inhomogeneous materials like SRF, experiments should involve (for each level of the parameters) three factors arranged in a hierarchy: with a factor "laboratories" at the highest level in the hierarchy, a factor "samples within laboratories" as the next level in the hierarchy, and a factor "test results within samples" as the lowest level of the hierarchy.

The general formulas from paragraph 5.9 of the above mentioned ISO standard have been employed, because in the chosen design the number of replicates per sample bottle was 6 for certain key parameters and 3 for the others, therefore the fixed design formulas of paragraph 5.4 (two bottles, two replicates per bottle) was not appropriate. Each of the data from the design for a heterogeneous material is represented by:

y_{ijtk}

where

subscript i represents the laboratory ($i = 1,2, ..., p$);

subscript j represents the level ($j = 1, 2, ..., q$);

subscript t represents the sample ($t = 1, 2, ..., g$);

subscript k represents the test result ($k = 1, 2, ..., n$).

In the QUOVADIS project, after a cost/benefit evaluation, we have chosen a design where q=5, g=2 and n=6 for key parameters (Calorific Value, Hg, Cl and related elements) and n=3 for the others. Anyway, after the rejection of outliers and <LOD this values may not be a fixed constant anymore, nor it is p for the various levels, and the script for the statistical computation has to take this into account. The number of responding laboratories, including those who found <LOD and outliers, is indicated at the beginning of the part of the report concerning the statistical calculations for each specific parameter.

2.3.1 Statistical model

The basic statistical model used for an experiment with a heterogeneous material is similar to the classical ANOVA approach given in equation (1) in clause 5 of IS0 5725-1:1994, expanded to become:

$$y_{ijtk} = m_j + B_{ij} + H_{ijt} + e_{ijtk} \qquad (1)$$

Where

m_j represents the general mean of the level *j*, defined by

$$m_j = \sum_i \sum_t \sum_k \frac{y_{ijtk}}{n_j} \qquad (2)$$

where n_j is the total number of test results included in the sum;

B_{ij} represents the laboratory component of bias under repeatability conditions in a particular laboratory $i = 1, ..., p$ at a particular level $j = 1, ..., q$, defined by

$$B_{ij} = \sum_i \sum_t \frac{(y_{ijtk} - m_j)}{n_{ij}} \text{ = laboratory average – general average} \qquad (3)$$

where n_{ij} is the total number of test results included in the sum;

H_{ijt} represents the extra term taking into account the variation between samples ($t=1,...,g$) for the laboratory *I* at the level *j*, defined by

$$H_{ijt} = \sum_i \frac{(y_{ijtk} - m_j - B_{ij})}{n_{ijt}} \text{ = bottle average – laboratory average} \qquad (4)$$

where n_{ijt} is the number of results included in the sum;

e_{ijtk} represents the random error of test result $k = 1, ..., n$, obtained for sample *t* in laboratory *i* at level *j*, under repeatability conditions, represented by the residual

$$z_{ijtk} = y_{ijtk} - m_j - B_{ij} - H_{ijk} \text{ = test result - sample average.} \qquad (5)$$

It is reasonable to assume that the variation between samples is random, it does not depend on the laboratory, but it may depend on the level of the experiment, so the term H_{ijt} has a zero expectation, and a variance:

$$\mathrm{var}(H_{ijt}) = \sigma^2_{H_j} \qquad (6)$$

Based on the described model, the statistics *m*, *B*, *H* and *z* are calculated according to the formulas (2)-(5), then the following sums of squares are calculated:

$$SS_{Lj} = \sum_i (n_{ij} B_{\mathrm{ij}})^2 \tag{7}$$

which represents the sums of squares for laboratory effects;

$$SS_{Hj} = \sum_i \sum_t (n_{ijt} H_{\mathrm{ijt}})^2 \tag{8}$$

which represents the sums of squares for sample effects;

$$SS_{Hj} = \sum_i \sum_t \sum_k (z_{ijtk})^2 \tag{9}$$

which represents the sums of squares for repeatability.

The degrees of freedom, respectively for laboratory effects, for sample effects and for repeatability are calculated according to the following formulas:

$$v_{Lj} = p_j - 1 \qquad v_{Hj} = g_j - p_j \qquad v_{Hj} = n_j - g_j \tag{10}$$

where p_j is the number of laboratories which report at least one valid test result, g_j is the number of samples for which at least one test result is reported and n_j is the total number of test results.

For each laboratory *i* the following factors are calculated:

$$n_{ij} = \sum_t n_{ijt}$$

$$K_{ij} = \sum_t n_{ijt}^2 \tag{11}$$

For each level j the following factors are calculated:

$$K_j = \sum_i n_{ij}^2 \tag{12}$$

$$K'_j = \sum_i K_{ij} \tag{13}$$

$$K''_j = \sum_i K_{ij} / n_{ij} \tag{14}$$

The repeatability standard deviation s_{rj}, between-samples standard deviation s_{Hj}, between-laboratory standard deviation s_{Lj}, and reproducibility standard deviations s_{Rj}, are calculated using:

$$s_{rj}^2 = SS_{rj} / v_{rj} \tag{15}$$

$$s_{Hj}^2 = \frac{\left[SS_{Hj} - v_{Hj} * s_{rj}^2\right]}{\left(n_j - K''_j\right)} \tag{16}$$

$$s_{Lj}^2 = \frac{\left[SS_{Lj} - \left(K''_j - K'_j / n_j\right) * s_{Hj}^2 - v_{Lj} * s_{rj}^2\right]}{\left(n_j - K_j / n_j\right)} \tag{17}$$

$$s_{Rj}^2 = s_{rj}^2 + s_{Lj}^2 \tag{18}$$

2.3.2 Outliers scrutiny and rejection

The dataset for each parameter is screened at first by a graphical approach based on Mandel h and k statistics, aimed at the rejection of clearly outlying laboratories. The formulation of the Mandel statistics comprising the homogeneity effects can be found in paragraph 5.6.1 of ISO 5725-5.

The stragglers and outliers data are the scrutinised according to Cochran's and Grubbs' tests, as generalised for inhomogeneous samples in 5.6.2. The former one is aimed at the rejection of anomalous range intervals, both between test results and between sample, while the latter tests for stragglers and outliers in the cell averages, corresponding to one participating laboratory *i* at one single level *j*.

These tests have been acted as in the scheme on the following page. As it becomes apparent from the flowchart, the procedure can be in principle iterated until no further outliers are found. Actually this *black-box* approach is not straightforwardly applicable to this kind of dataset because of the following limitations:

- The statistical models underlying the computation are based the assumption of normality of the statistical distributions of the results, but the relatively small number of repetitions can bring to distorted distributions with consequent excessive rejection of the extremes, especially for the within-bottle variation Cochran test;
- The presence of a single too strong outlier can have a major influence on the standard deviation of the statistical distribution, therefore avoiding the rejection of the outlier itself purely according to the Cochran test;
- Finally, the presence of several laboratories with very precise test results can cause a very tiny distribution and the consequent rejection of data by laboratories whose results are only a bit more broadly distributed and whose range is still within reasonable values.

To cope with these limitations the iterative procedure is applied manually and the Mandel *h* and *k* statistics are used as check whether it makes sense or not to stop the iterations.

3 Organization of the validation exercises

3.1 Participants

In total, a group of 28 European laboratories had been selected for the intercomparison experiments. The analyses carried out by each of those laboratories are listed in the following pages.

Table 1 – List of participants

Affiliation	*Measured parameter*
Analytica Srl Corte Olona (PV) – Italy	- Major Elements after acid dissolution - Minor Elements after acid dissolution
Analytical srl Arzignano (Vicenza) – Italy	- Major Elements after acid dissolution - Ash content - Volatile Matter - Minor Elements after acid dissolution
Camera di Commercio, Industria, Artigianato e Agricoltura di Trieste (C.C.I.A.A.) Trieste – Italy	- Cl, F, S, Br by oxygen combustion + IC - B - ashing (ASTM D6722 + XRF) - Major Elements after acid dissolution - Minor Elements after acid dissolution - Moisture Content - Biomass/biodegradable - Calorific Value - Ash content - Volatile Matter
Consiglio Nazionale Delle Ricerche (CNR) *Istituto Ricerche sulla Combustione* Naples – Italy	- Bridging Properties
Dresden University of Technology *Department of Waste Management* Dresden – Germany	- Cl, F, S, Br by oxygen combustion + IC - Major Elements after acid dissolution - Volatile Matter - Minor Elements after acid dissolution - Moisture Content - Calorific Value - B - ashing (ASTM D6722 + XRF) - C-H-N Flash Combustion - Ash content
ENEL *Produzione Ricerca* Tuturano (Brindisi) – Italy	- Major Elements after acid dissolution - Ash content - Moisture Content - B - ashing (ASTM D6722 + XRF) - Minor Elements after acid dissolution
Energy research Centre of the Netherlands (ECN) *SF-CA* Petten – The Netherlands	- trace elements - 5 - Cl, F, S, Br by oxygen combustion + IC - Volatile Matter - Major Elements after acid dissolution - Biomass/biodegradable - Moisture Content - Calorific Value - Ash content - Minor Elements after acid dissolution - C-H-N Flash Combustion
Fachhochschule Münster *Labor für Abfallwirtschaft, Siedlungswasserwirtschaft, Umweltchemie (LASU)* Münster – Germany	- Calorific Value - Major Elements after acid dissolution - Moisture Content - Ash content - C-H-N Flash Combustion - Minor Elements after acid dissolution - trace elements - 5 - Cl, F, S, Br by oxygen combustion + IC - B - ashing (ASTM D6722 + XRF) - Bulk Density - Biomass/biodegradable
IMAT-UVE GmbH Mönchengladbach – Germany	- Particle dimesion & Particle size distribution - Ash content - Density of pellets and briquettes - Moisture Content

Affiliation	*Measured parameter*
	- Bulk Density - Durability - Volatile Matter - Calorific Value
Institute for Chemical Processing of Coal - ICHPW Zabrze – Poland	- Biomass/biodegradable - Major Elements after acid dissolution - Volatile Matter - Ash content - ash melting behaviour - Calorific Value - Moisture Content - C-H-N Flash Combustion - B - ashing (ASTM D6722 + XRF)
Institute of Power Engineering Warsaw – Poland	- Bulk Density - Calorific Value - Volatile Matter - Moisture Content - Bridging Properties - Durability - Biomass/biodegradable - Ash content - Density of pellets and briquettes - Particle dimesion & Particle size distribution
Labanalysis Casanova Lonati (PV) – Italy	- Cl, F, S, Br by oxygen combustion + IC - Major Elements after acid dissolution - Minor Elements after acid dissolution - trace elements - 5
Montanuniversität Leoben *Institut für Nachhaltige Abfallwirtschaft und Entsorgungstechnik,* Leoben – Austria	- Biomass/biodegradable; - Major Elements after acid dissolution - B - ashing (ASTM D6722 + XRF) - Ash content - Minor Elements after acid dissolution - Cl, F, S, Br by oxygen combustion + IC - Calorific Value - Moisture Content
Montanuniversität Leoben *Lehrstuhl für Thermoprozesstechnik,* Leoben – Austria	- Volatile Matter - Ash content - ash melting behaviour - C-H-N Flash Combustion - Calorific Value - Density of pellets and briquettes - Moisture Content - Bulk Density
Ofi **Österreichisches Forschungsinstitut für Chemie und Technik** Vienna – Austria	- Durability - A - nitric acid + ICP - Moisture Content - Cl, F, S, Br by oxygen combustion + IC - Major Elements after acid dissolution - Calorific Value - Minor Elements after acid dissolution - Volatile Matter - Bulk Density - Density of pellets and briquettes - C-H-N Flash Combustion - Ash content
Politecnico di Milano *DIAAR* Milan – Italy	- Major Elements after acid dissolution - Moisture Content - B - ashing (ASTM D6722 + XRF) - Minor Elements after acid dissolution - Cl, F, S, Br by oxygen combustion + IC
Ramboll Finland Oy Espoo – Finland	- Ash content - Volatile Matter - Moisture Content - Major Elements after acid dissolution - Calorific Value - C-H-N Flash Combustion - Cl, F, S, Br by oxygen combustion + IC - B - ashing (ASTM D6722 + XRF) - Biomass/biodegradable
S.A.F.A.S. Srl Divisione Analitica Ferrara – Italy	- Calorific Value - C-H-N Flash Combustion - B - ashing (ASTM D6722 + XRF) - Ash content - Cl, F, S, Br by oxygen combustion + IC - Major Elements after acid dissolution

Affiliation	*Measured parameter*
	- Minor Elements after acid dissolution
SGS INSTITUT FRESENIUS GmbH *Environmental Services* Berlin – Germany	- Minor Elements after acid dissolution - C-H-N Flash Combustion - Calorific Value - Bulk Density - Density of pellets and briquettes - Particle dimesion & Particle size distribution - Volatile Matter - Ash content - Moisture Content - Biomass/biodegradable - Cl, F, S, Br by oxygen combustion + IC
SP – Swedish National Testing and Research Institute Boras – Sweden	- A - nitric acid + ICP - Cl, F, S, Br by oxygen combustion + IC - Ash content - Minor Elements after acid dissolution - Calorific Value - C-H-N Flash Combustion - ash melting behaviour - trace elements - 5 - Moisture Content
TL Energopomiar Centralne Gliwice – Poland	- C-H-N Flash Combustion - Cl, F, S, Br by oxygen combustion + IC - Calorific Value - Moisture Content - trace elements - 5 - Major Elements after acid dissolution - B - ashing + DTA - A - nitric acid + ICP - Minor Elements after acid dissolution - Ash content - Volatile Matter
Umwelt Control Labor GmbH (UCL) Lünen – Germany	- Ash content - Moisture Content - Volatile Matter - Calorific Value - Major Elements after acid dissolution - Minor Elements after acid dissolution - Cl, F, S, Br by oxygen combustion + IC - Biomass/biodegradable - B - ashing (ASTM D6722 + XRF) - C-H-N Flash Combustion
Università politecnica delle Marche *IAAN- Area di Ingegneria Agraria* Ancona – Italy	- Calorific Value - Cl, F, S, Br by oxygen combustion + IC - Bulk Density - Major Elements after acid dissolution - Density of pellets and briquettes - Ash content - B - ashing (ASTM D6722 + XRF) - Moisture Content - C-H-N Flash Combustion - B - ashing + DTA - Bridging Properties - Particle dimesion & Particle size distribution - Volatile Matter - Minor Elements after acid dissolution - Durability - Biomass/biodegradable - A - nitric acid + ICP - trace elements - 5 - ash melting behaviour
Universität Stuttgart *Institut für Verfahrenstechnik und Dampfkesselwesen (IVD)* Stuttgart – Germany	- Major Elements after acid dissolution - B - ashing (ASTM D6722 + XRF) - C-H-N Flash Combustion - Biomass/biodegradable - Calorific Value - Particle dimesion & Particle size distribution - Ash content - Volatile Matter - Cl, F, S, Br by oxygen combustion + IC - Moisture Content - Minor Elements after acid dissolution - ash melting behaviour
University of Wolverhampton *School of Applied Sciences*	- C-H-N Flash Combustion - B - ashing + DTA

Affiliation	***Measured parameter***
Wolverhampton – United Kingdom	- Cl, F, S, Br by oxygen combustion + IC - Minor Elements after acid dissolution - B - ashing (ASTM D6722 + XRF) - A - nitric acid + ICP - trace elements - 5
Veolia Eau *Centre d'Analyses Environnementales (CAE) - Laboratoire CENTRAL* Saint-Maurice – France	- Biomass/biodegradable - Bulk Density - Ash content - Major Elements after acid dissolution - Moisture Content - Calorific Value - Particle dimesion & Particle size distribution - B - ashing (ASTM D6722 + XRF) - Density of pellets and briquettes - Minor Elements after acid dissolution - Volatile Matter - Cl, F, S, Br by oxygen combustion + IC - C-H-N Flash Combustion
Verein Deutscher Zementwerke e.V. *Forschungszentrum* Düsseldorf – Germany	- Major Elements after acid dissolution - Cl, F, S, Br by oxygen combustion + IC - trace elements - 5 - Moisture Content - Ash content - B - ashing (ASTM D6722 + XRF) - Biomass/biodegradable - Minor Elements after acid dissolution - Calorific Value - Volatile Matter - C-H-N Flash Combustion
VTT **Technical Research Centre of Finland** Espoo – Finland	- Cl, F, S, Br by oxygen combustion + IC - B - ashing + DTA - A - nitric acid + ICP - ash melting behaviour - Volatile Matter - Density of pellets and briquettes - Ash content - Major Elements after acid dissolution - C-H-N Flash Combustion - Moisture Content - Calorific Value - Minor Elements after acid dissolution - Biomass/biodegradable

3.2 Overview on submitted datasets

The number of analyzing laboratories for each method and parameter to be validated is listed hereafter. The parameters whose number of laboratories is below 4 have not to be considered from the statistical point of view.

Table 2 – Overview on submitted datasets

Technical specification	*Parameter Name*	*A*	*B*	*C*	*D*	*E*	*F*
TS - 15412 Al metal (nitric acid + ICP)	Metallic Al	3	3	3	3	3	
TS 15400 - Calorific Value	Calorific value	14	14	14	14	14	
TS 15402 - Volatile Matter	Volatile matter	12	12	12	12	10	
TS 15403 - Ash content	Ash content	16	16	16	16	16	
TS 15404 - ash melting behaviour	sintering T	3	3	3	3	3	
TS 15404 - ash melting behaviour	def. T	3	3	3	3	3	
TS 15404 - ash melting behaviour	hemisphere T	3	3	3	3	3	
TS 15404 - ash melting behaviour	flow T	3	3	3	3	3	
TS 15408 - Cl, F, S, Br by oxygen combustion + IC	S	14	14	14	14	14	
TS 15408 - Cl, F, S, Br by oxygen combustion + IC	Cl	14	14	14	14	14	
TS 15408 - Cl, F, S, Br by oxygen combustion + IC	Br	11	11	11	11	11	
TS 15408 - Cl, F, S, Br by oxygen combustion + IC	F	12	12	12	12	12	
TS 15410 - C-H-N Flash Combustion	C	13	13	13	13	13	
TS 15410 - C-H-N Flash Combustion	H	11	12	11	12	12	
TS 15410 - C-H-N Flash Combustion	N	11	12	11	12	12	
TS 15410 A - EN 13656 (aqua regia + HF + boric, MW)	Al	6	6	6	6	6	
TS 15410 A - EN 13656 (aqua regia + HF + boric, MW)	Ca	7	7	7	7	7	
TS 15410 A - EN 13656 (aqua regia + HF + boric, MW)	Fe	8	8	8	8	8	
TS 15410 A - EN 13656 (aqua regia + HF + boric, MW)	K	7	7	7	7	7	
TS 15410 A - EN 13656 (aqua regia + HF + boric, MW)	Mg	7	7	7	7	7	
TS 15410 A - EN 13656 (aqua regia + HF + boric, MW)	Na	7	7	7	7	7	
TS 15410 A - EN 13656 (aqua regia + HF + boric, MW)	P	6	6	6	6	6	
TS 15410 A - EN 13656 (aqua regia + HF + boric, MW)	Si	6	6	6	6	6	
TS 15410 A - EN 13656 (aqua regia + HF + boric, MW)	Ti	7	7	7	7	7	
TS 15410 B - ashing (ASTM D6722 + XRF)	Al	3	3	3	3	3	

Technical specification	*Parameter Name*	*A*	*B*	*C*	*D*	*E*	*F*
TS 15410 B - ashing (ASTM D6722 + XRF)	Ca	3	3	3	3	3	
TS 15410 B - ashing (ASTM D6722 + XRF)	Fe	3	3	3	3	3	
TS 15410 B - ashing (ASTM D6722 + XRF)	K	3	3	3	3	3	
TS 15410 B - ashing (ASTM D6722 + XRF)	Mg	3	3	3	3	3	
TS 15410 B - ashing (ASTM D6722 + XRF)	Na	3	3	3	3	3	
TS 15410 B - ashing (ASTM D6722 + XRF)	P	3	3	3	3	3	
TS 15410 B - ashing (ASTM D6722 + XRF)	Si	3	3	3	3	3	
TS 15410 B - ashing (ASTM D6722 + XRF)	Ti	3	3	3	3	3	
TS 15410 C - perchloric acid	Al	2	2	2	2	2	
TS 15410 C - perchloric acid	Ca	2	2	2	2	2	
TS 15410 C - perchloric acid	Fe	2	2	2	2	2	
TS 15410 C - perchloric acid	K	2	2	2	2	2	
TS 15410 C - perchloric acid	Mg	2	2	2	2	2	
TS 15410 C - perchloric acid	Na	2	2	2	2	2	
TS 15410 C - perchloric acid	P	2	2	2	2	2	
TS 15410 C - perchloric acid	Si	2	2	2	2	2	
TS 15410 C - perchloric acid	Ti	2	2	2	2	2	
TS 15410 D - EN 13657 (aqua regia, MW)	Al	4	4	4	4	4	
TS 15410 D - EN 13657 (aqua regia, MW)	Ca	4	4	4	4	4	
TS 15410 D - EN 13657 (aqua regia, MW)	Fe	4	4	4	4	4	
TS 15410 D - EN 13657 (aqua regia, MW)	K	4	4	4	4	4	
TS 15410 D - EN 13657 (aqua regia, MW)	Mg	4	4	4	4	4	
TS 15410 D - EN 13657 (aqua regia, MW)	Na	4	4	4	4	4	
TS 15410 D - EN 13657 (aqua regia, MW)	P	3	3	3	3	3	
TS 15410 D - EN 13657 (aqua regia, MW)	Si	3	3	3	3	3	
TS 15410 D - EN 13657 (aqua regia, MW)	Ti	3	3	3	3	3	
TS 15411 - Acidic digest	Al						8
TS 15411 - Acidic digest	Ca						9
TS 15411 - Acidic digest	Fe						10
TS 15411 - Acidic digest	K						9
TS 15411 - Acidic digest	Mg						9

Technical specification	*Parameter Name*	*A*	*B*	*C*	*D*	*E*	*F*
TS 15411 - Acidic digest	Na						9
TS 15411 - Acidic digest	P						7
TS 15411 - Acidic digest	Si						8
TS 15411 - Acidic digest	Ti						8
TS 15411 - Acidic digest	As						12
TS 15411 - Acidic digest	Ba						11
TS 15411 - Acidic digest	Be						10
TS 15411 - Acidic digest	Cd						12
TS 15411 - Acidic digest	Cr						11
TS 15411 - Acidic digest	Co						10
TS 15411 - Acidic digest	Cu						12
TS 15411 - Acidic digest	Hg						10
TS 15411 - Acidic digest	Mn						12
TS 15411 - Acidic digest	Mo						11
TS 15411 - Acidic digest	Ni						11
TS 15411 - Acidic digest	Pb						12
TS 15411 - Acidic digest	Sb						11
TS 15411 - Acidic digest	Se						11
TS 15411 - Acidic digest	Tl						11
TS 15411 - Acidic digest	V						12
TS 15411 - Acidic digest	Zn						12
TS 15411 - Hg by automatic analizer	Hg	2	2	2	2	2	
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	As	9	9	9	9	9	
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	Ba	9	9	9	9	9	
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	Be	7	7	7	7	7	
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	Cd	9	9	9	9	9	
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	Cr	9	9	9	9	9	
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	Co	8	8	8	8	8	
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	Cu	9	9	9	9	9	
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	Hg	8	8	8	8	8	
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	Mn	9	9	9	9	9	

Technical specification	Parameter Name	A	B	C	D	E	F
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	Mo	9	9	9	9	9	
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	Ni	8	8	8	8	8	
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	Pb	9	9	9	9	9	
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	Sb	8	8	8	8	8	
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	Se	8	8	8	8	8	
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	Tl	8	8	8	8	8	
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	V	9	9	9	9	9	
TS 15411 A - EN 13656 (aqua regia + HF + boric, MW)	Zn	9	9	9	9	9	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	As	1	1	1	1	1	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	Ba	1	1	1	1	1	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	Be	1	1	1	1	1	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	Cd	1	1	1	1	1	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	Cr	1	1	1	1	1	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	Co	1	1	1	1	1	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	Cu	1	1	1	1	1	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	Hg	1	1	1	1	1	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	Mn	1	1	1	1	1	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	Mo	1	1	1	1	1	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	Ni	1	1	1	1	1	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	Pb	1	1	1	1	1	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	Sb	1	1	1	1	1	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	Se	1	1	1	1	1	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	Tl	1	1	1	1	1	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	V	1	1	1	1	1	
TS 15411 B - ashing (ASTM D6722 + EN 13656)	Zn	1	1	1	1	1	
TS 15411 C - perchloric acid	As	2	2	2	2	2	
TS 15411 C - perchloric acid	Ba	2	2	2	2	2	
TS 15411 C - perchloric acid	Be	2	2	2	2	2	
TS 15411 C - perchloric acid	Cd	2	2	2	2	2	
TS 15411 C - perchloric acid	Cr	2	2	2	2	2	
TS 15411 C - perchloric acid	Co	2	2	2	2	2	

Technical specification	*Parameter Name*	*A*	*B*	*C*	*D*	*E*	*F*
TS 15411 C - perchloric acid	Cu	2	2	2	2	2	
TS 15411 C - perchloric acid	Hg	1	1	1	1	1	
TS 15411 C - perchloric acid	Mn	2	2	2	2	2	
TS 15411 C - perchloric acid	Mo	2	2	2	2	2	
TS 15411 C - perchloric acid	Ni	2	2	2	2	2	
TS 15411 C - perchloric acid	Pb	2	2	2	2	2	
TS 15411 C - perchloric acid	Sb	2	2	2	2	2	
TS 15411 C - perchloric acid	Se	2	2	2	2	2	
TS 15411 C - perchloric acid	Tl	2	2	2	2	2	
TS 15411 C - perchloric acid	V	2	2	2	2	2	
TS 15411 C - perchloric acid	Zn	2	2	2	2	2	
TS 15411 D - EN 13657 (aqua regia, MW)	As	3	3	3	3	3	
TS 15411 D - EN 13657 (aqua regia, MW)	Ba	3	3	3	3	3	
TS 15411 D - EN 13657 (aqua regia, MW)	Be	3	3	3	3	3	
TS 15411 D - EN 13657 (aqua regia, MW)	Cd	3	3	3	3	3	
TS 15411 D - EN 13657 (aqua regia, MW)	Cr	3	3	3	3	3	
TS 15411 D - EN 13657 (aqua regia, MW)	Co	4	3	3	3	3	
TS 15411 D - EN 13657 (aqua regia, MW)	Cu	4	4	4	4	4	
TS 15411 D - EN 13657 (aqua regia, MW)	Hg	3	3	3	3	3	
TS 15411 D - EN 13657 (aqua regia, MW)	Mn	4	4	4	4	4	
TS 15411 D - EN 13657 (aqua regia, MW)	Mo	3	3	3	3	3	
TS 15411 D - EN 13657 (aqua regia, MW)	Ni	4	3	4	3	4	
TS 15411 D - EN 13657 (aqua regia, MW)	Pb	4	3	4	4	4	
TS 15411 D - EN 13657 (aqua regia, MW)	Sb	3	3	3	3	3	
TS 15411 D - EN 13657 (aqua regia, MW)	Se	3	3	3	3	3	
TS 15411 D - EN 13657 (aqua regia, MW)	Tl	3	3	3	3	3	
TS 15411 D - EN 13657 (aqua regia, MW)	V	3	3	3	3	3	
TS 15411 D - EN 13657 (aqua regia, MW)	Zn	4	4	4	4	4	
TS 15414 - Moisture Content	Moisture	14	14	14	14	14	
TS 15415 - Particle dimesion & Particle size distribution	3.15 mm	3	4	3	4	4	
TS 15415 - Particle dimesion & Particle size distribution	1.6 mm	3	4	3	4	4	

Technical specification	*Parameter Name*	*A*	*B*	*C*	*D*	*E*	*F*
TS 15415 - Particle dimesion & Particle size distribution	800 um	3	4	3	4	4	
TS 15415 - Particle dimesion & Particle size distribution	400 um	3	4	3	4	4	
TS 15415 - Particle dimesion & Particle size distribution	200 um	3	4	3	4	4	
TS 15415 - Particle dimesion & Particle size distribution	<200 um	3	4	3	4	4	
TS 15440-B	XB: Biomass		9		9	9	
TS 15440-B	XNB: Non Biomass		8		8	8	
TS 15440-D	XB: Biomass		3		4	4	
TS 15440-D	XNB: Non Biomass		2		3	3	
TS 15440-E	XB: Biomass		3		4	4	

3.3 Results of the validation exercises: physical parameters

3.3.1 TS 15400 (Calorific value)

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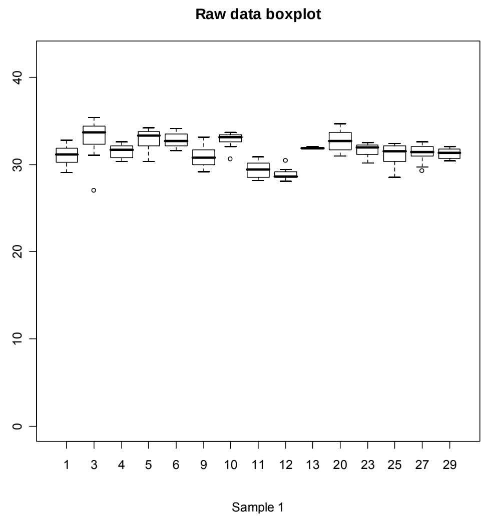
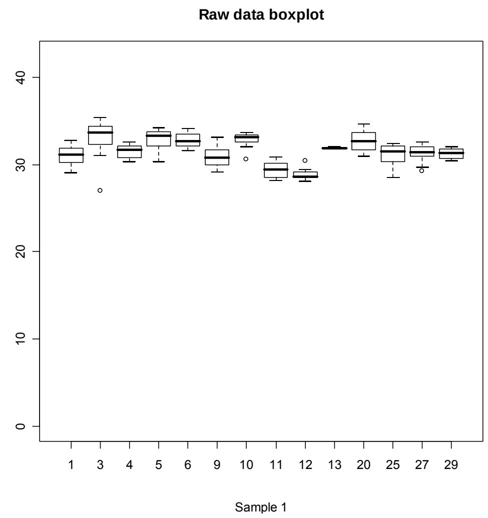
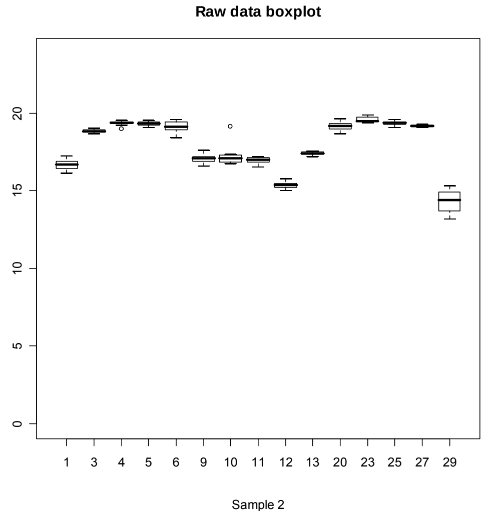
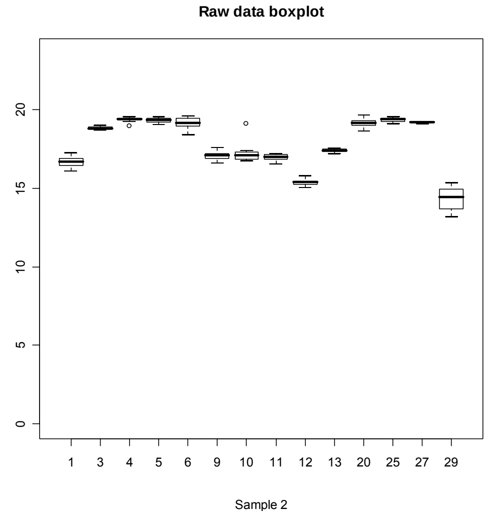
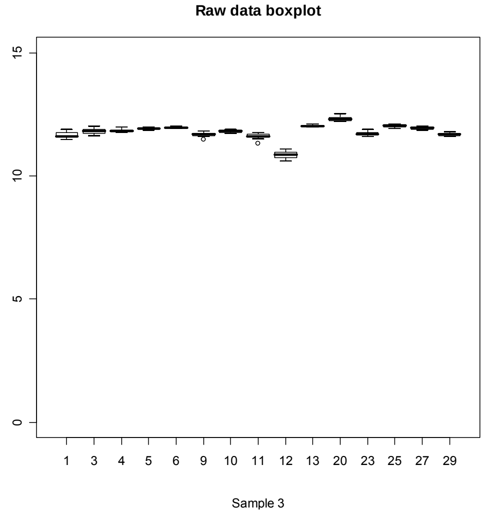
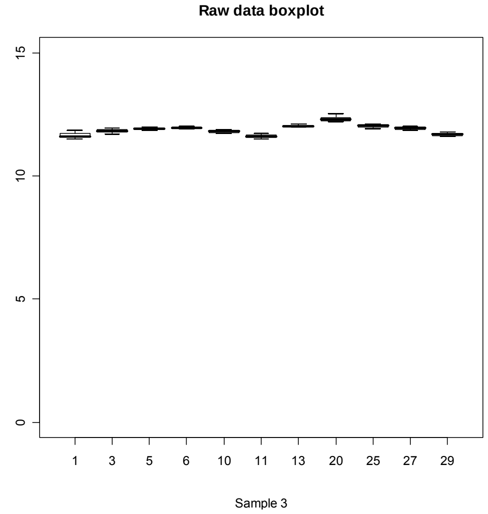
	
	
	

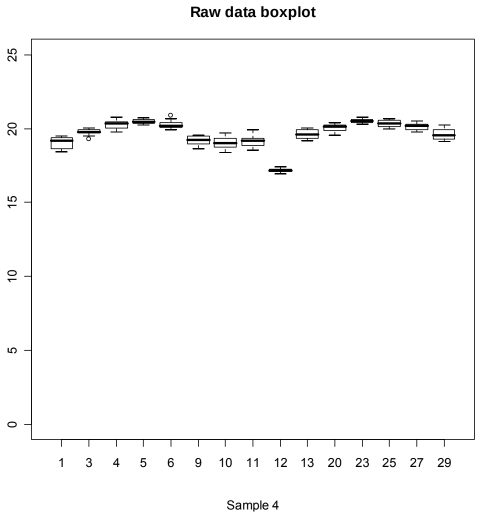
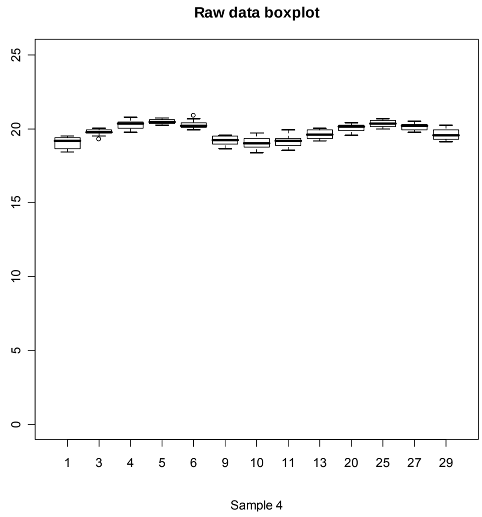
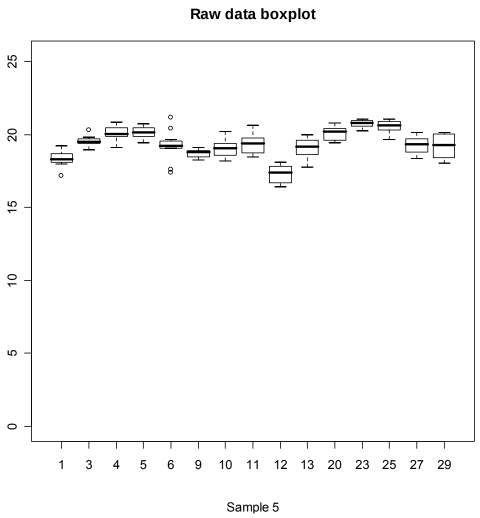
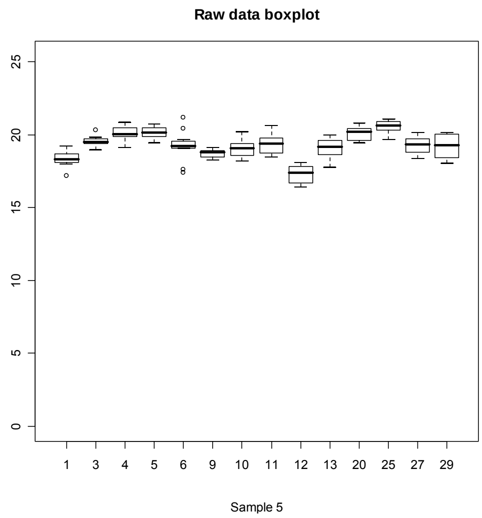
Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	All results are expressed in MJ/kg

Table 3: Validation of TS 15400 (Calorific value) - General Averages. Results are expressed in MJ/kg.

Level	m_j	*Number of valid results*
1	31.55	174
2	18.07	172
3	11.88	138
4	19.82	168
5	19.38	180

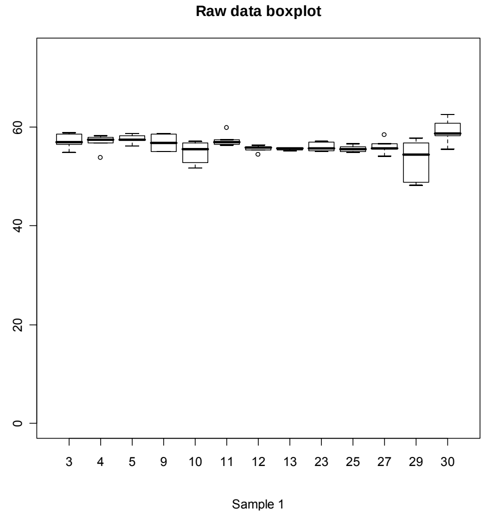
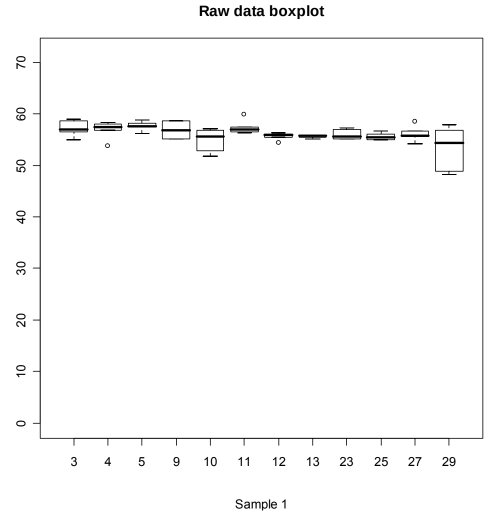
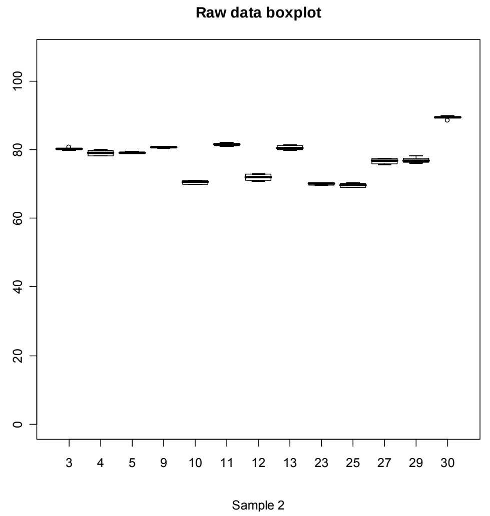
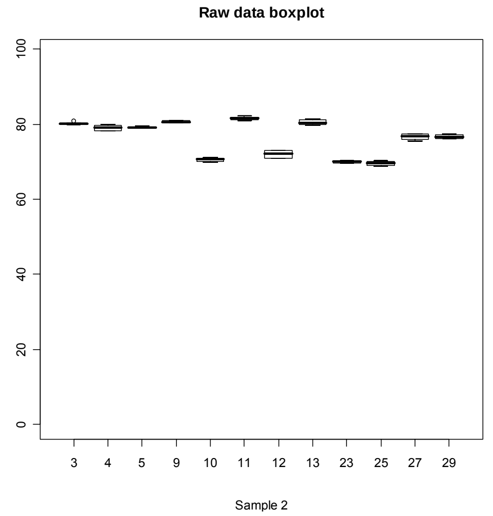
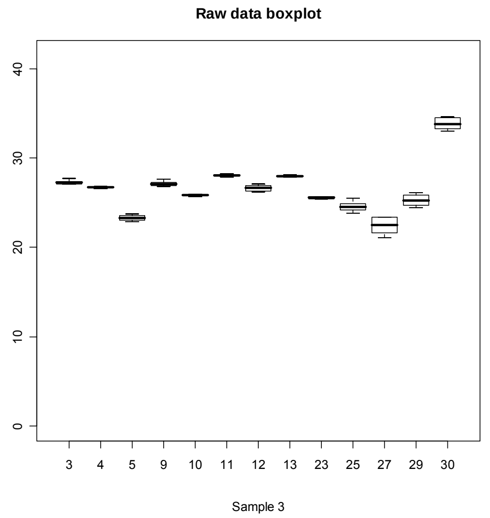
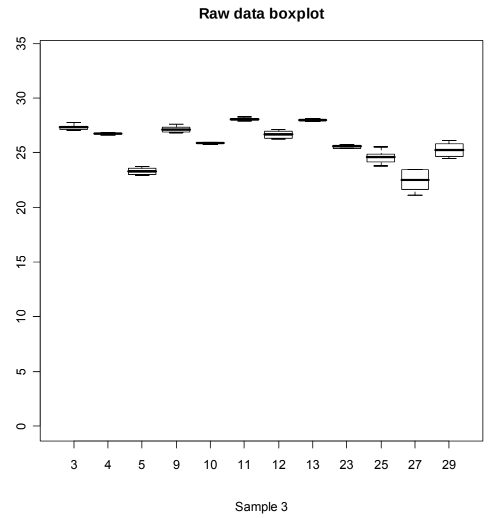
Table 4: Validation of TS 15400 (Calorific value) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	256.35	14	36.62	14	161.26	145	2052	1044	90
2	370.39	14	1.30	14	5.70	143	2008	1024	89.2
3	5.24	11	0.06	17	0.46	139	1920	984	87.6
4	46.14	13	3.63	15	10.62	145	2052	1044	90
5	132.31	14	8.03	15	47.76	150	2160	1080	90

Table 5: Validation of TS 15404 (Calorific value) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}), for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in MJ/kg.

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	1.16	0.5	1.05	2.94	1.57	4.396
2	1.52	0.09	0.2	0.56	1.53	4.284
3	0.18	0.01	0.06	0.168	0.19	0.532
4	0.51	0.17	0.27	0.756	0.58	1.624
5	0.86	0.19	0.56	1.568	1.03	2.884

3.3.2 TS 15402 (Volatile matter)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	

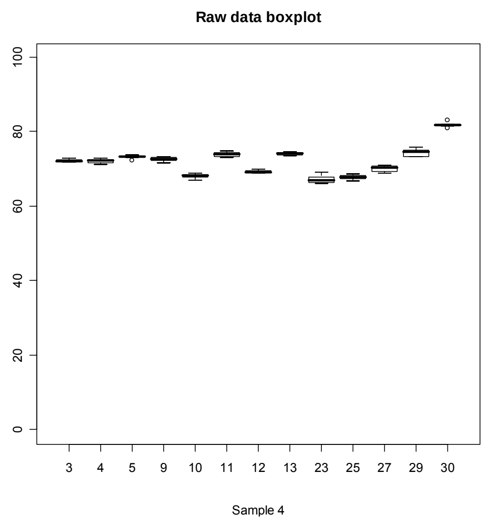
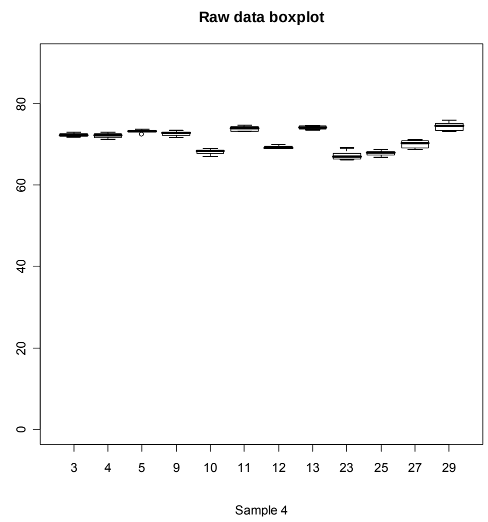
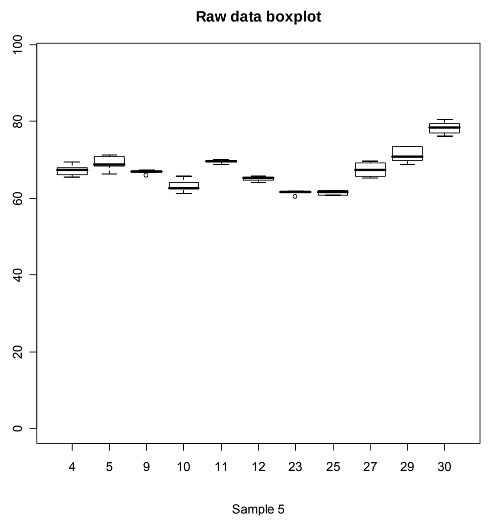
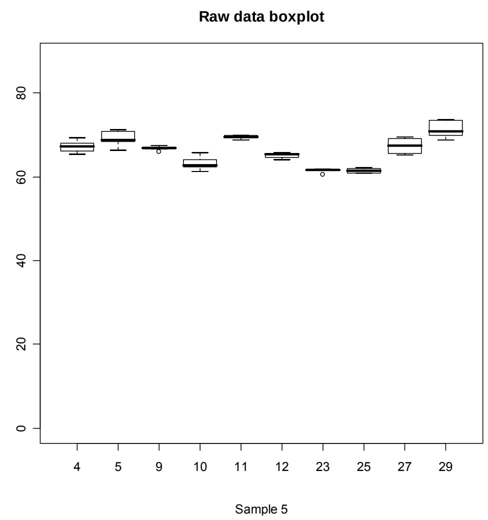
Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	Results are expressed in percentage of weight (%)

Table 6: TS 15402 (Volatile matter) - General Averages. Results are expressed in percentage of weight (%).

Level	m_j	*Number of valid results*
1	56.10	69
2	76.40	70
3	25.90	72
4	71.21	69
5	66.23	60

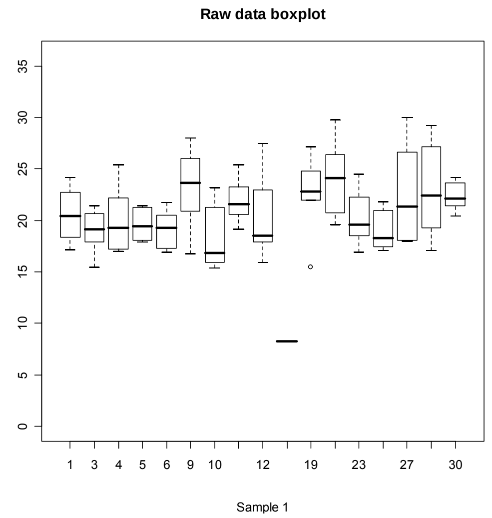
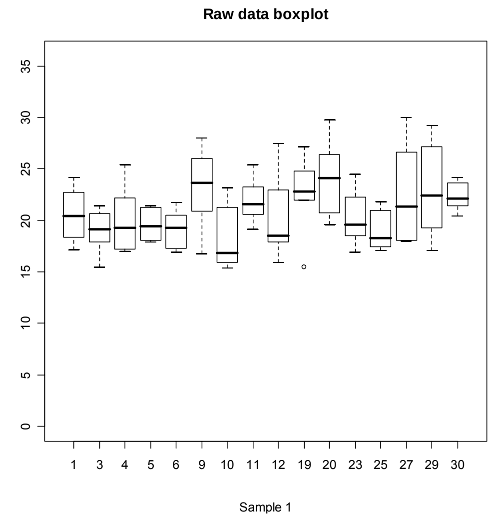
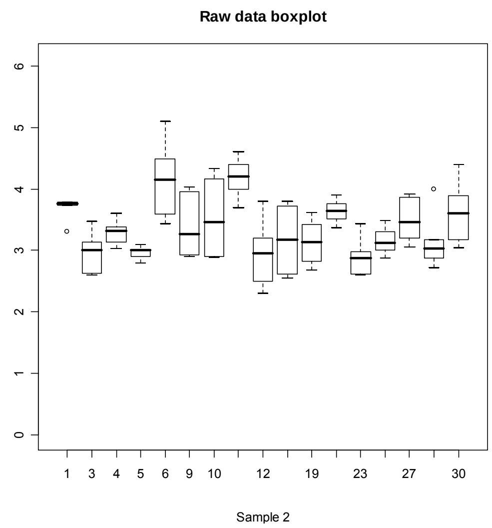
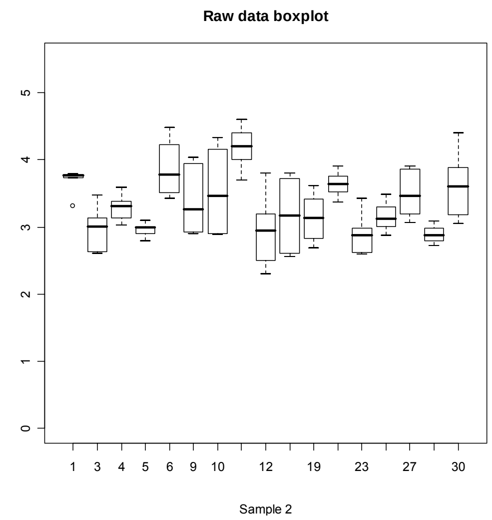
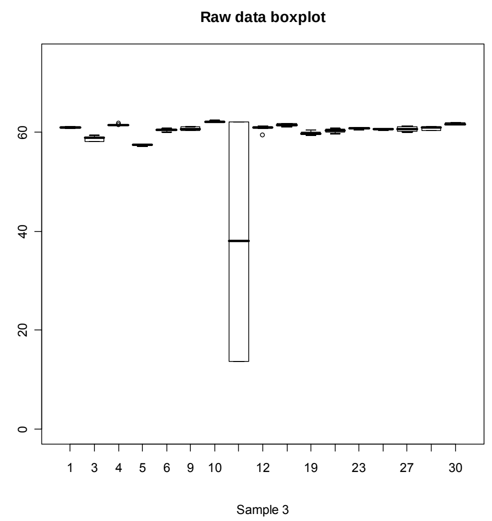
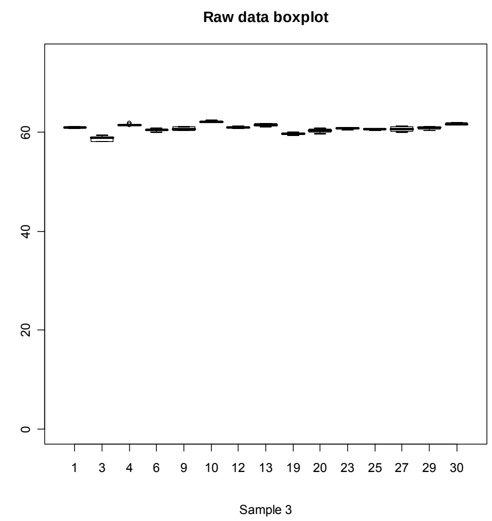
Table 7: TS 15402 (Volatile matter) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

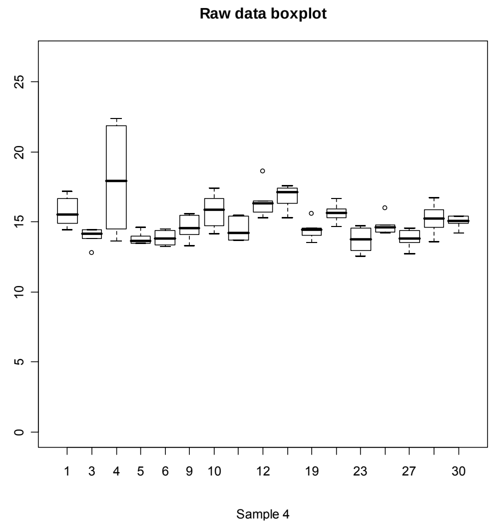
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	91.17	11	20.91	11	154.74	46	405	207	36
2	1394.91	11	16.99	12	3.43	46	412	208	35.5
3	208.78	11	2.55	12	8.97	48	432	216	36
4	452.15	11	5.93	11	22.66	46	405	207	36
5	618.69	9	21.63	14	58.62	48	432	216	36

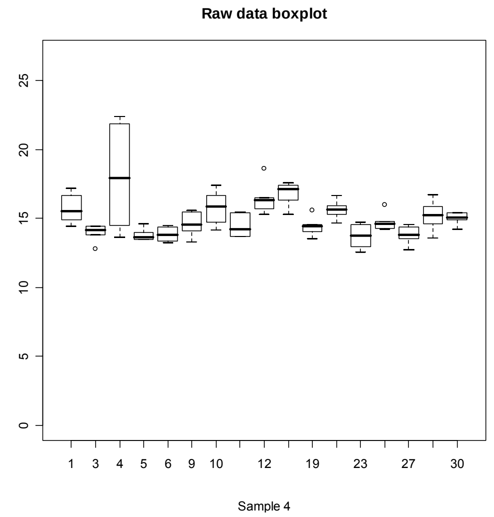
Table 8: TS 15402 (Volatile matter) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}), for repeatability (s_{rj}) and for reproducibility (s_{R1j}). Results are expressed in percentage of weight (%).

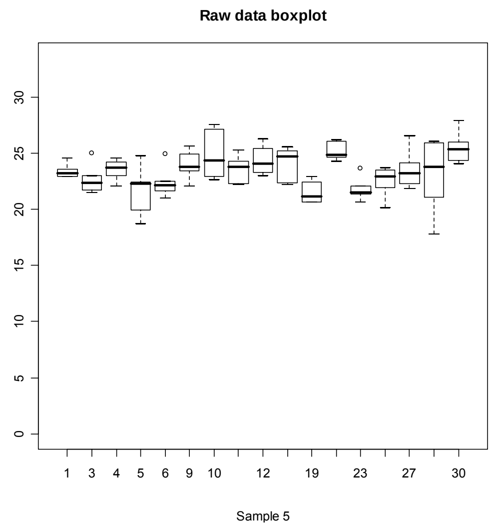
Level	s_{Lj}	s_{Hj}	s_{rj}	r	s_{Rj}	R
1	NA	NA	1.83	5.124	1.83	5.124
2	4.64	0.68	0.27	0.756	4.65	13.02
3	1.77	0.09	0.43	1.204	1.82	5.096
4	2.66	0.12	0.7	1.96	2.75	7.7
5	3.02	0.35	1.11	3.108	3.22	9.016

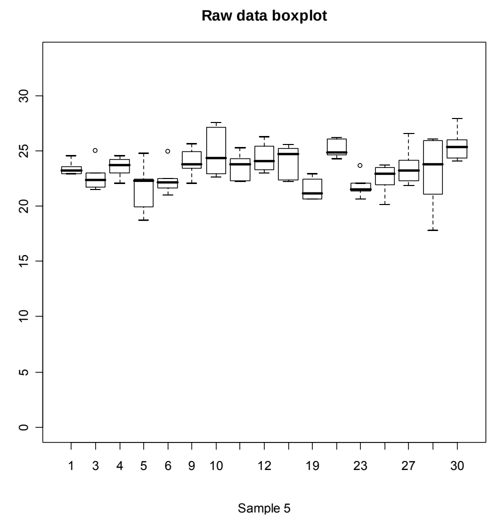
3.3.3 TS 15403 (Ash content)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	









Results are expressed in percentage of weight (%) d.m.

Table 9: TS 15403 (Ash content) - General Averages. Results are expressed in percentage of weight (%) d.m..

Level	m_j	*Number of valid results*
1	20.98	96
2	3.38	102
3	60.80	90
4	14.89	96
5	23.39	102

Table 10: TS 15403 (Ash content) - Sums of squares and degrees of freedom, for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

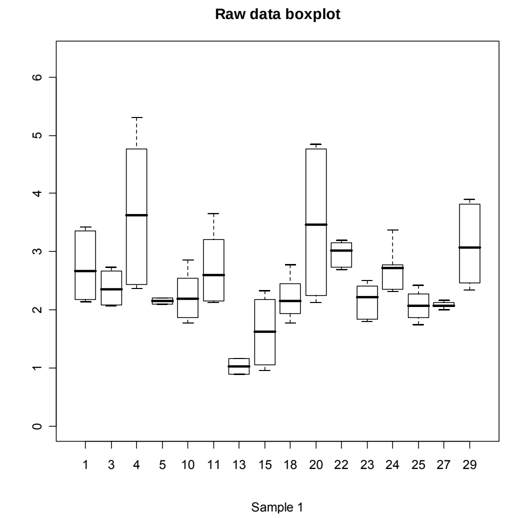
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	293.38	15	265.48	18	573.83	62	576	288	48
2	14.78	16	10.48	17	4.03	68	612	306	51
3	55.32	14	4.80	19	4.80	68	612	306	51
4	87.05	15	15.83	18	39.30	68	612	306	51
5	136.37	16	43.12	17	156.00	68	612	306	51

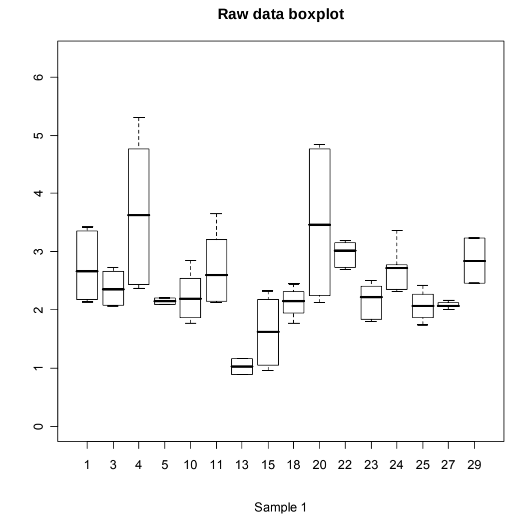
Table 11: TS 15403 (Ash content) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in percentage of weight (%) d.m.

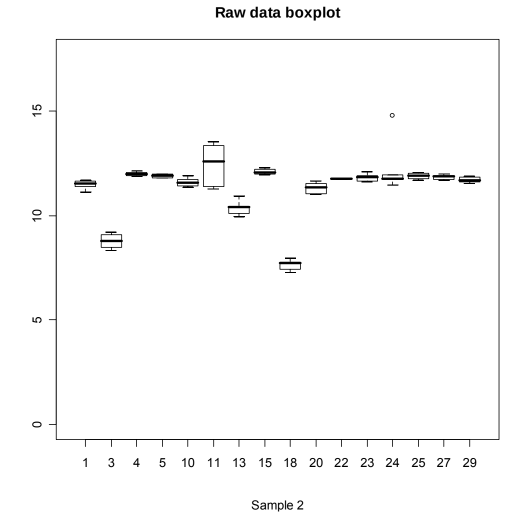
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	0.83	1.44	3.04	8.512	3.15	8.82
2	0.23	0.43	0.24	0.672	0.33	0.924
3	0.73	0.26	0.27	0.756	0.78	2.184
4	0.87	0.33	0.76	2.128	1.16	3.248
5	1	0.28	1.51	4.228	1.81	5.068

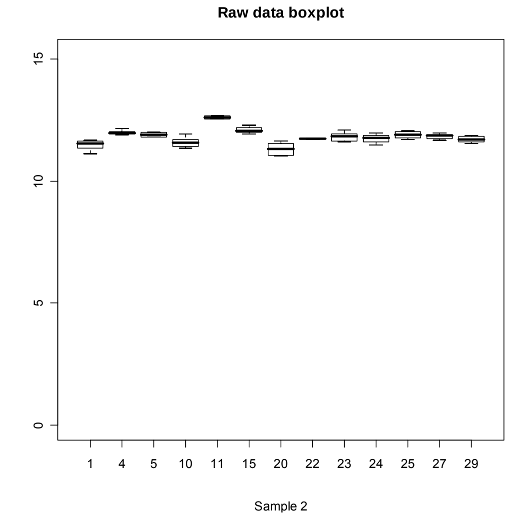
3.3.4 TS 15414 (Moisture content)

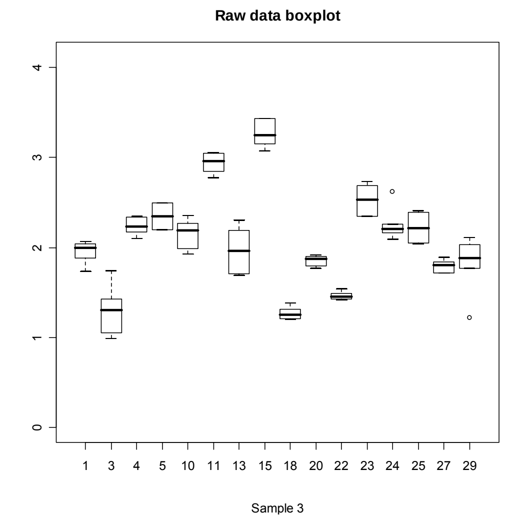
Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*

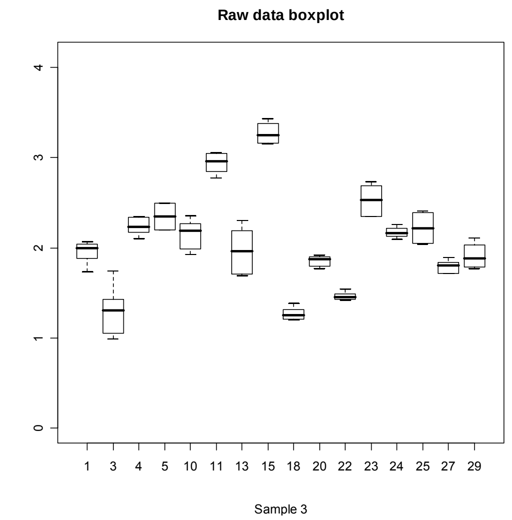


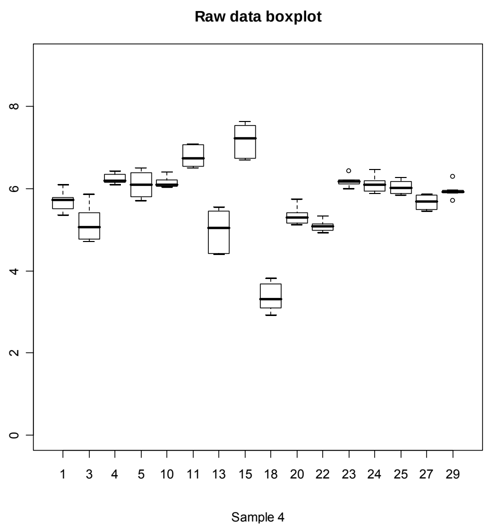


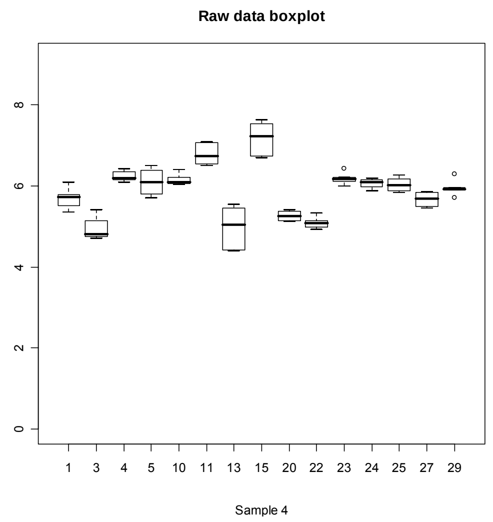


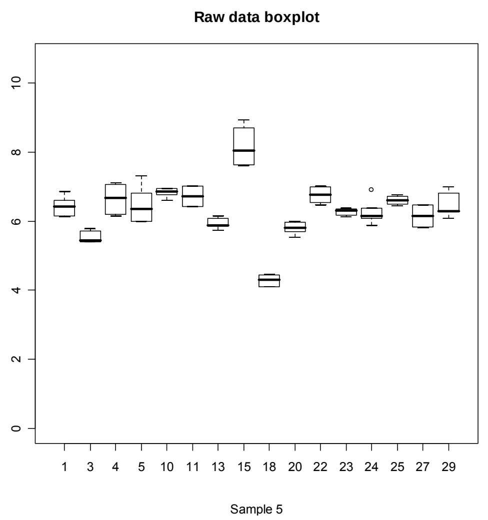


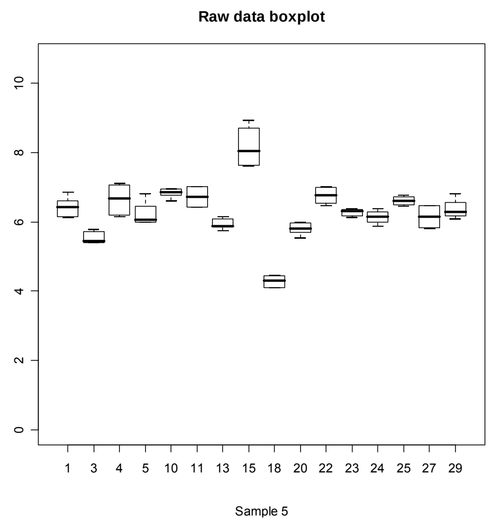












Results are expressed in percentage of weight (%) on a wet basis

Table 12: TS 15414 (Moisture content) - General Averages. Results are expressed in percentage of weight (%) on a wet basis

Level	m_j	*Number of valid results*
1	2.48	50
2	11.79	50
3	2.13	50
4	5.96	52
5	6.49	50

Table 13: TS 15414 (Moisture content) - Sums of squares and degrees of freedom, for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	5.66	8	5.78	9	1.15	32	292	146	25
2	2.72	8	0.53	9	2.75	32	284	146	26
3	8.71	8	0.54	9	0.22	32	284	146	26
4	10.23	8	0.85	9	0.64	34	304	154	26.5
5	3.09	8	1.68	9	0.67	32	284	146	26

Table 14: TS 15414 (Moisture content) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in percentage of weight (%) on a wet basis

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	0.11	0.47	0.19	0.532	0.22	0.616
2	NA	NA	0.29	0.812	0.29	0.812
3	0.43	0.14	0.08	0.224	0.44	1.232
4	0.45	0.16	0.14	0.392	0.47	1.316
5	0.18	0.25	0.14	0.392	0.23	0.644

3.4 Results of the validation exercises: Biogenic content

3.4.1 TS 15440 B (Biomass content)

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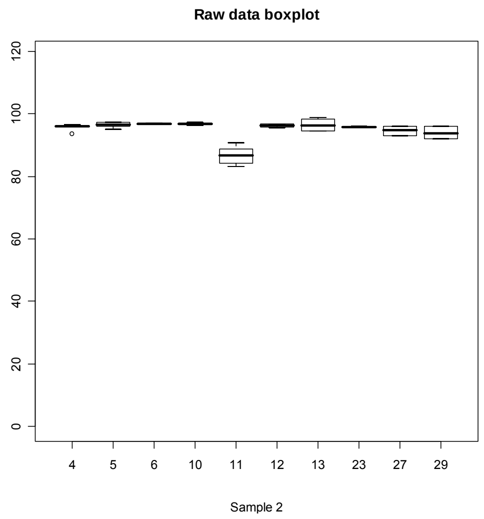
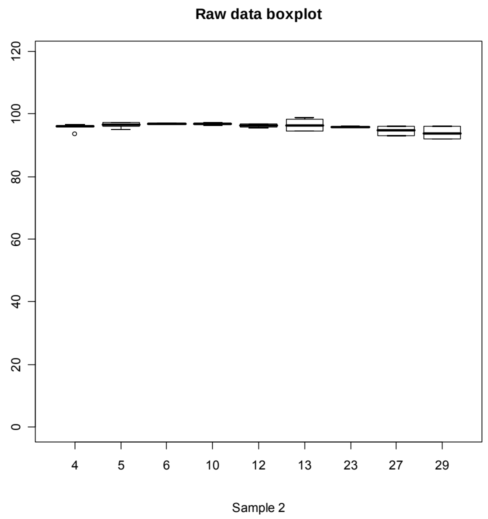
<tr><td>
</td><td>
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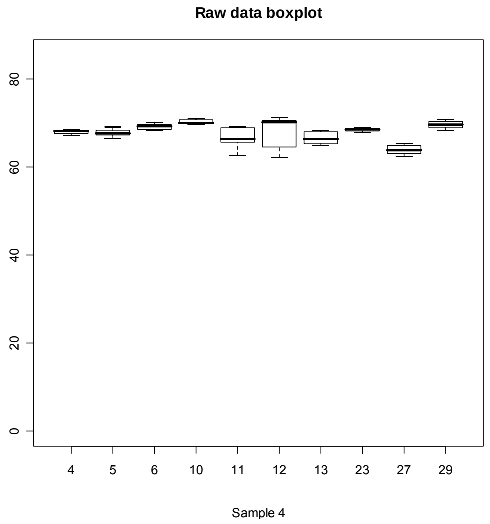
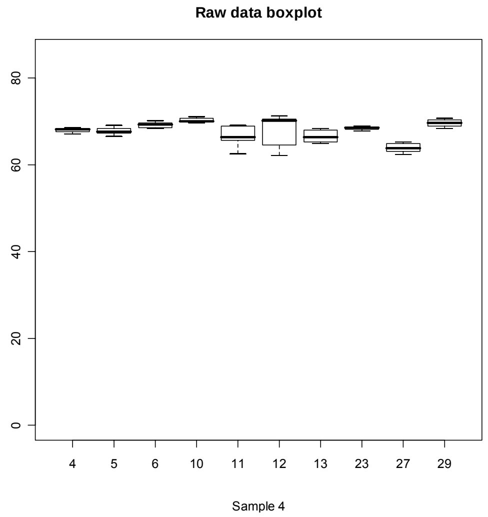
<tr><td>
</td><td>
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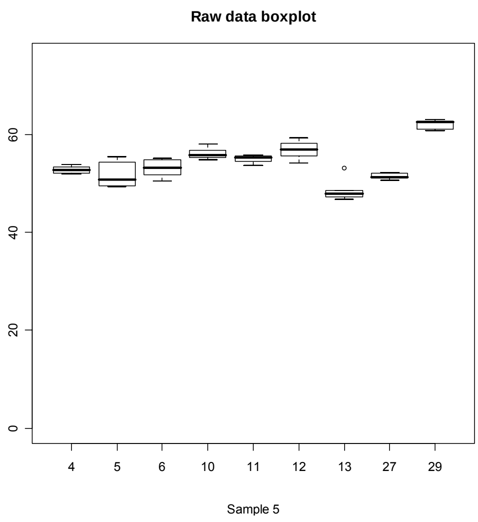
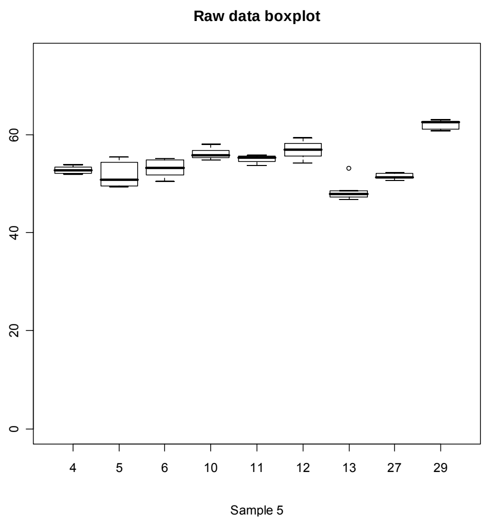




Results are expressed in percentage of weight (%) d.m.

Table 15: TS 15440 B (Biomass content) - General Averages. Results are expressed in percentage of weight (%) d.m.

Level	m_j	*Number of valid results*
2	95.92823	54
4	67.79178	57
5	54.17492	54

Table 16: TS 15440 B (Biomass content) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS_{.Hj}$, ni_{Hj}) and for repeatability ($SS_{.rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
2	47.05	8	41.17	11	21.43	34	324	162	27
4	182.97	9	34.51	9	96.14	38	333	171	30
5	737.42	8	53.39	11	65.13	40	360	180	30

Table 17: TS 15440 B (Biomass content) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in percentage of weight (%) d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
2	0.49	1.13	0.79	2.212	0.93	2.604
4	1.7	0.66	1.59	4.452	2.33	6.524
5	3.58	1.09	1.28	3.584	3.8	10.64

3.4.2 TS 15440 B (Non-biomass content)

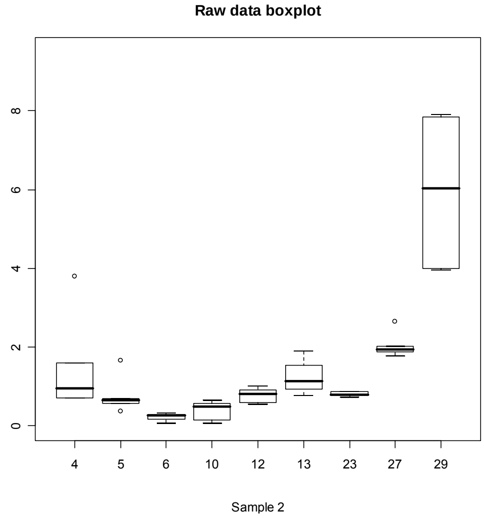
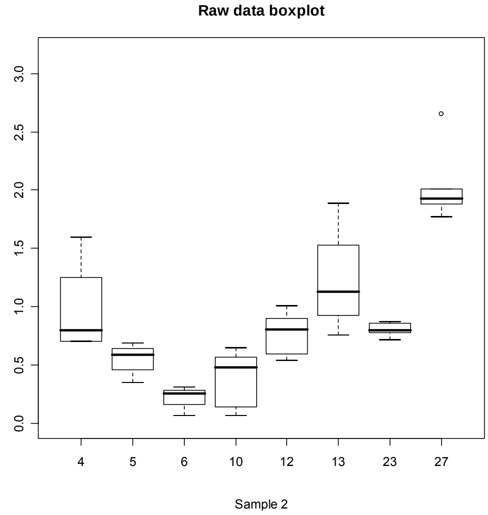
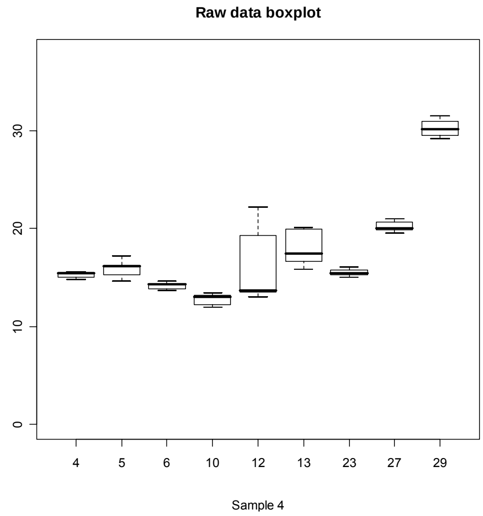
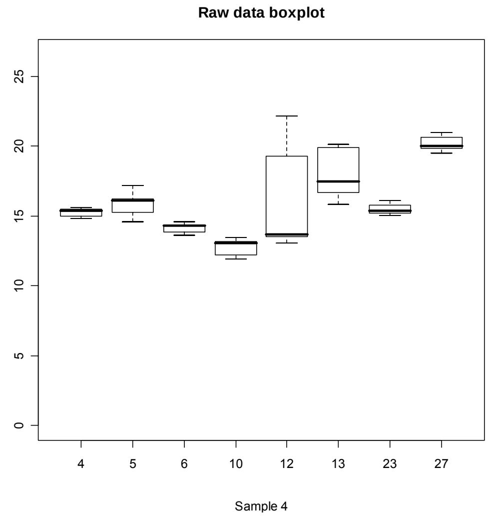
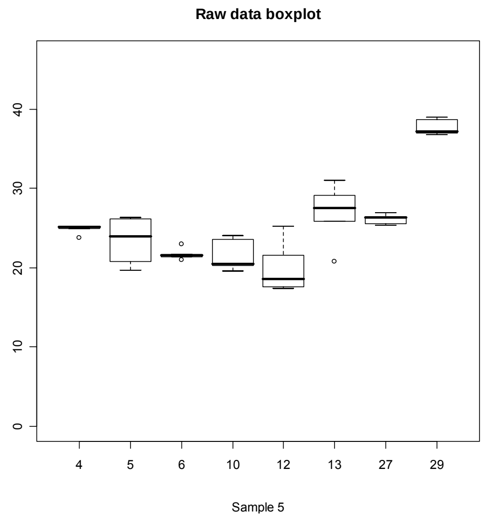
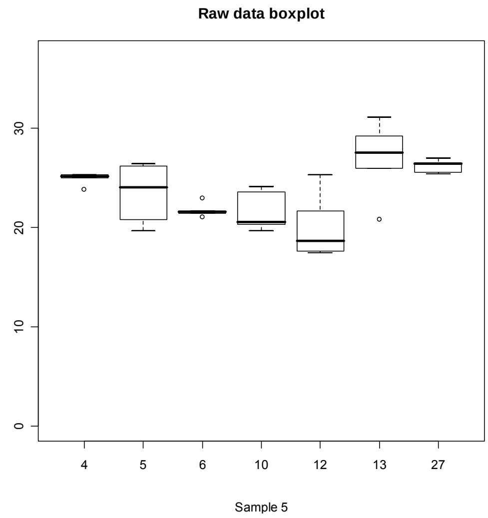
Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	
	Results are expressed in percentage of weight (%) d.m.

Table 18: (Non-biomass content) - General Averages. Results are expressed in percentage of weight (%) d.m.

Level	m_j	*Number of valid results*
2	0.93	41
4	15.99	45
5	23.52	42

Table 19: (Non-biomass content) - Sums of squares and degrees of freedom, for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
2	11.27	7	1.34	8	1.92	25	221	113	21.167
4	211.61	7	5.64	7	92.82	30	261	135	24
5	253.06	6	75.76	9	103.15	32	288	144	24

Table 20: (Non-biomass content) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in percentage of weight (%) d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	r	s_{Rj}	R
2	0.53	0.19	0.28	0.784	0.6	1.68
4	NA	NA	1.76	4.928	1.76	4.928
5	2.14	1.4	1.8	5.04	2.8	7.84

3.4.3 TS 15440-D (Biomass content)

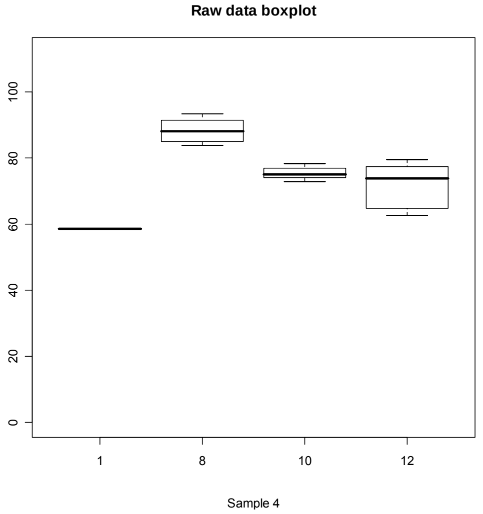
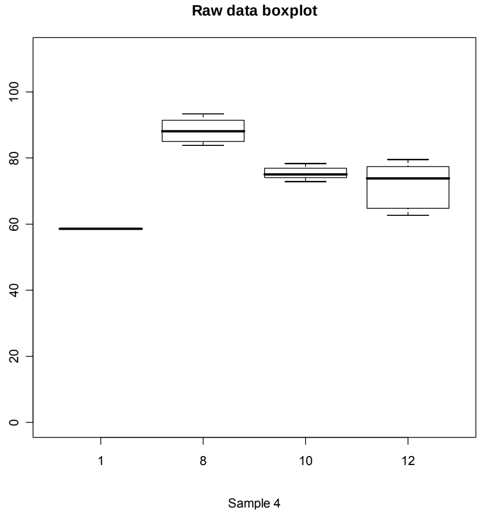
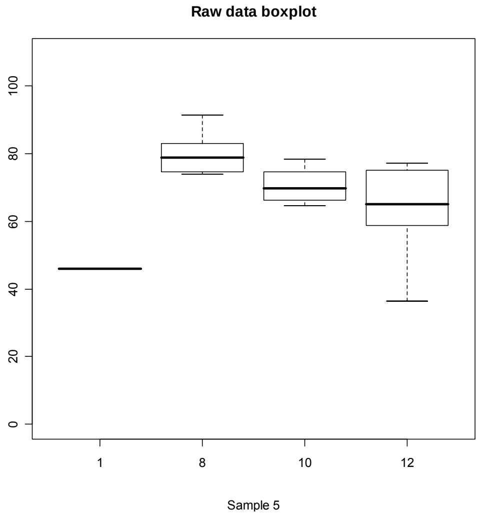
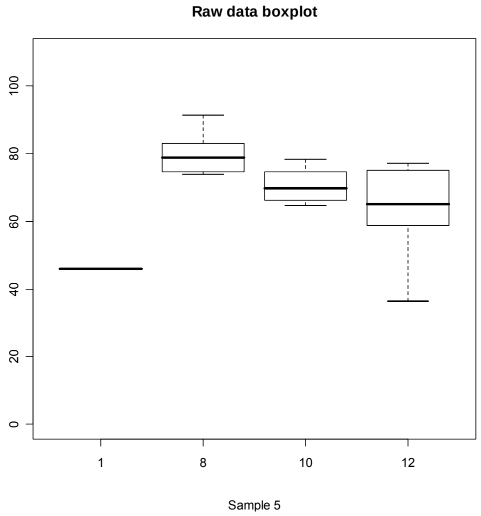
Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	N.B. Data set to small for meaningful outlier testing *Results are expressed in percentage of weight (%) d.m as a percentage by calorific value.*

Table 21: TS 15440-D (Biomass content) - General Averages. Results are expressed in percentage of weight (%) d.m as a percentage by calorific value.

Level	m_j	*Number of valid results*
2	99.03	7
4	77.53	19
5	69.92	19

Table 22: TS 15440-D (Biomass content) - Sums of squares and degrees of freedom, for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

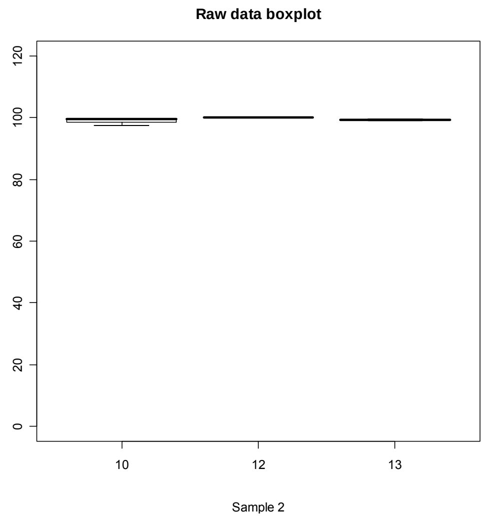
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
2	39.6	1	0	5	0	0	37	19	4
4	1258.6	3	48.7	3	282.2	12	109	55	10
5	1497.9	3	245.0	3	1206.9	12	109	55	10

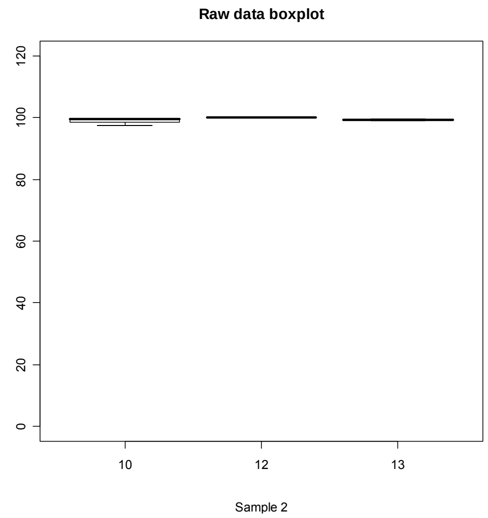
Table 23: TS 15440-D (Biomass content) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in percentage of weight (%) d.m as a percentage by calorific value.

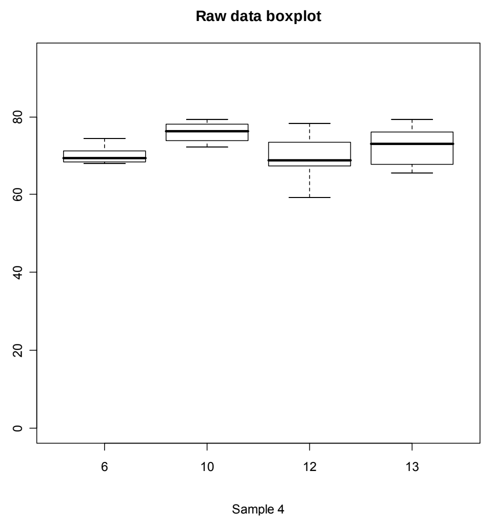
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
2	NA	NA	NA		NA	
4	NA	NA	4.85	13.58	4.85	13.58
5	NA	NA	10.03	28.084	10.03	28.084

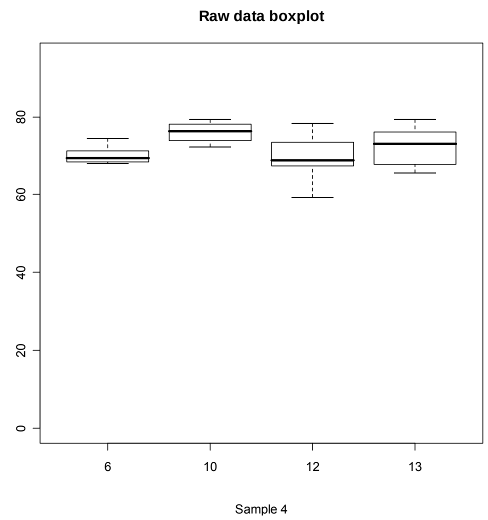
3.4.4 TS 15440-E (Biomass content)

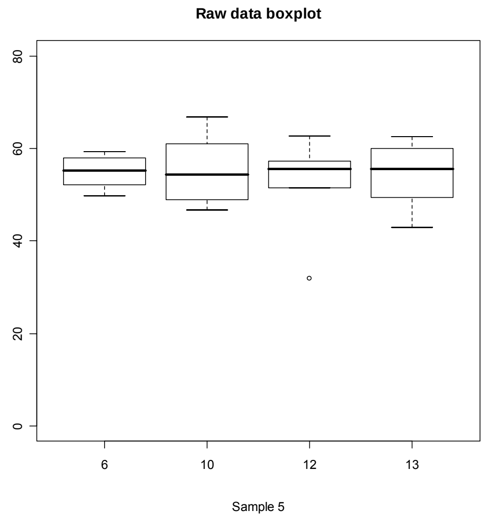
Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*

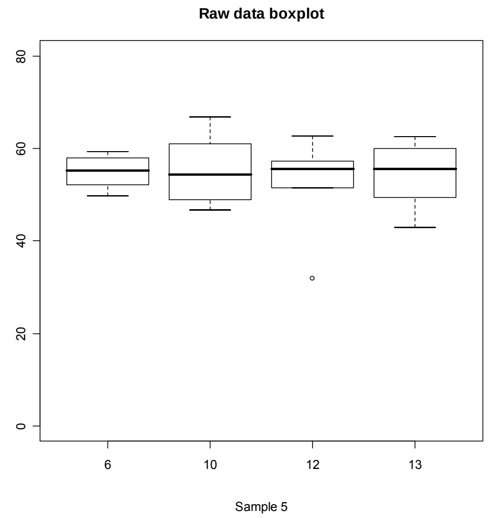












	N.B. Data set to small for meaningful outlier testing *Results are expressed in percentage of weight (%) by carbon content*

Table 24: TS 15440-E (Biomass content) - General Averages. Results are expressed in percentage of weight (%) by carbon content

Level	m_j	*Number of valid results*
2	99.47	16
4	71.94	24
5	54.31	24

Table 25: TS 15440-E (Biomass content) - Sums of squares and degrees of freedom, for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
2	2.84	2	1.05	5	2.06	8	88	44	8
4	160.84	3	123.21	4	284.20	16	144	72	12
5	30.96	3	446.47	4	752.73	16	144	72	12

Table 26: TS 15440-E (Biomass content) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in percentage of weight (%) by carbon content

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
2	NA	NA	0.51	1.428	0.51	1.428
4	1.95	2.08	4.21	11.788	4.64	12.992
5	NA	4.64	6.86	19.208	6.86	19.208

3.5 Results of the validation exercises: Chemical parameters

3.5.1 TS 15407 (CHN Flash Combustion: Carbon)

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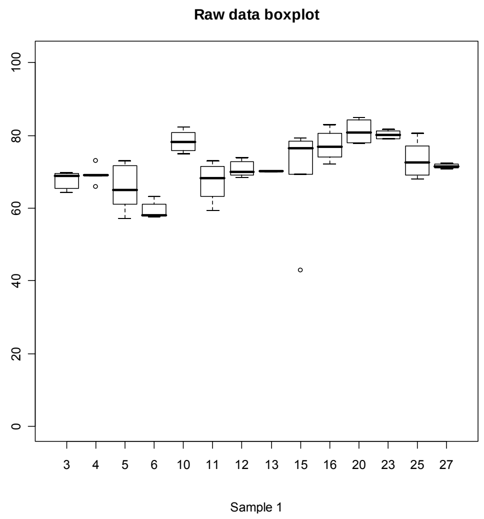

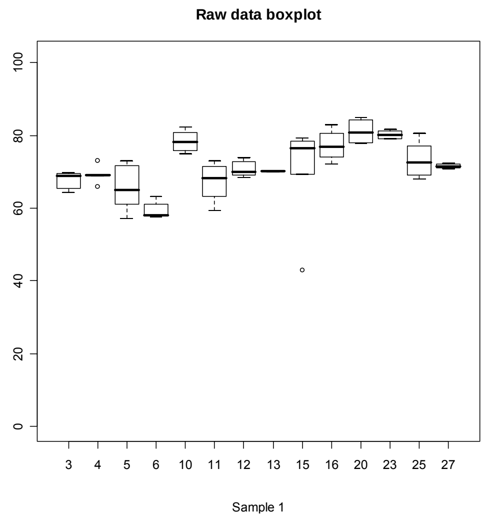

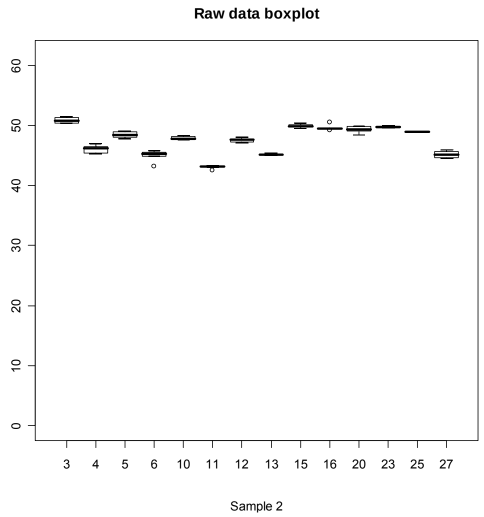

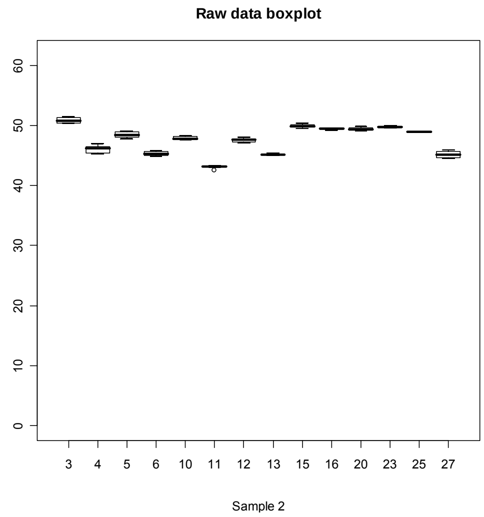

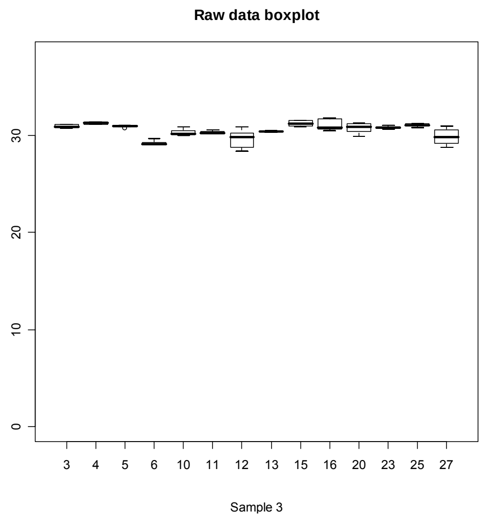

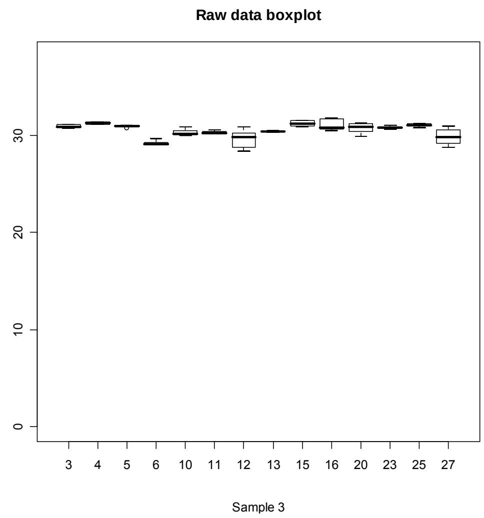

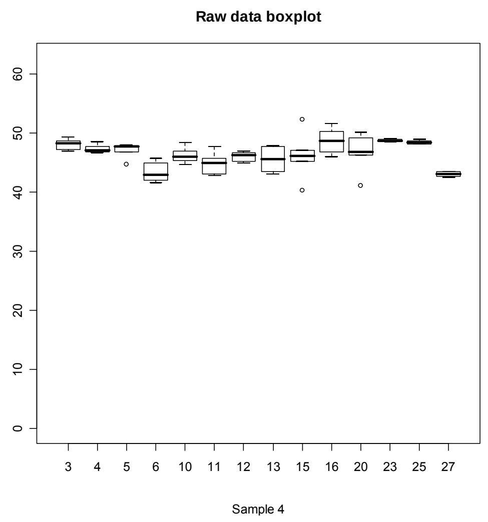

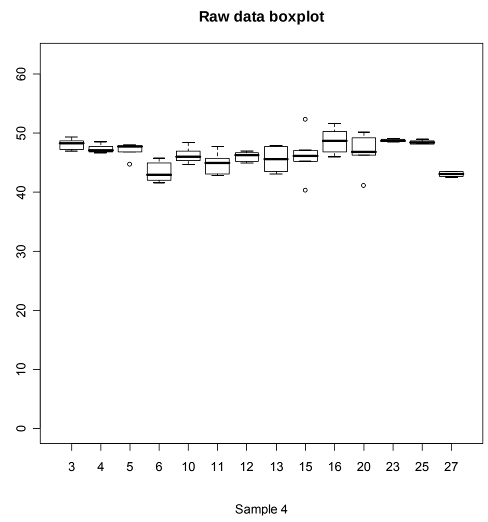

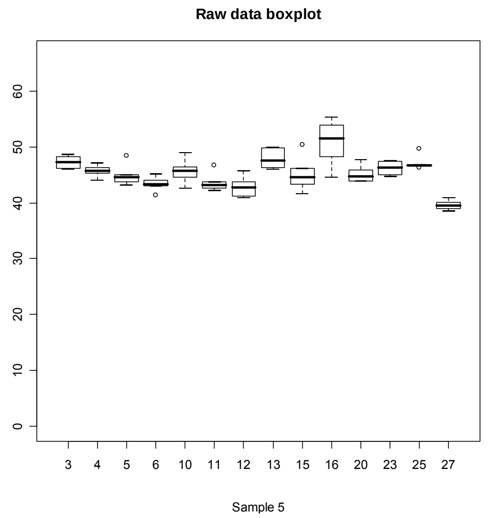

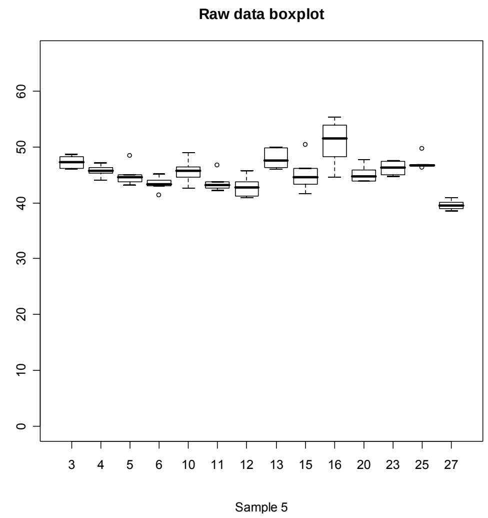

Results are expressed in % d.m.

Table 27: TS 15407 (CHN Flash Combustion: Carbon) - General Averages. Results are expressed in % d.m..

Level	m_j	*Number of valid results*
1	71.6	81
2	47.7	80
3	30.5	84
4	46.4	84
5	45.4	84

Table 28: TS 15407 (CHN Flash Combustion: Carbon) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	5.66	8	5.78	9	1.15	32	292	146	25
2	2.72	8	0.53	9	2.75	32	284	146	26
3	8.71	8	0.54	9	0.22	32	284	146	26
4	10.23	8	0.85	9	0.64	34	304	154	26.5
5	3.09	8	1.68	9	0.67	32	284	146	26

Table 29: TS 15407 (CHN Flash Combustion: Carbon) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in % d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	r	s_{Rj}	R
1	NA	NA	5.12	14.336	5.12	14.336
2	2.31	0.4	0.26	0.728	2.33	6.524
3	NA	NA	0.43	1.204	0.43	1.204
4	1.52	1.09	1.63	4.564	2.23	6.244
5	2.42	1.18	1.71	4.788	2.97	8.316

3.5.2 TS 15407 (CHN Flash Combustion: Hydrogen)

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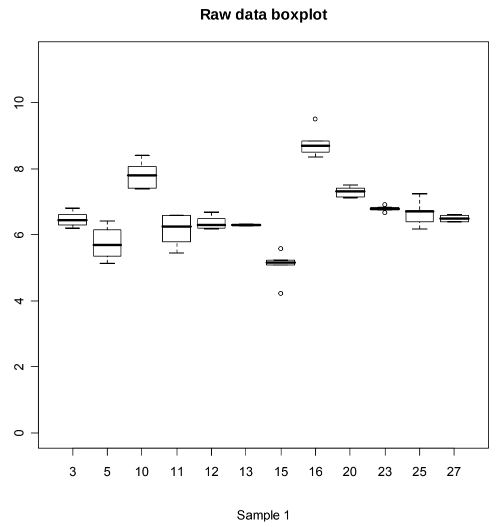

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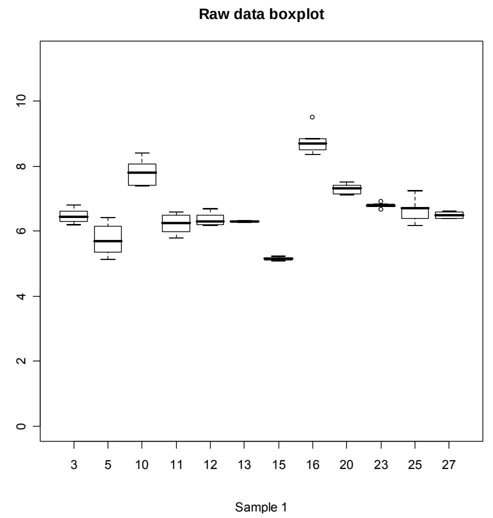

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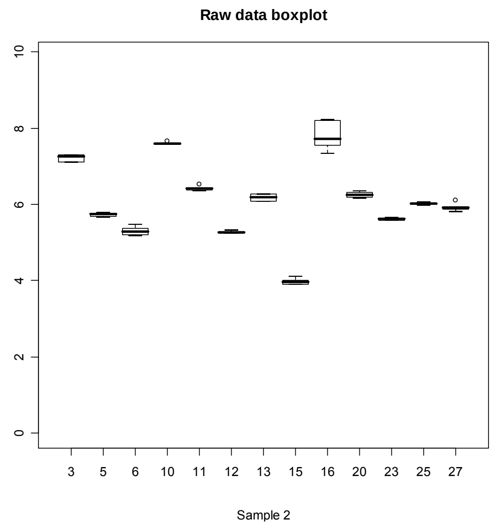

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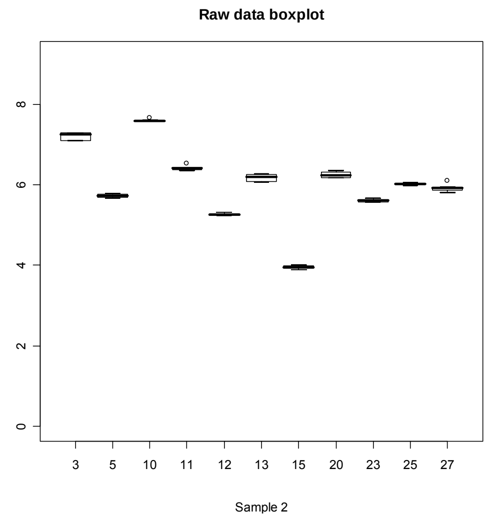

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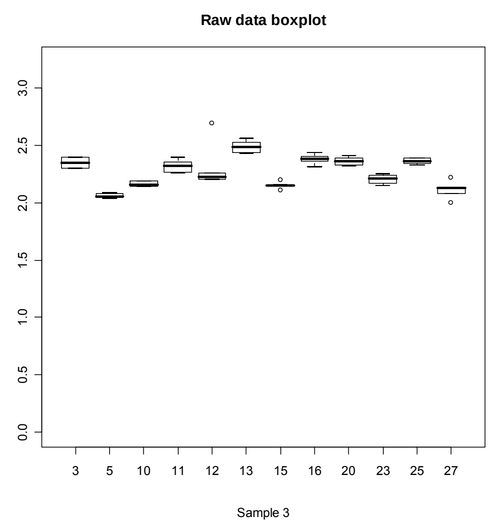

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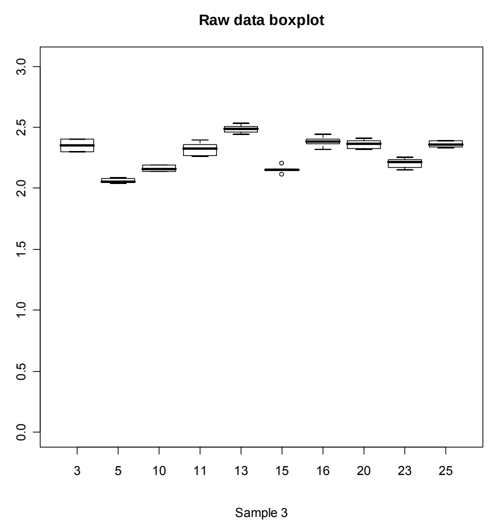

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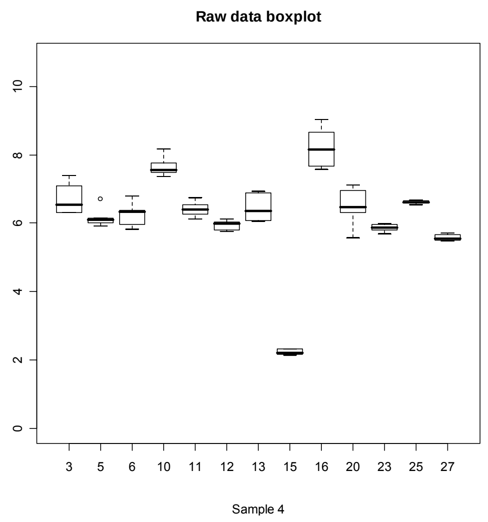

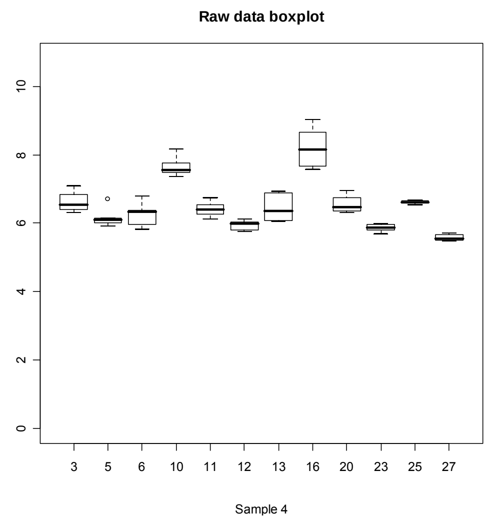

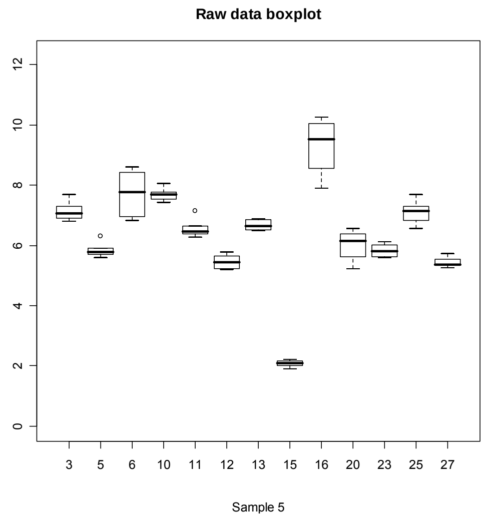

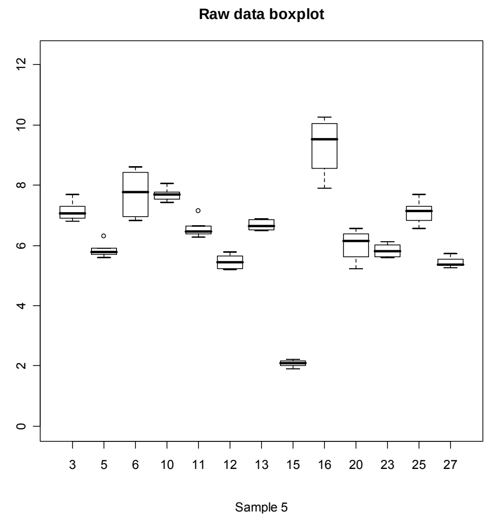

Results are expressed in % d.m.

Table 30: TS 15407 (CHN Flash Combustion: Hydrogen) - General Averages. Results are expressed in % d.m.

Level	m_j	*Number of valid results*
1	6.83	63
2	6.14	66
3	2.28	60
4	6.53	70
5	6.73	72

Table 31: TS 15407 (CHN Flash Combustion: Hydrogen) - Sums of squares and degrees of freedom, for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	43.28	10	1.46	12	3.69	40	369	189	33
2	32.19	10	0.08	13	0.20	42	396	198	33
3	1.01	9	0.02	14	0.08	42	396	198	33
4	36.05	11	3.00	12	2.62	46	412	208	35.5
5	85.30	11	2.64	12	8.99	48	432	216	36

Table 32: TS 15407 (CHN Flash Combustion: Hydrogen) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in % d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	r	s_{Rj}	R
1	0.86	0.11	0.3	0.84	0.91	2.548
2	0.73	0.02	0.07	0.196	0.73	2.044
3	NA	NA	0.04	0.112	0.04	0.112
4	0.72	0.26	0.24	0.672	0.76	2.128
5	1.12	0.1	0.43	1.204	1.2	3.36

3.5.3 TS 15407 (CHN Flash Combustion: Nitrogen)

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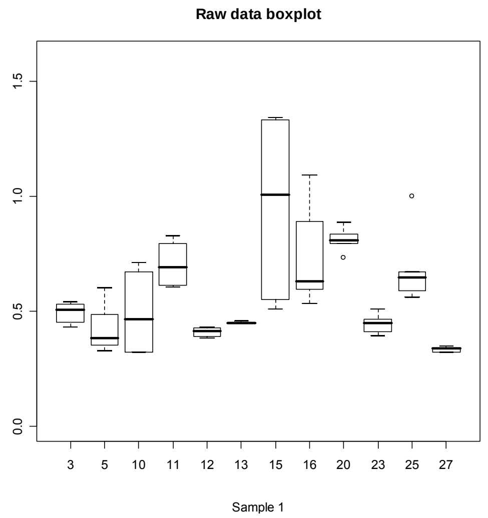

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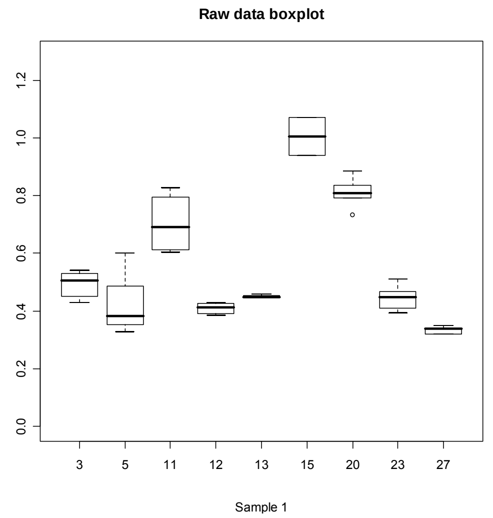

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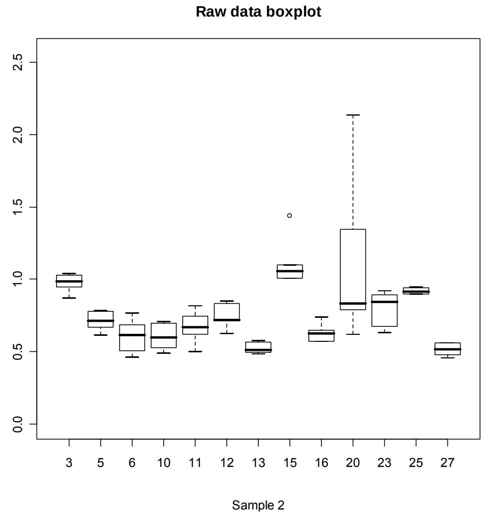

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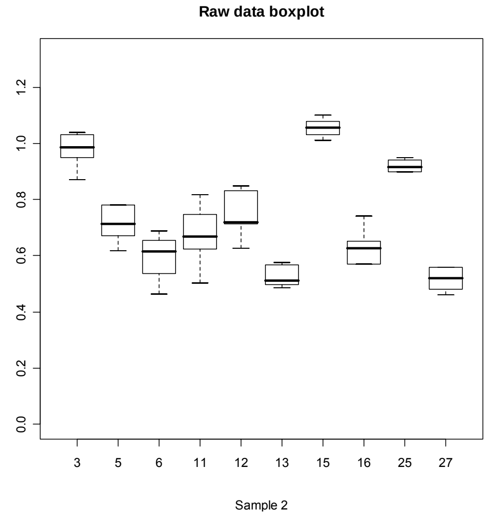

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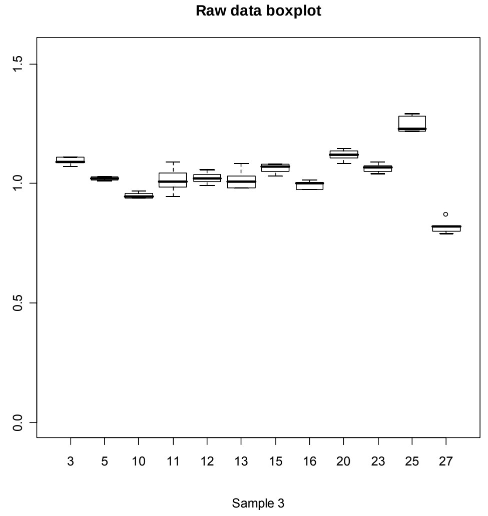

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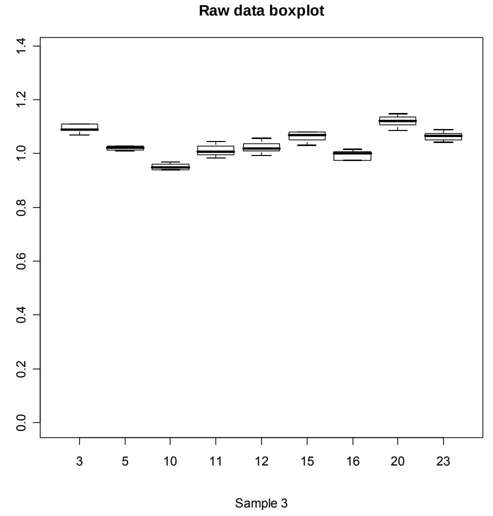

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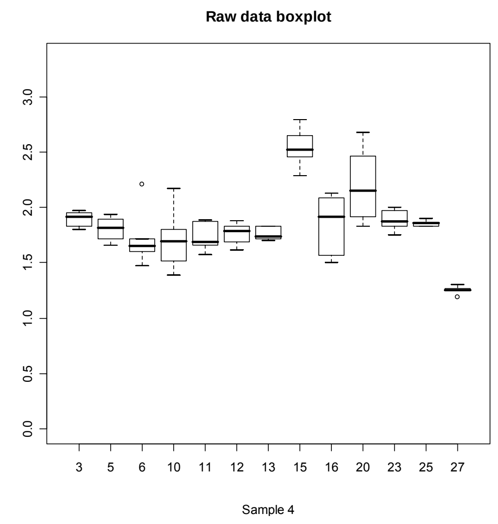

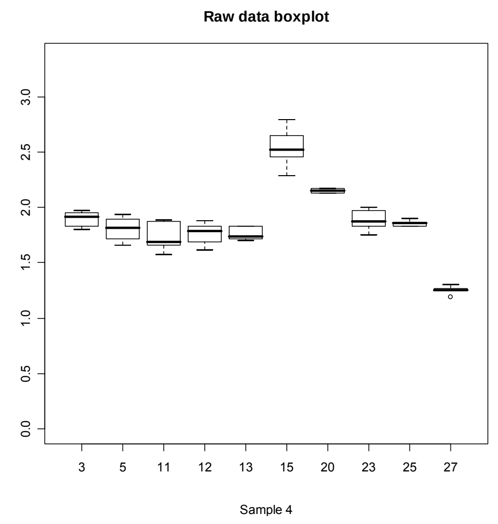

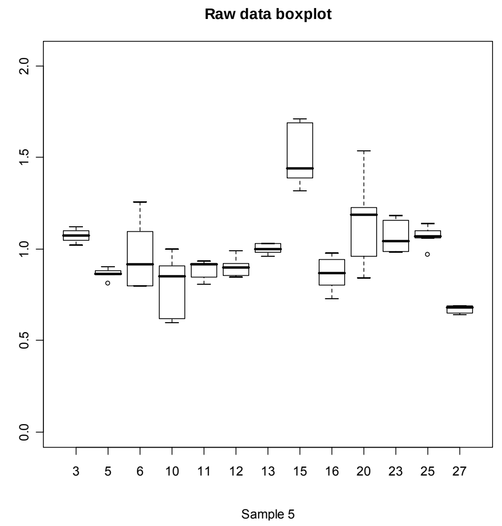

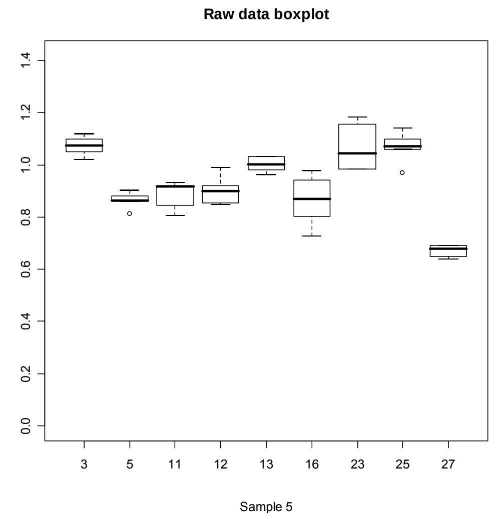

Results are expressed in % d.m.

Table 33: TS 15407 (CHN Flash Combustion: Nitrogen) - General Averages. Results are expressed in % d.m.

Level	m_j	*Number of valid results*
1	0.55	53
2	0.71	64
3	1.02	58
4	1.83	62
5	0.95	58

Table 34: TS 15407 (CHN Flash Combustion: Nitrogen) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	1.68	9	0.10	11	0.17	32	301	155	28
2	1.78	10	0.12	11	0.16	42	376	190	32.5
3	0.39	9	0.00	12	0.02	42	376	190	32.5
4	5.48	10	0.16	11	0.60	40	364	182	31
5	0.96	9	0.08	12	0.16	42	376	190	32.5

Table 35: TS 15407 – (CHN Flash Combustion: Nitrogen) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in % d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	r	s_{Rj}	R
1	0.18	0.04	0.07	0.196	0.2	0.56
2	0.17	0.05	0.06	0.168	0.18	0.504
3	NA	NA	0.02	0.056	0.02	0.056
4	NA	NA	0.12	0.336	0.12	0.336
5	0.12	0.03	0.06	0.168	0.14	0.392

3.5.4 TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Sulfur)

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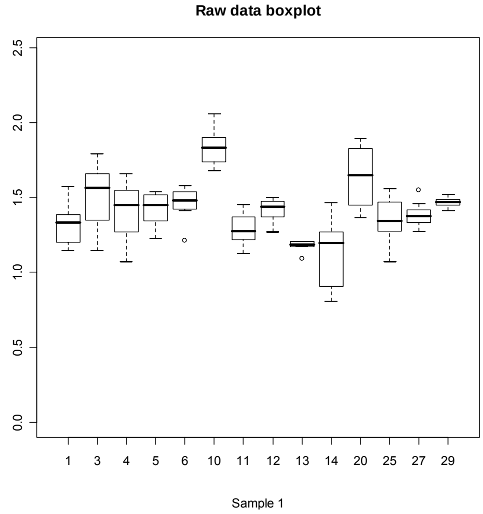

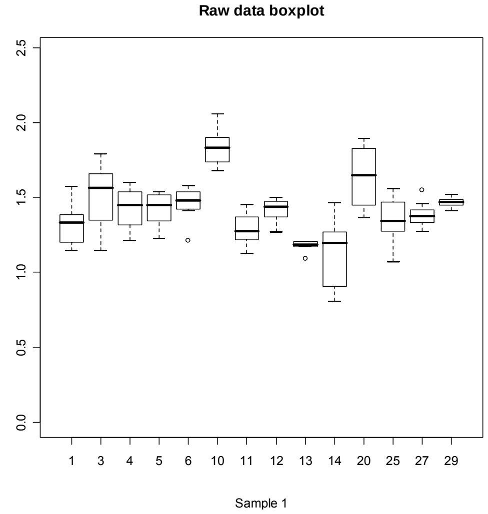

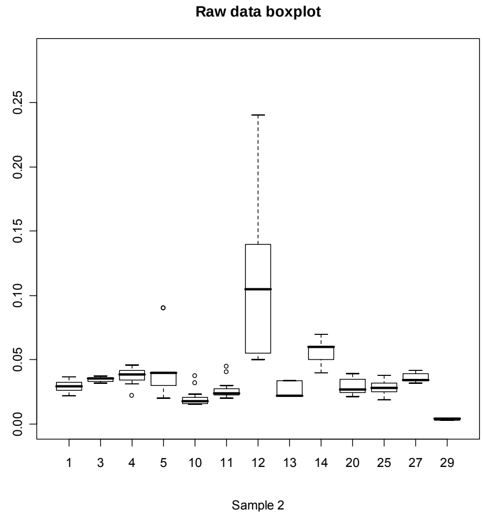

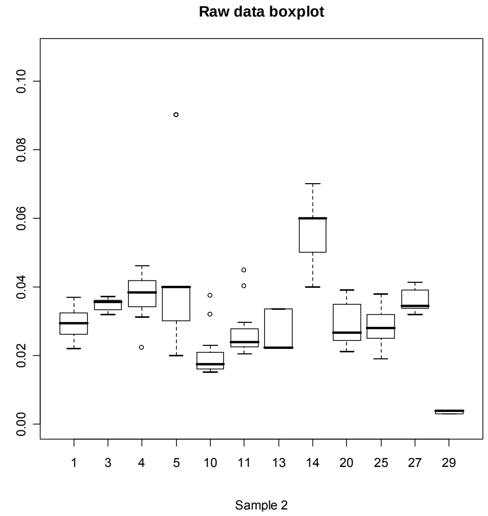

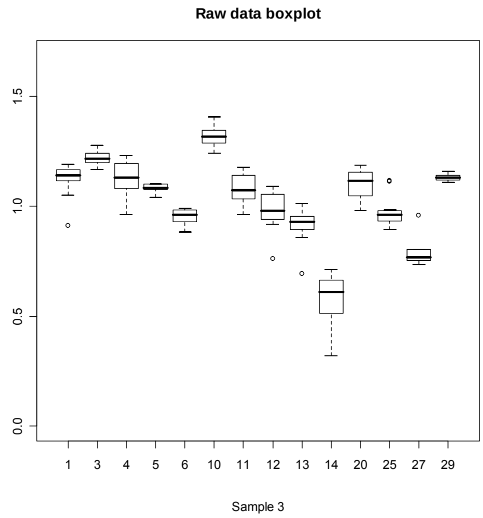

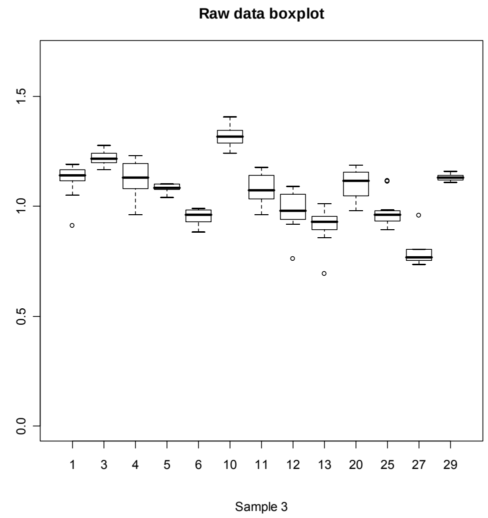

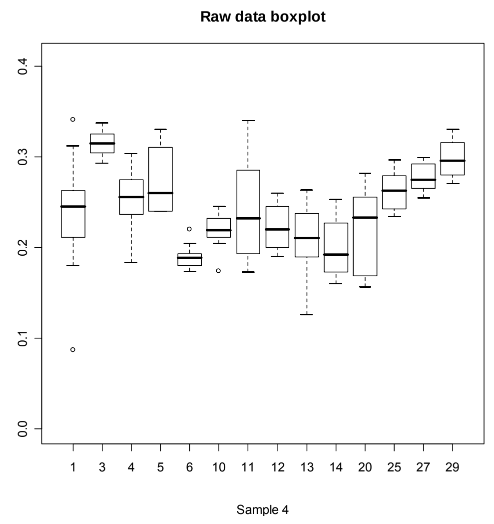

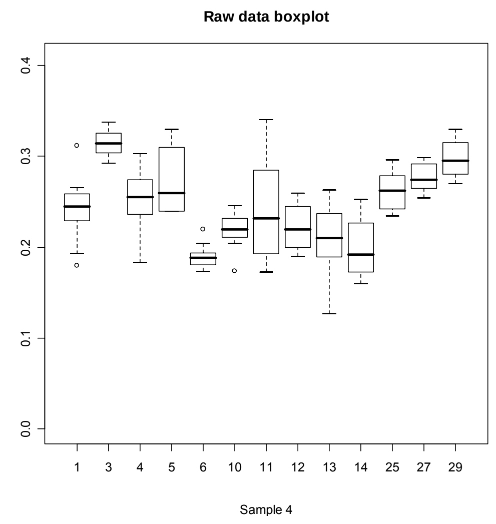

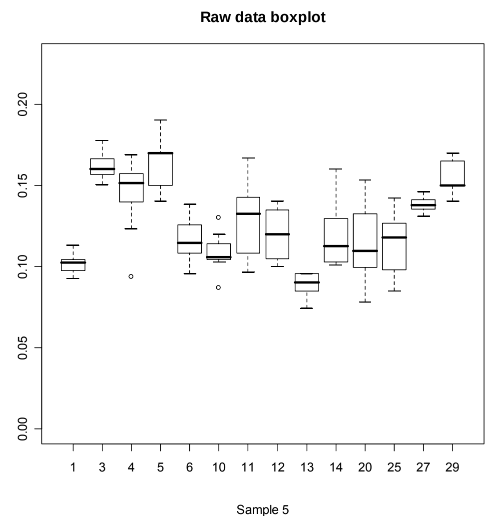

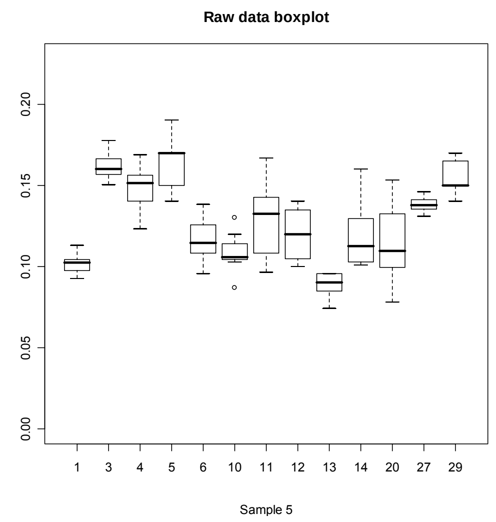

Results are expressed in % d.m.

Table 36: TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Sulfur) - General Averages. Results are expressed in % d.m.

Level	m_j	*Number of valid results*
1	1.42	160
2	0.03	144
3	1.06	156
4	0.25	154
5	0.13	154

Table 37: TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Sulfur) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	4.51	13	1.25	13	1.41	133	1864	952	83.2
2	0.02	11	0.00	16	0.01	140	2016	1008	84
3	2.77	12	0.07	15	0.48	140	2016	1008	84
4	0.21	12	0.01	15	0.12	138	1972	988	83.2
5	0.08	12	0.00	15	0.03	138	1972	988	83.2

Table 38: TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Sulfur) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in % d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	r	s_{Rj}	R
1	0.148	0.12	0.103	0.2884	0.181	0.5068
2	0.011	0.004	0.007	0.0196	0.013	0.0364
3	0.132	0.016	0.059	0.1652	0.144	0.4032
4	NA	NA	0.029	0.0812	0.029	0.0812
5	0.022	0.002	0.013	0.0364	0.026	0.0728

3.5.5 TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Chlorine)

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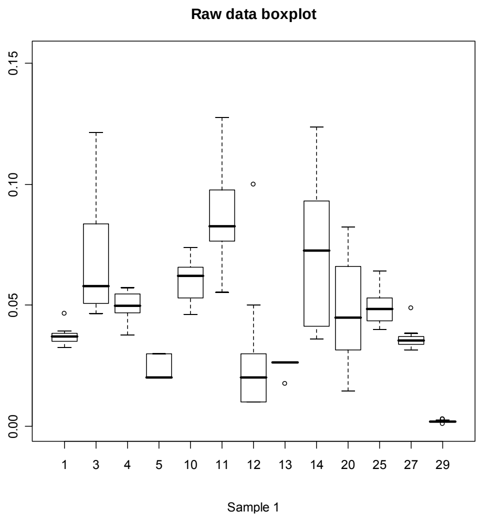	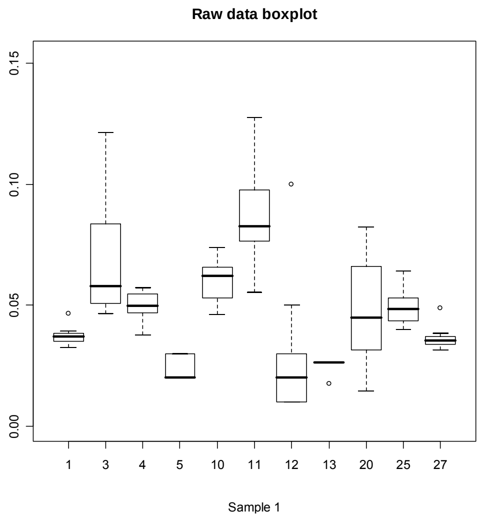
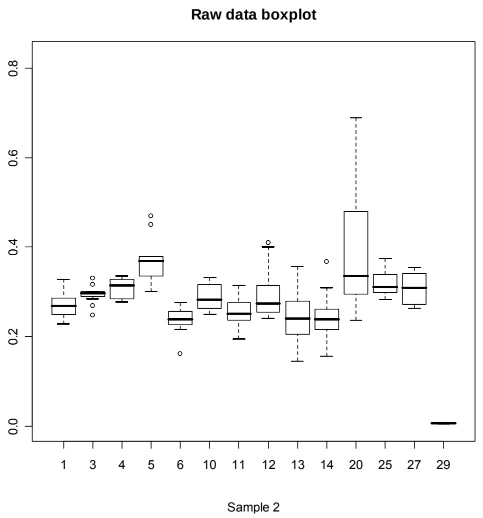	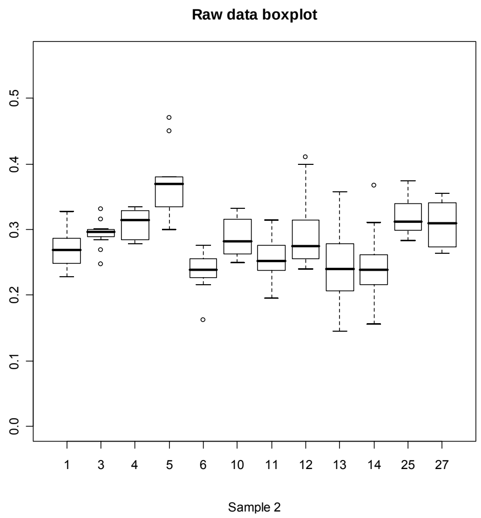
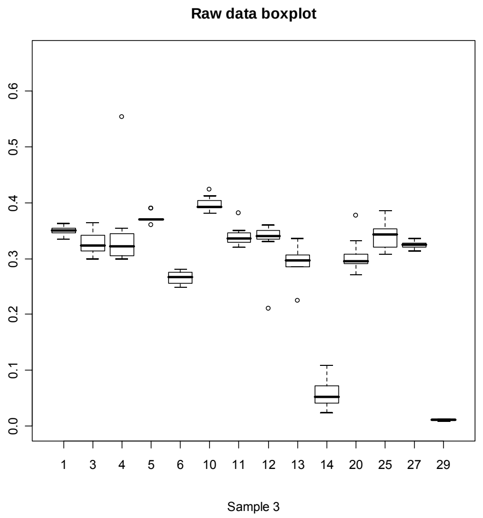	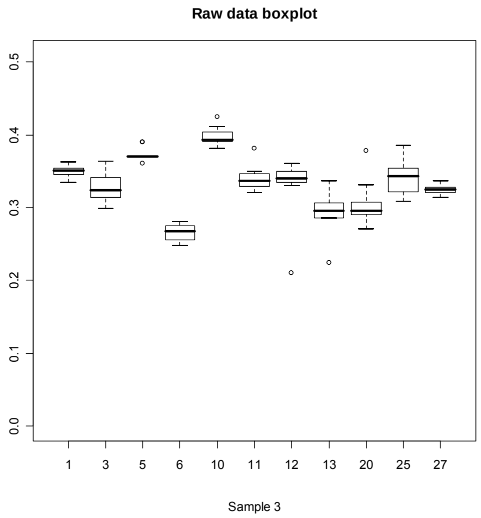

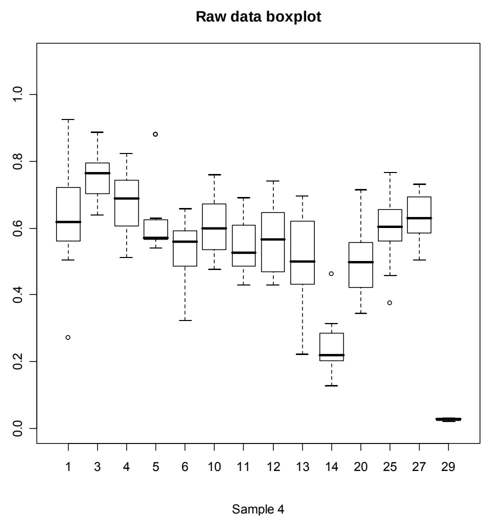

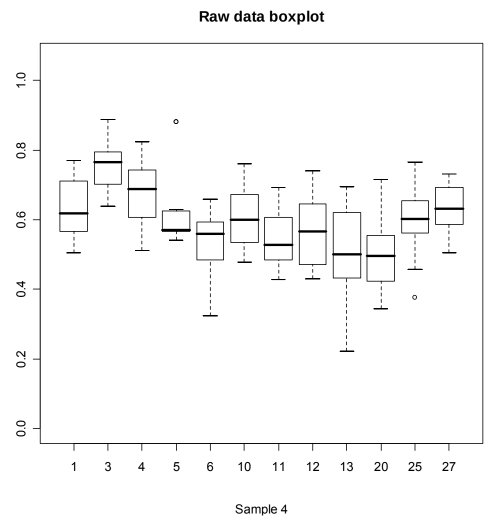

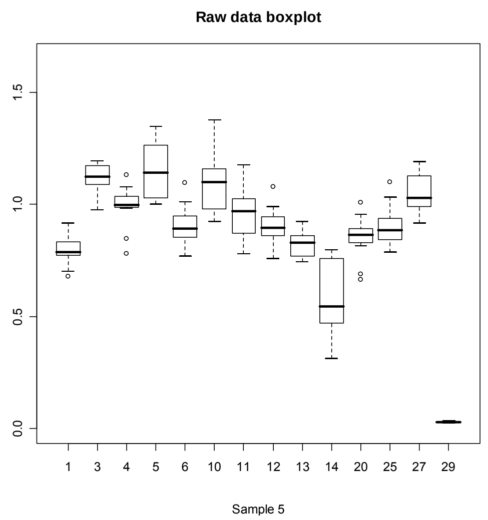

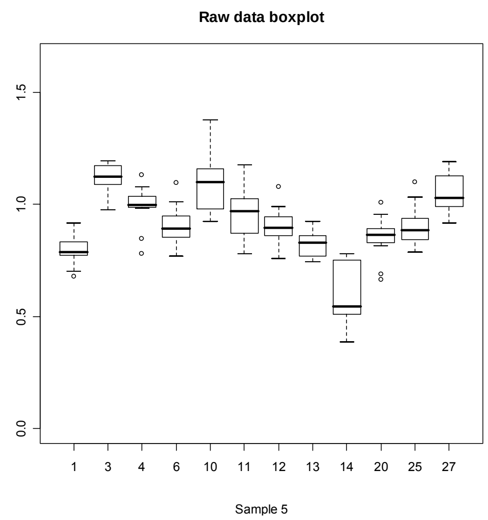

Results are expressed in % d.m.

Table 39: TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Chlorine) - General Averages. Results are expressed in % d.m.

Level	m_j	*Number of valid results*
1	0.049955	138
2	0.294215	156
3	0.31127	156
4	0.570634	156
5	0.931919	156

Table 40: TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Chlorine) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS_{.Hj}$, ni_{Hj}) and for repeatability ($SS_{.rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	0.051197	11	0.009629	13	0.027162	113	1620	828	72
2	0.328162	12	0.173844	13	0.254496	130	1872	936	78
3	0.991389	12	0.015776	13	0.096359	130	1872	936	78
4	2.081723	12	0.244242	13	1.351699	130	1872	936	78
5	3.46214	12	0.188359	13	1.285999	130	1872	936	78

Table 41: TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Chlorine) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in % d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	r	s_{Rj}	R
1	0.018	0.01	0.016	0.0448	0.024	0.0672
2	0.034	0.044	0.044	0.1232	0.056	0.1568
3	0.082	0.009	0.027	0.0756	0.087	0.2436
4	0.114	0.037	0.102	0.2856	0.153	0.4284
5	0.151	0.028	0.099	0.2772	0.181	0.5068

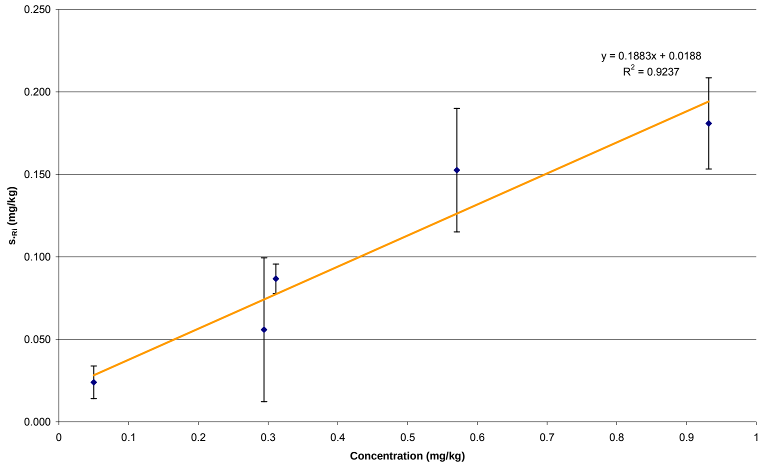

Figure 22: TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Chlorine) - Concentration dependence of reproducibility. Error bars represent the homogeneity effects-related standard deviation (s_{Hi})

3.5.6 TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Bromine)

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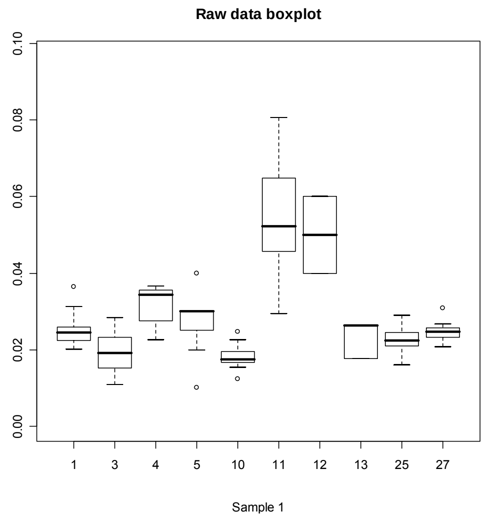	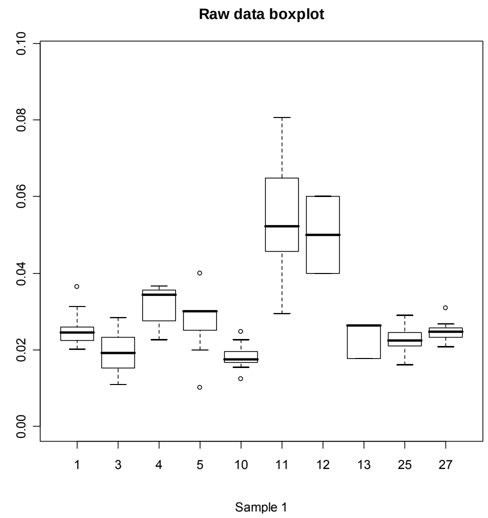
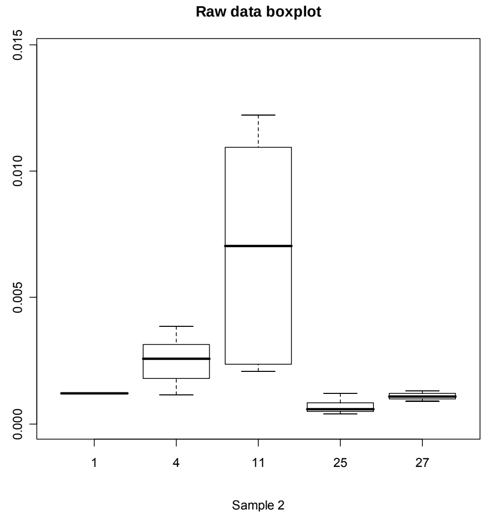	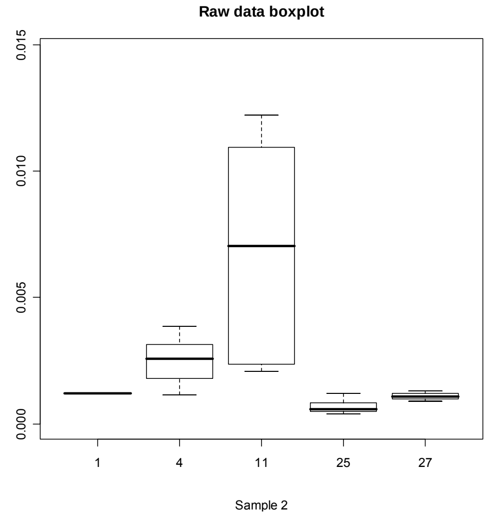
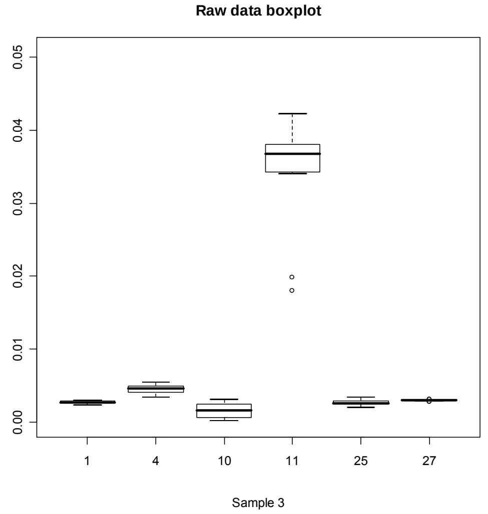	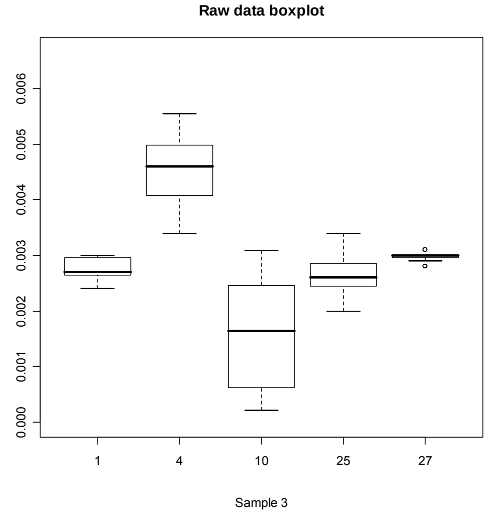

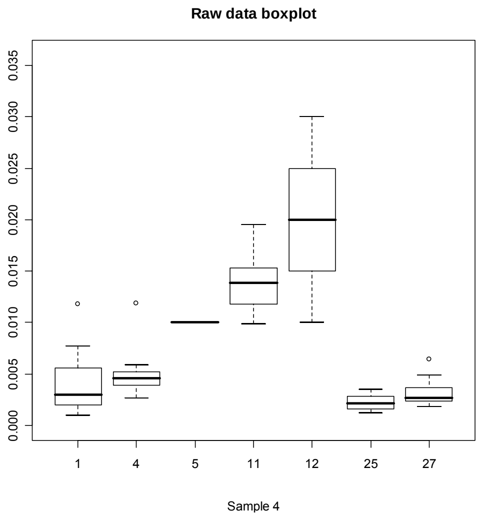

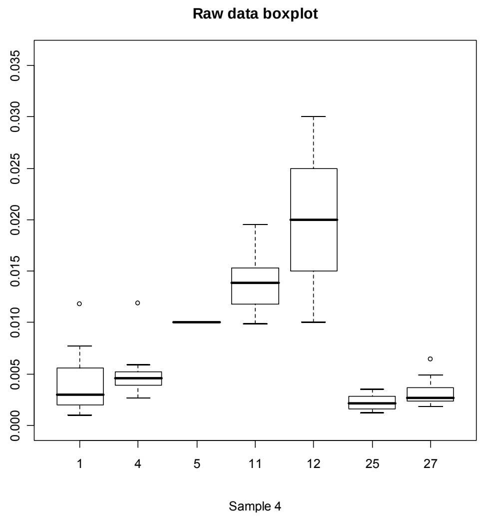

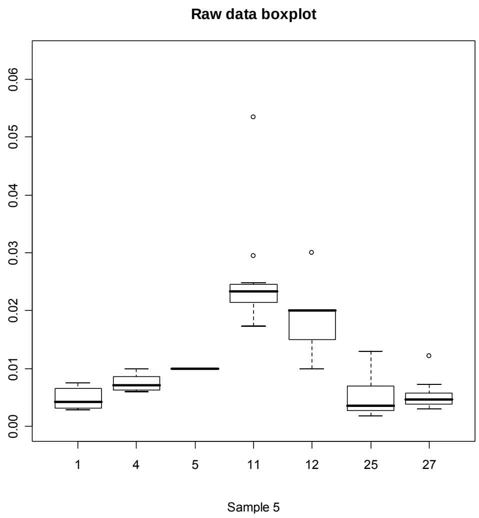

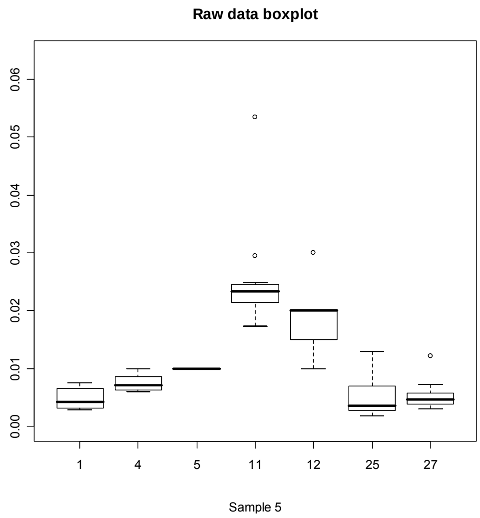

Results are expressed in % d.m.

Table 42: TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Bromine) - General Averages. Results are expressed in % d.m.

Level	m_j	*Number of valid results*
1	0.030	114
2	0.0027	49
3	0.0029	60
4	0.0065	65
5	0.0110	76

Table 43: TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Bromine) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS_{.Hj}$, ni_{Hj}) and for repeatability ($SS_{.rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	0.0157	9	0.00081	9	0.00433	95	1332	684	60
2	0.0003	4	0.00018	13	0.00003	85	1189	613	55
3	0.0001	4	0.00001	14	0.00001	95	1332	684	60
4	0.0017	6	0.00003	10	0.00044	78	1057	553	53
5	0.0046	6	0.00002	12	0.00153	87	1204	620	56

Table 44: TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Bromine) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in % d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	0.012	0.0027	0.0067	0.01876	0.0138	0.03864
2	0.001	0.0019	0.0006	0.00168	0.0012	0.00336
3	0.0007	0.0003	0.0003	0.00084	0.0008	0.00224
4	NA	NA	0.0024	0.00672	0.0024	0.00672
5	NA	NA	0.0042	0.01176	0.0042	0.01176

3.5.7 TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Fluorine)

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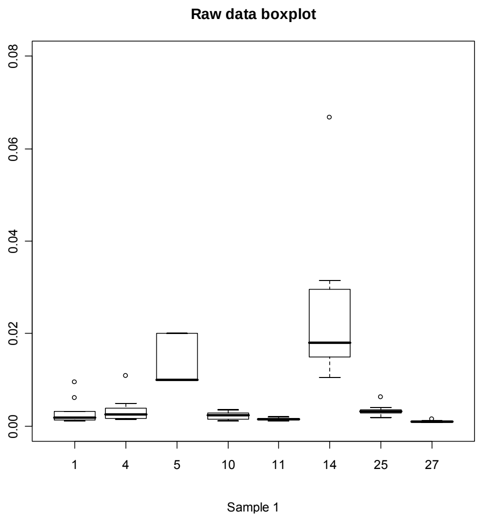

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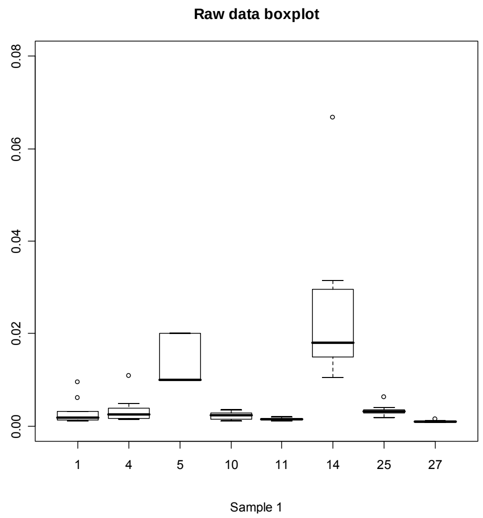

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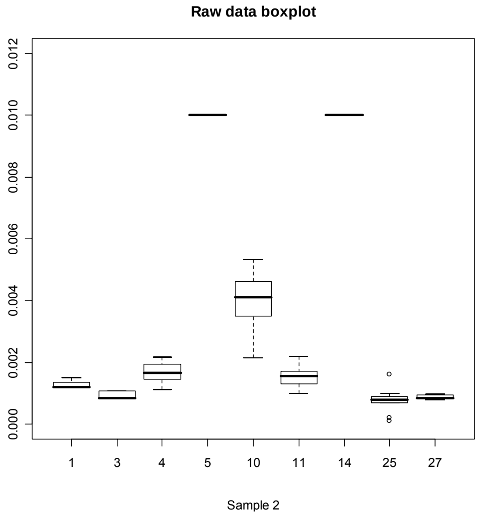

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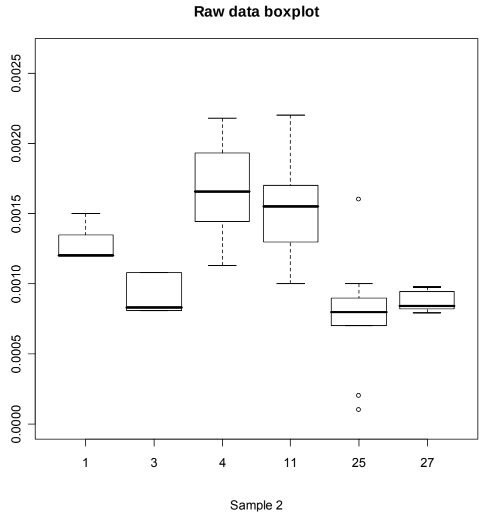

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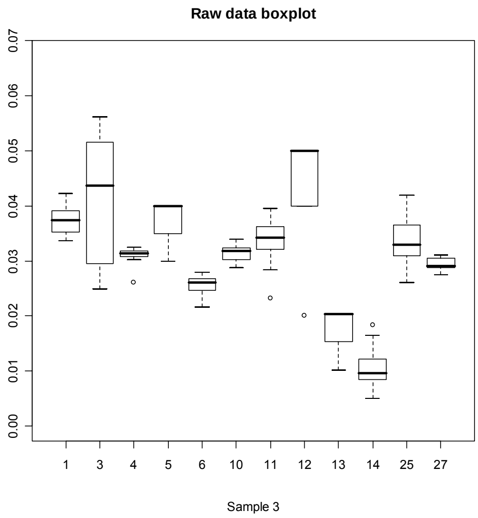

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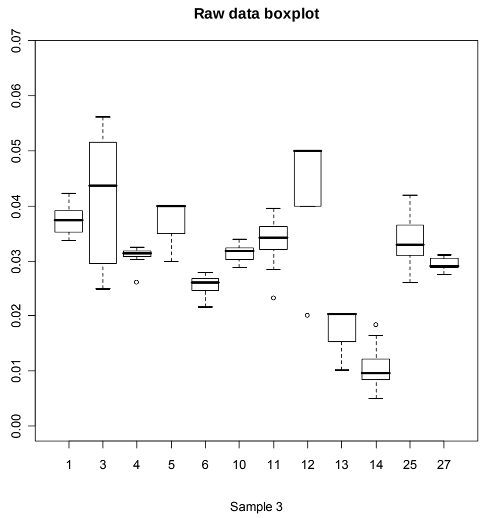

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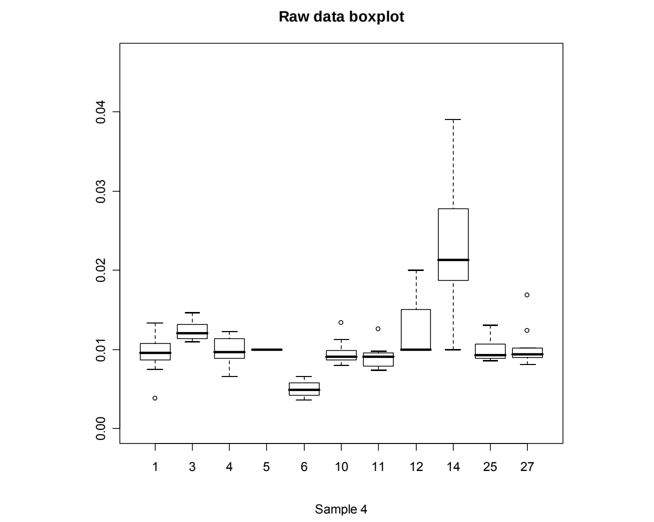

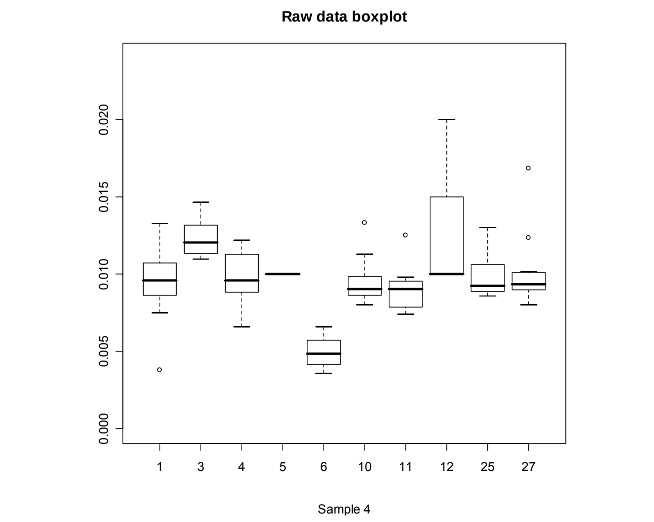

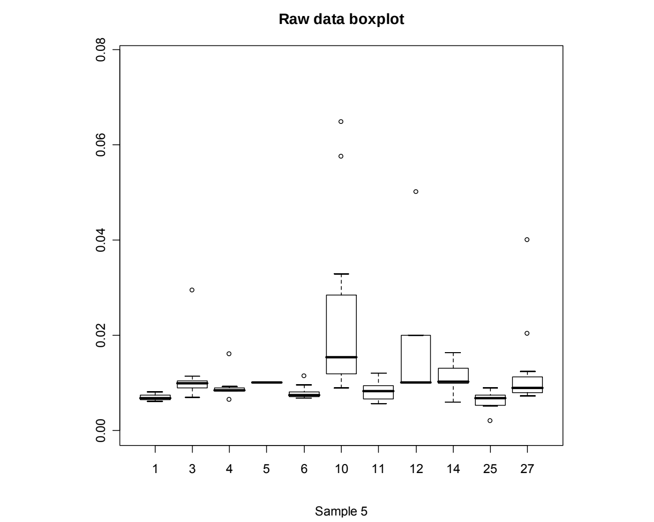

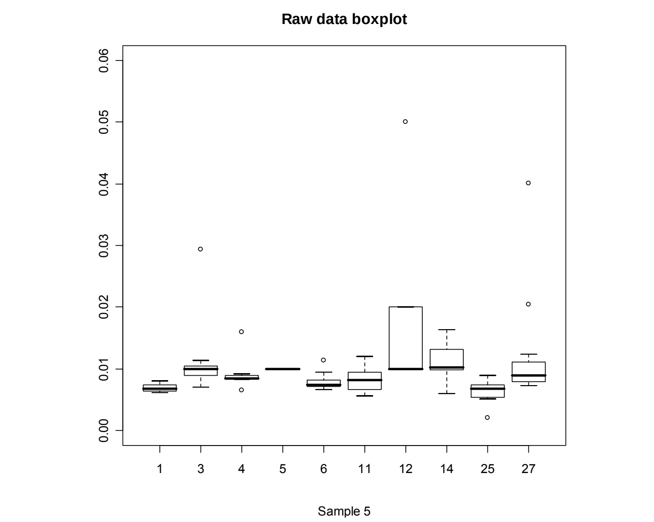

Results are expressed in % d.m.

Table 45: TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Fluorine) - General Averages. Results are expressed in % d.m.

Level	m_j	*Number of valid results*
1	0.0063	92
2	0.0027	86
3	0.0311	144
4	0.0098	118
5	0.0100	117

Table 46: TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Fluorine) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	0.00518	7	0.000561	16	0.002341	68	1064	534	46.2
2	0.000763	8	4.88E-07	14	1.35E-05	63	974	490	43.867
3	0.01201	11	0.000457	12	0.003071	120	1728	864	72
4	0.000458	9	5.12E-05	14	0.000427	118	1684	844	71.2
5	0.000866	9	0.000369	14	0.002723	117	1665	833	70.56

Table 47: TS 15408 (Cl, F, S, Br by oxygen combustion and IC: Fluorine) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in % d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	0.0078	0.0005	0.0059	0.01652	0.0098	0.02744
2	NA	NA	0.0005	0.0014	0.0005	0.0014
3	0.0094	0.0014	0.0051	0.01428	0.0106	0.02968
4	0.0018	0.0001	0.0019	0.00532	0.0026	0.00728
5	0.0022	0.0008	0.0048	0.01344	0.0053	0.01484

3.5.8 Digestion verification: Analysis of pre-digested SRF sample QV-F

Table 48: Analysis of pre-digested SRF sample QV-F - Average values per laboratory on QV-F analysis. Results are expressed in mg/L.

Laboratory	*Al*	*Ca*	*Fe*	*K*	*Mg*	*Na*	*P*	*Si*	*Ti*
1	163.8						32.0		
4			48.8						
12	179.2						36.1		
13							4.1		
14	173.0	417.2	50.1	41.3	33.1	60.6	18.4	20.0	0.66
16	164.8	419.5	45.5	37.1	33.1	51.0		151.5	0.61
19	172.9	436.7	46.2	31.1	36.8	55.8		29.9	0.64
20	173.4	495.4	53.6	47.4	37.2	58.8	18.0	82.7	0.58
21	175.5						36.7		
22	145.7						32.9	36.9	
27	172.0	450.4	47.5	47.3	34.9	66.2	14.9		0.63
29		226.5	48.7	43.4	21.9	66.2			

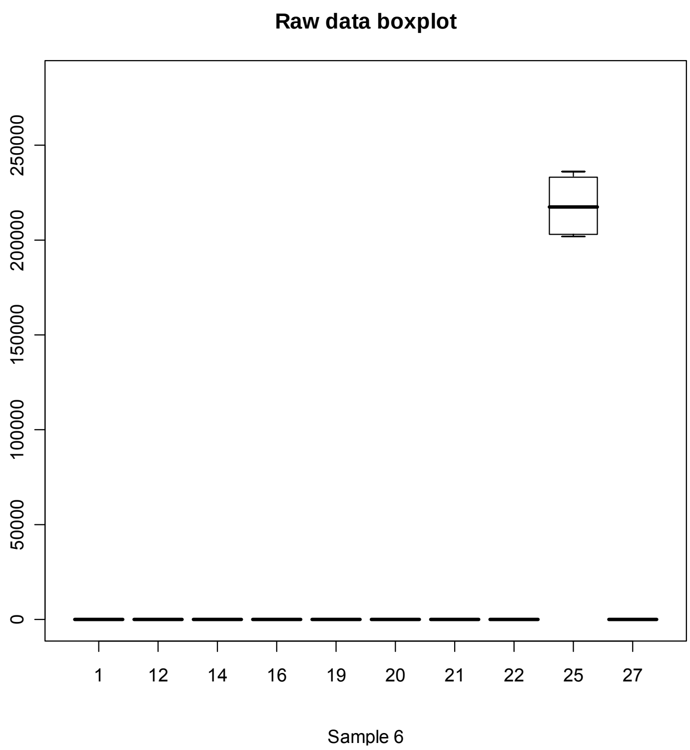

Figure 23: Box-and-Wiskers plot for Al in the acidic digested SRF. Laboratory 25 has been discarded as a clear outlier, the same situation occurring for all the other elements. Results are expressed in mg/L.

3.5.9 TS 15410 A - EN 13656 (aqua regia, HF, boric acid, micro-wave assisted digestion: Aluminium)

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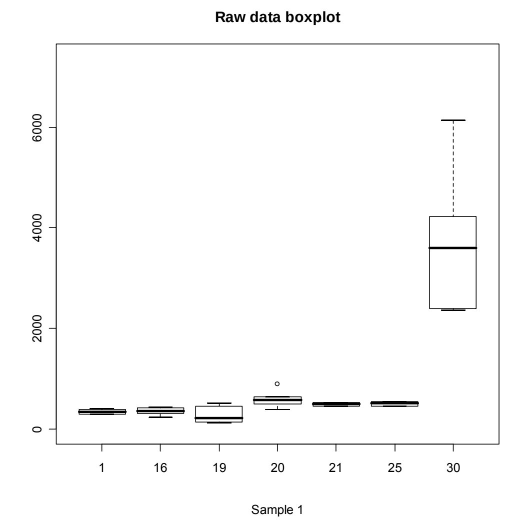

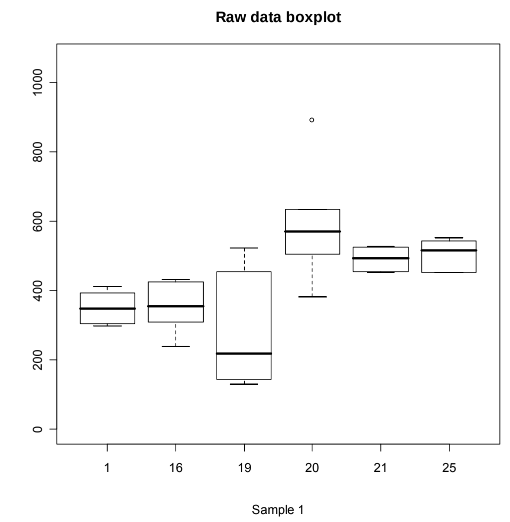

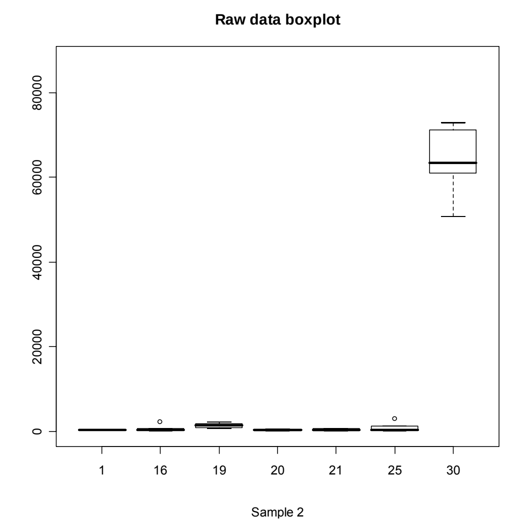

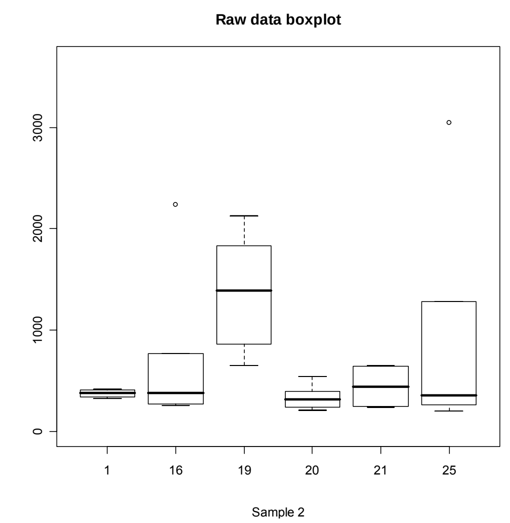

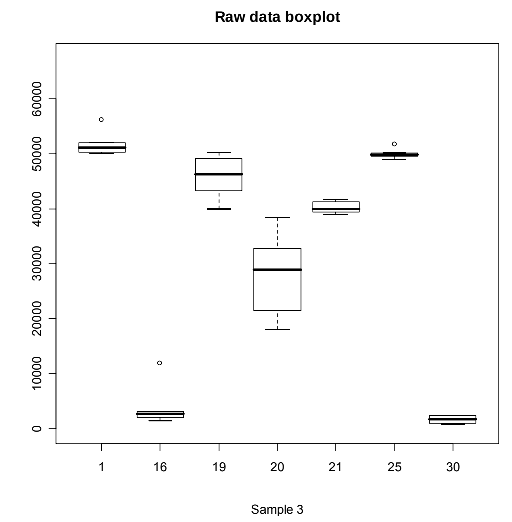

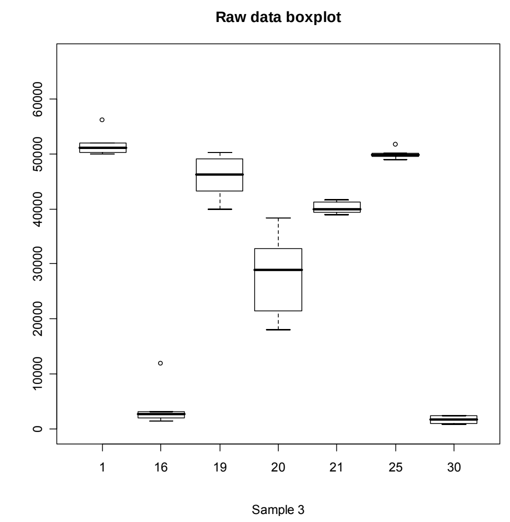

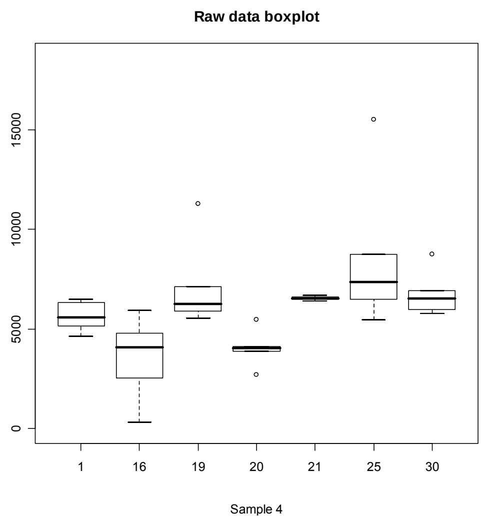	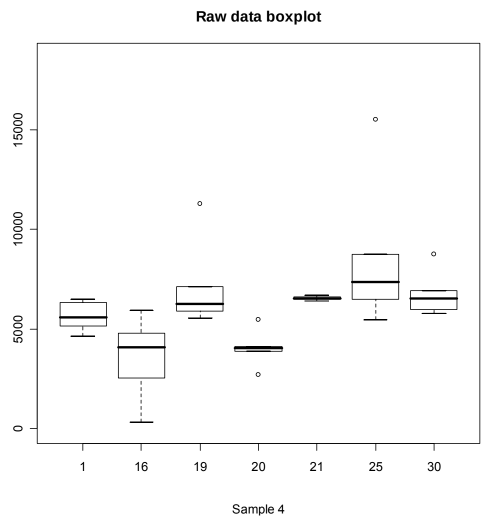
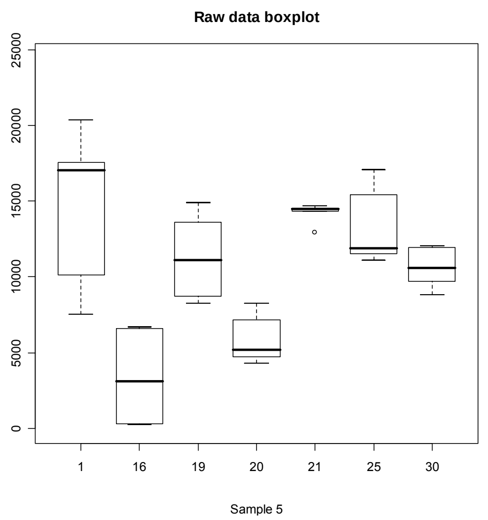	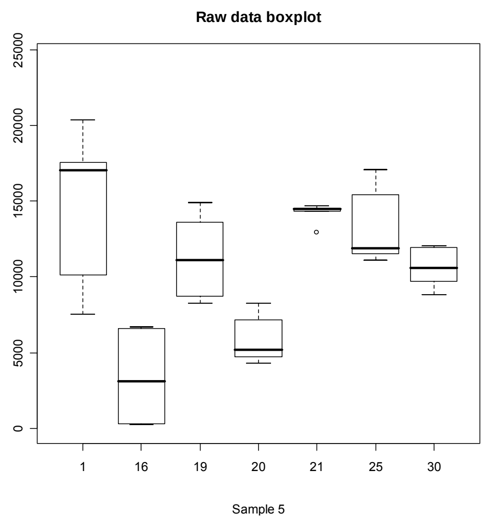
	Results are expressed in mg/kg d.m.

Table 49: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Aluminium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	428.2	36
2	693.4	36
3	31630.3	42
4	6014.5	42
5	10475.3	42

Table 50: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Aluminium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	423193.6	5	174036.5	8	169810.4	22	216	108	18
2	4841448	5	4372266	8	6699168	22	216	108	18
3	1.61E+10	6	2.49E+08	7	2.28E+08	28	252	126	21
4	1.07E+08	6	19404890	7	1.01E+08	28	252	126	21
5	6.88E+08	6	34478170	7	2.24E+08	28	252	126	21

Table 51: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Aluminium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	98	79	88	246.4	132	369.6
2	238	328	552	1545.6	601	1682.8
3	21026	3028	2851	7982.8	21219	59413.2
4	NA	NA	1898	5314.4	1898	5314.4
5	NA	NA	2826	7912.8	2826	7912.8

3.5.10 TS 15410 A - EN 13656 (aqua regia, HF, boric acid, micro-wave assisted digestion: Calcium)

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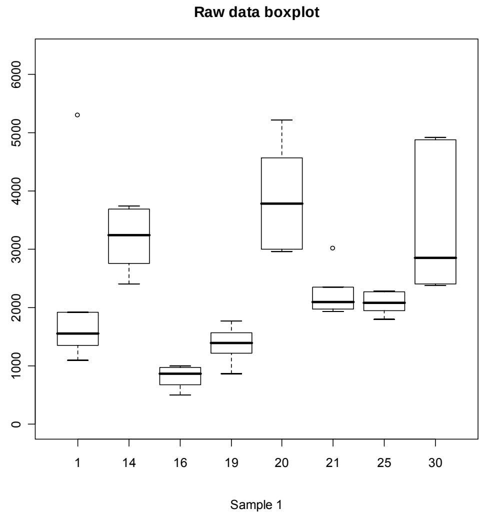	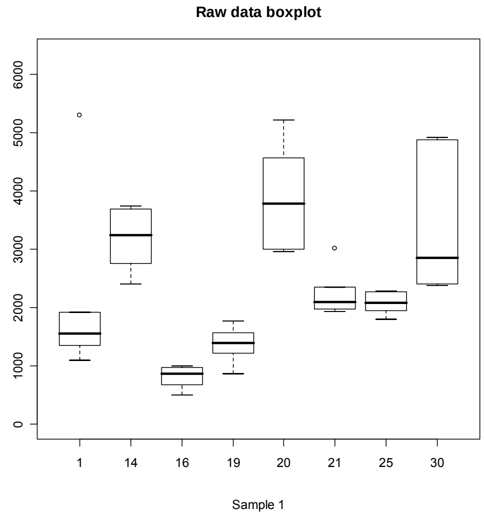
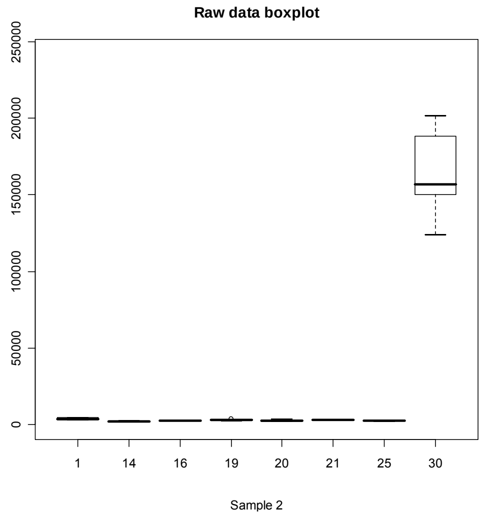	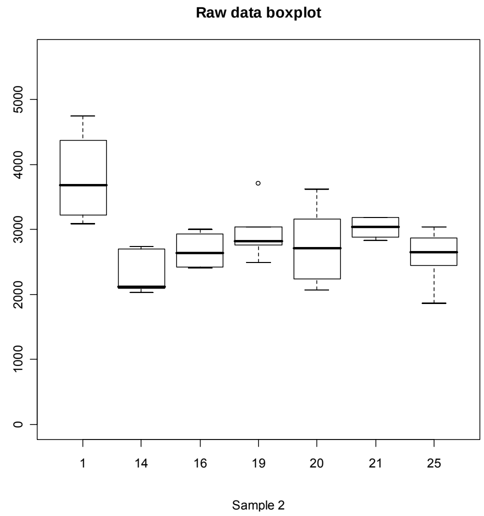
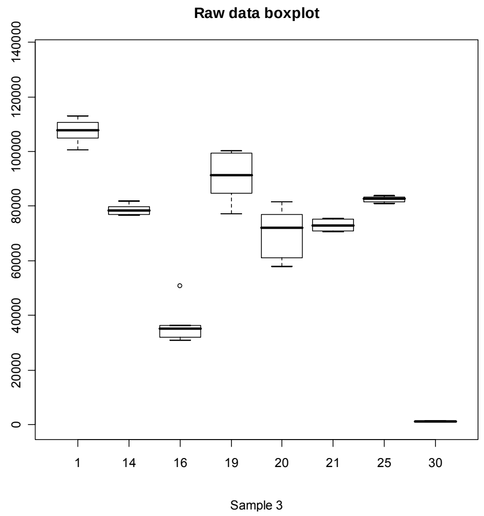	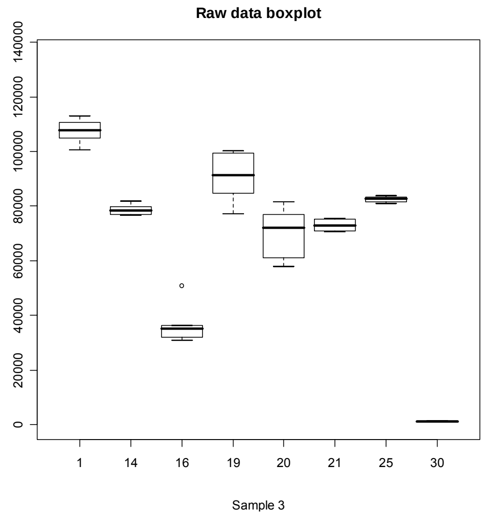

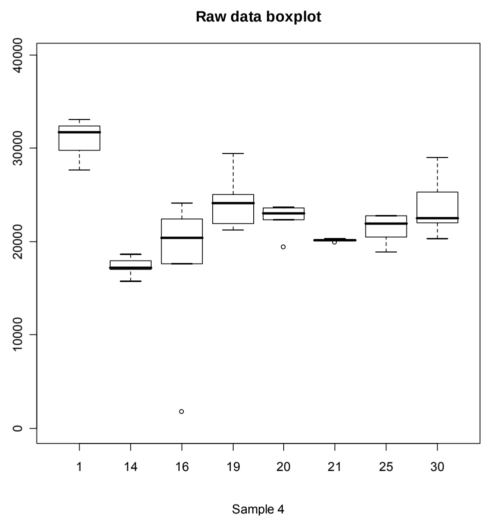

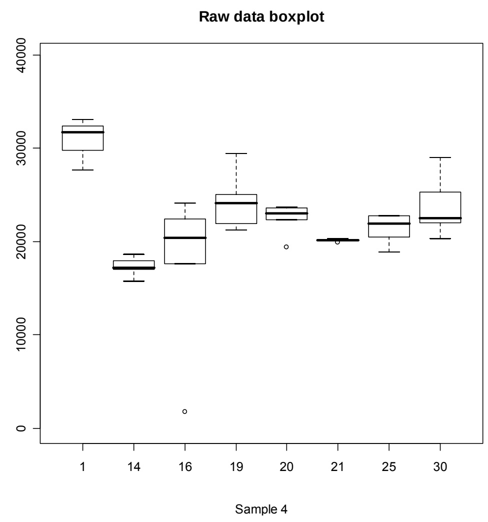

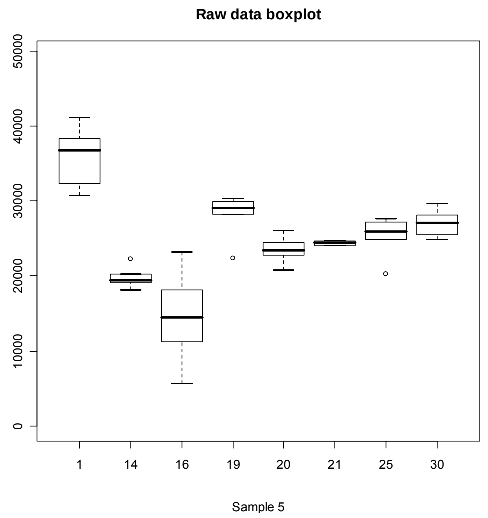

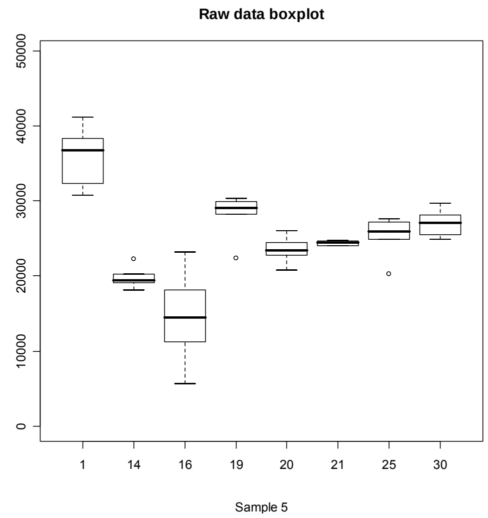

Results are expressed in mg/kg d.m.

Table 52: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Calcium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	2385	48
2	2870	42
3	67564	48
4	22272	48
5	24836	48

Table 53: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Calcium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	45452477	7	6445511	8	20818393	32	288	144	24
2	8055680	6	2155786	9	4693195	32	288	144	24
3	4.72E+10	7	5.74E+08	8	6.84E+08	32	288	144	24
4	7.97E+08	7	97672575	8	3.76E+08	32	288	144	24
5	1.64E+09	7	60903760	8	3.17E+08	32	288	144	24

Table 54: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Calcium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	973.612	227.3866	806.5822	2258.43016	1264.316	3540.0848
2	391.7105	186.6173	382.9652	1072.30256	547.8134	1533.87752
3	33337.29	4100.14	4622.112	12941.9136	33656.19	94237.332
4	4115.79	393.4193	3427.059	9595.7652	5355.788	14996.2064
5	NA	NA	3145.137	8806.3836	3145.137	8806.3836

3.5.11 TS 15410 A - EN 13656 (aqua regia, HF, boric acid, micro-wave assisted digestion: Iron)

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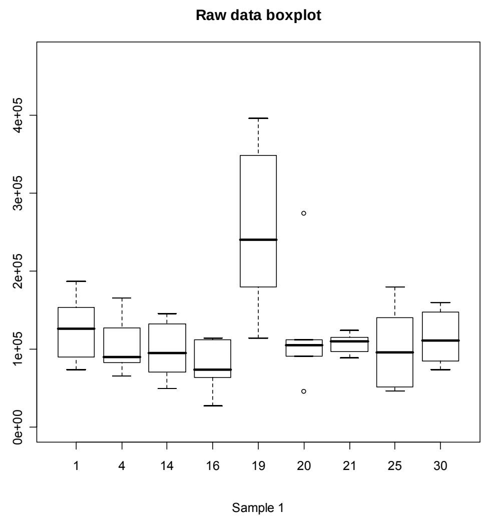

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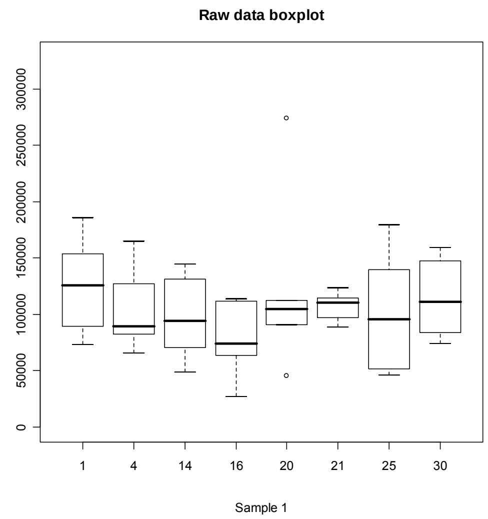

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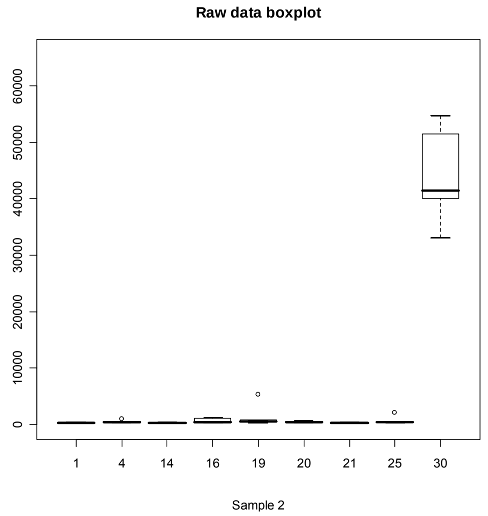

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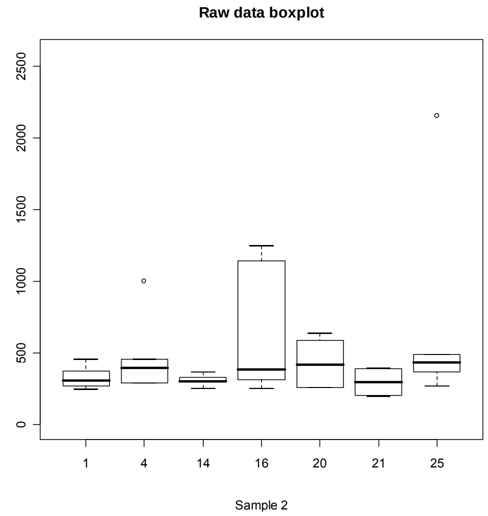

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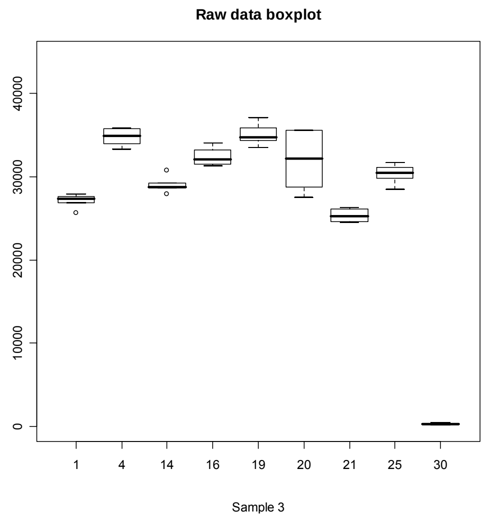

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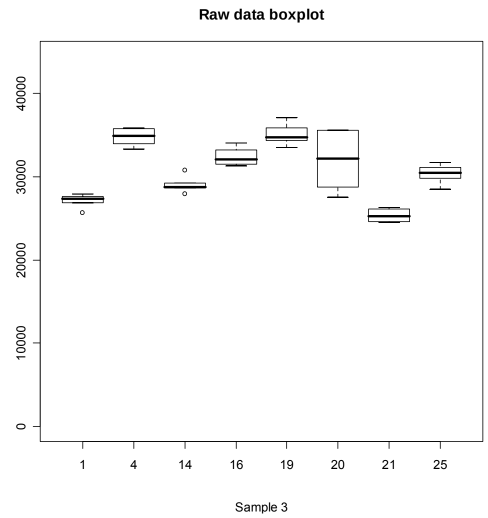

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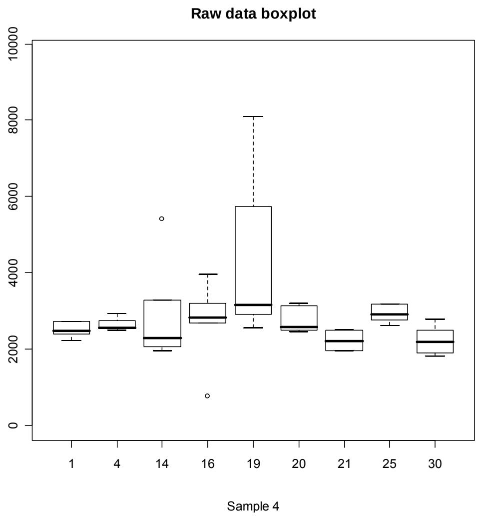
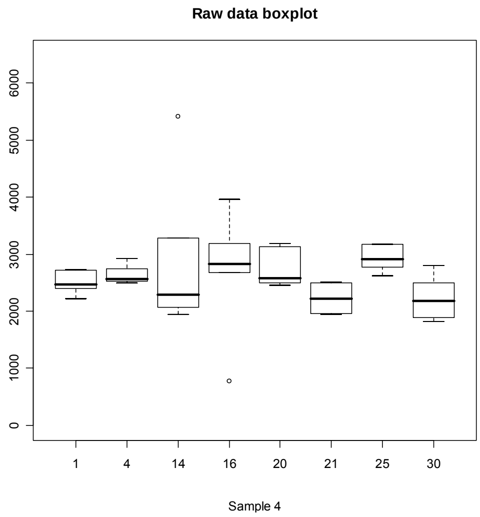
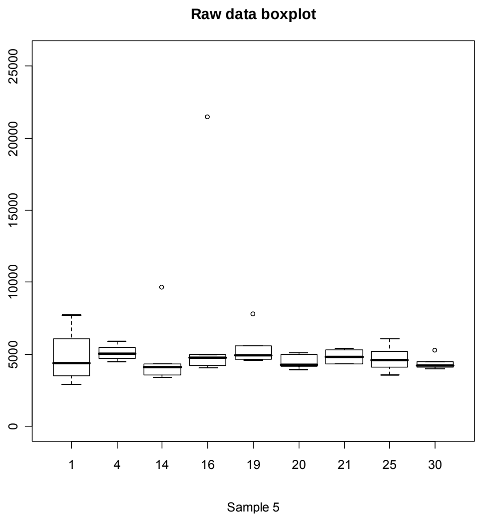
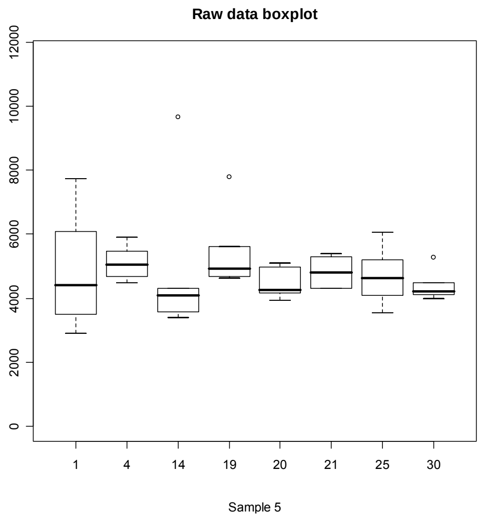
	
	
	Results are expressed in mg/kg d.m.

Table 55: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Iron) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	106166	48
2	558	48
3	30761	48
4	2606	48
5	4818	48

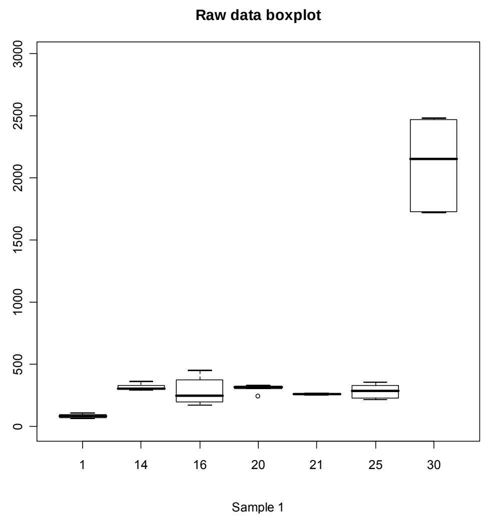
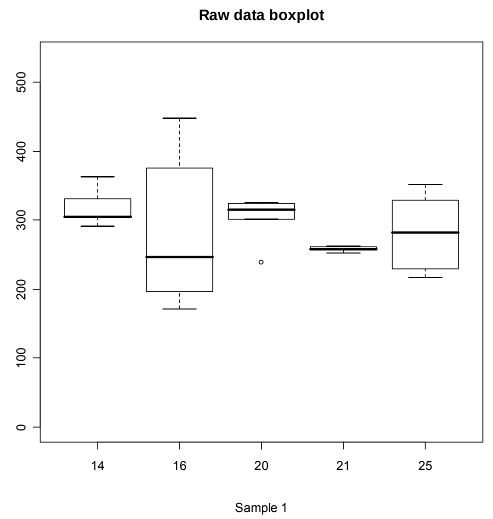
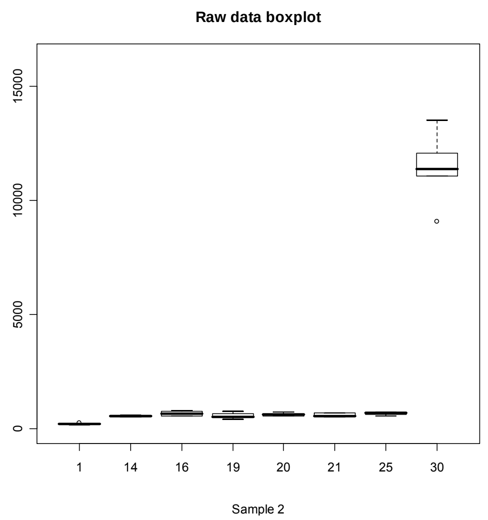
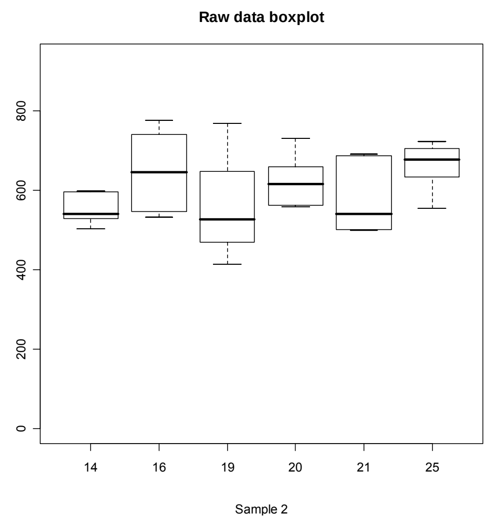
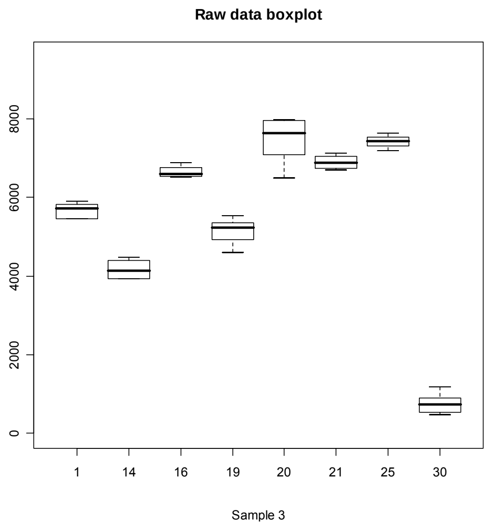
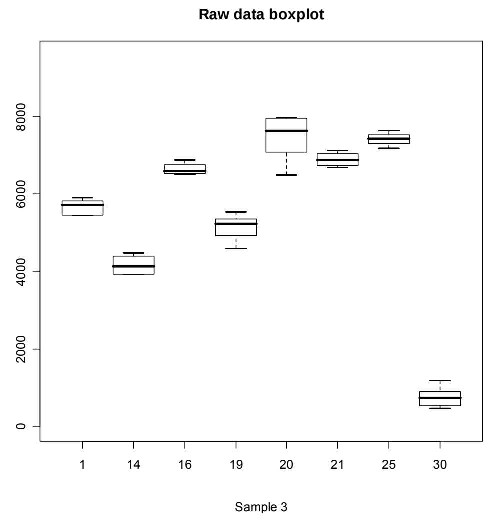
Table 56: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Iron) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	9.84E+09	7	7.62E+09	8	7.4E+10	32	288	144	24
2	4882021	7	5419162	8	17483056	32	288	144	24
3	5.07E+08	7	15617730	8	77213147	32	288	144	24
4	3042296	7	3773619	8	12872600	32	288	144	24
5	4697614	7	14053039	8	47213998	32	288	144	24

Table 57: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Iron) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	NA	NA	48077	134615.6	48077	134615.6
2	57.8	209	739	2069.2	741	2074.8
3	NA	NA	1553	4348.4	1553	4348.4
4	0	152.1333	634	1775.2	634	1775.2
5	0	306.1549	1215	3402	1215	3402

3.5.12 TS 15410 A - EN 13656 (aqua regia, HF, boric acid, micro-wave assisted digestion: Potassium)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	

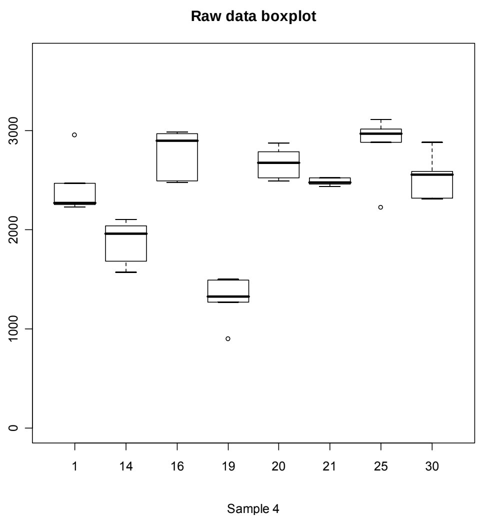
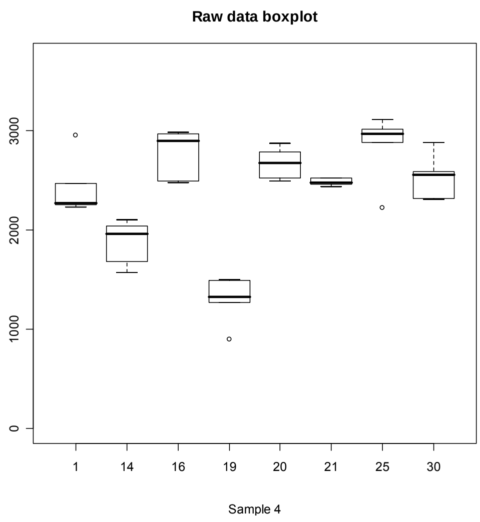
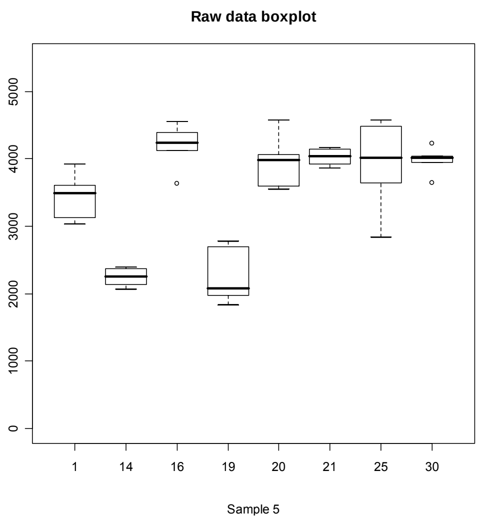
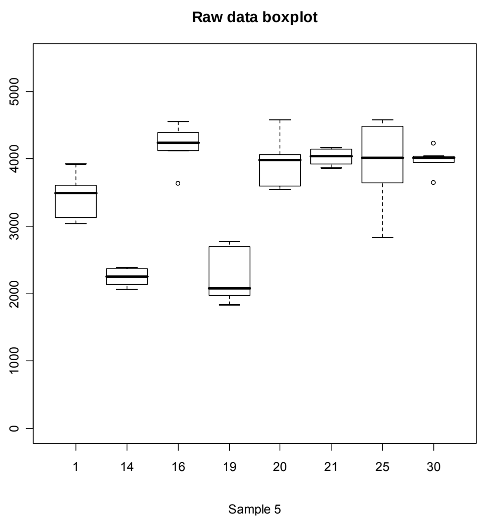
	
	
	Results are expressed in mg/kg d.m.

Table 58: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Potassium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	520	42
2	1994	45
3	5527	48
4	2367	48
5	3502	48

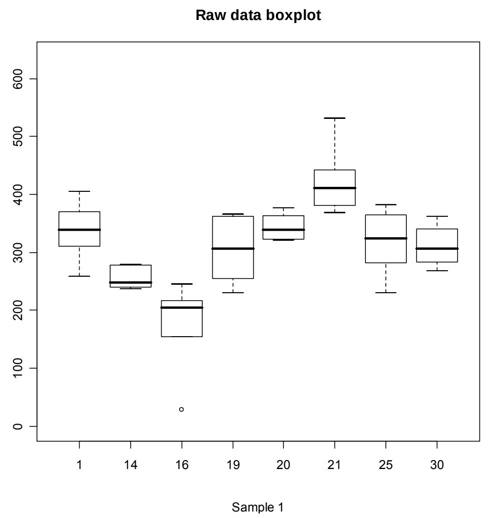
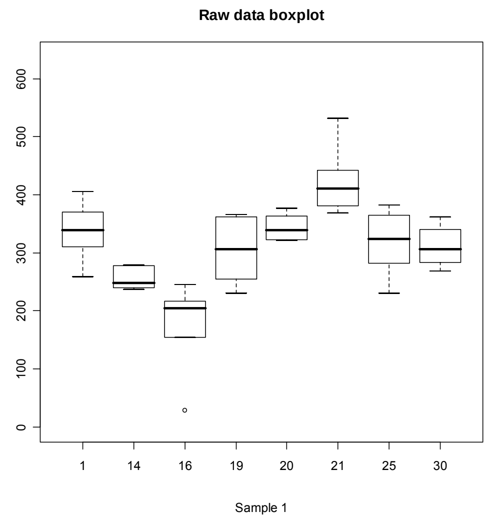
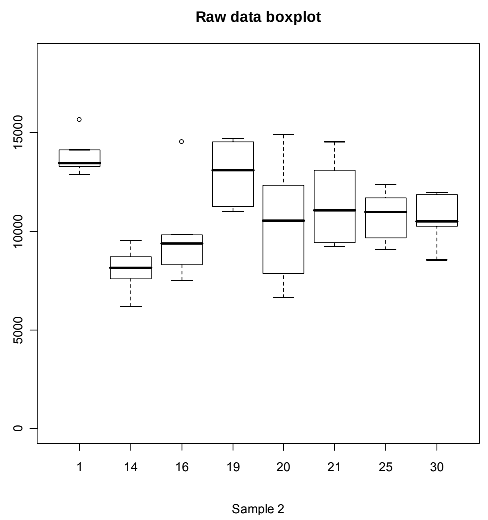
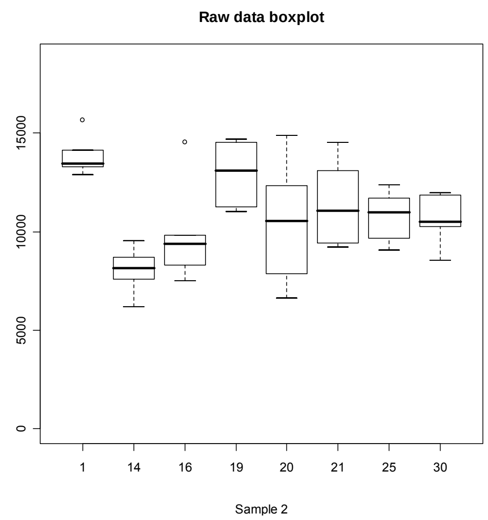
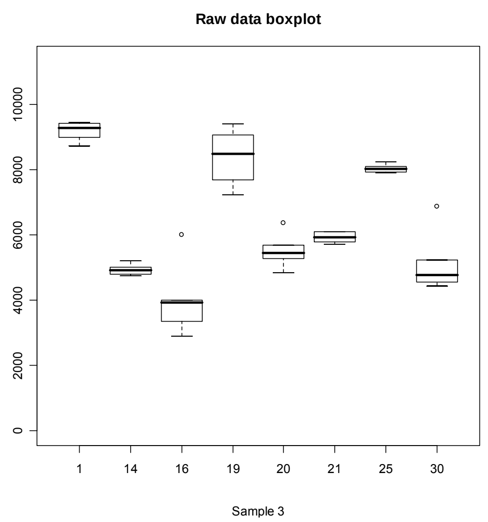
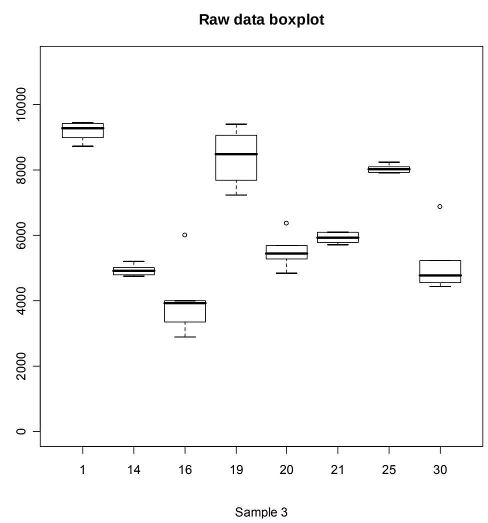
Table 59: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Potassium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

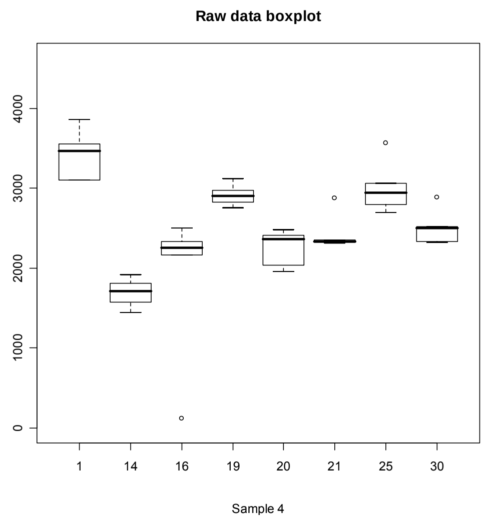
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	18115057	6	131627.4	9	706299.4	26	252	126	21
2	6.15E+08	7	345520.3	8	10354646	29	261	131	22.67
3	2.12E+08	7	764412.3	8	2748212	32	288	144	24
4	11544038	7	229397.2	8	1793305	32	288	144	24
5	27375338	7	669275.2	8	4273014	32	288	144	24

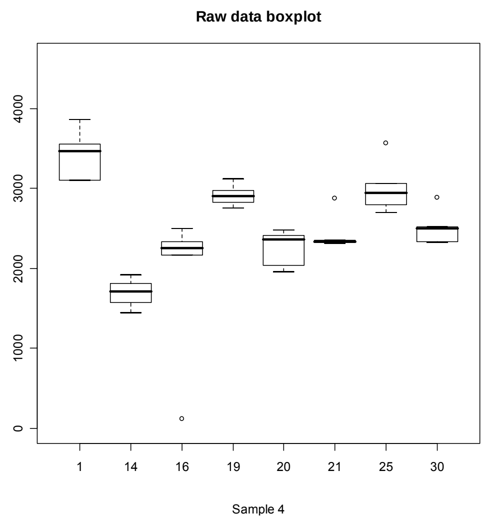
Table 60: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Potassium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

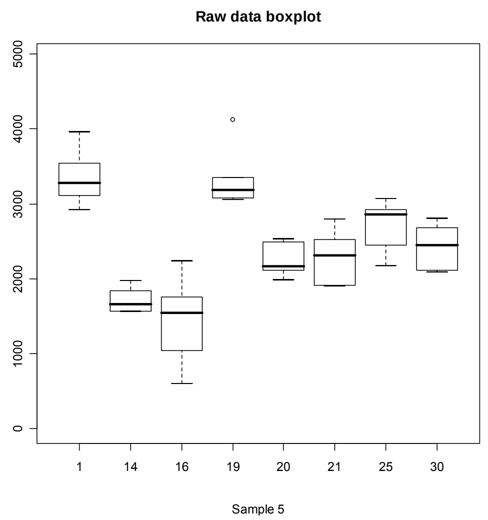
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	NA	NA	165	462	165	462
2	NA	NA	598	1674.4	598	1674.4
3	2241	57	293	820.4	2260	6328
4	NA	NA	237	663.6	237	663.6
5	NA	NA	365	1022	365	1022

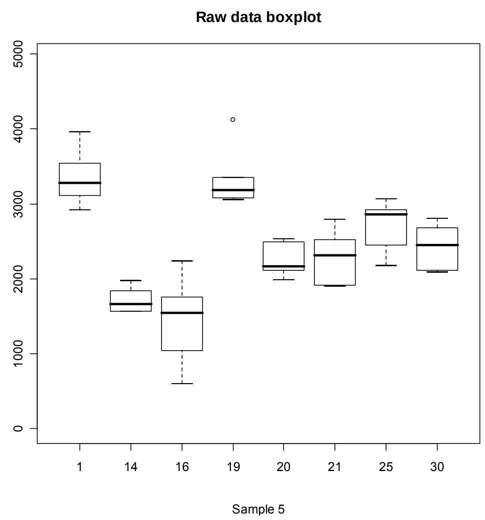
3.5.13 TS 15410 A - EN 13656 (aqua regia, HF, boric acid, micro-wave assisted digestion: Magnesium)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	









Results are expressed in mg/kg d.m.

Table 61: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Magnesium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	309	48
2	10993	48
3	6383	48
4	2521	48
5	2441	48

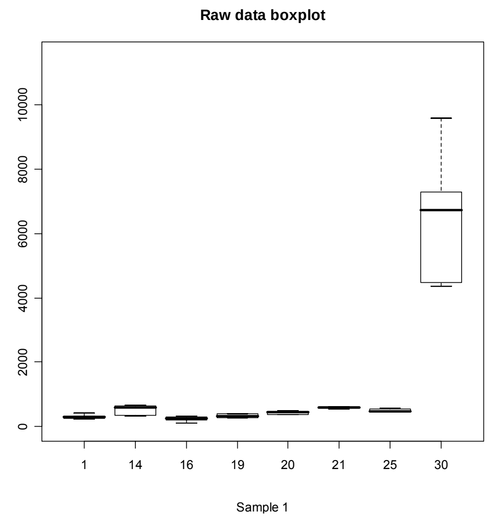
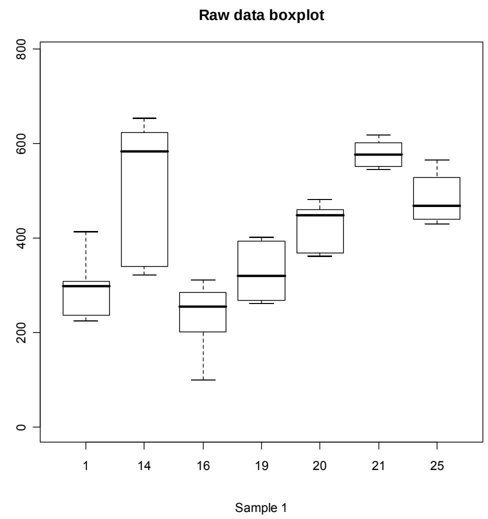
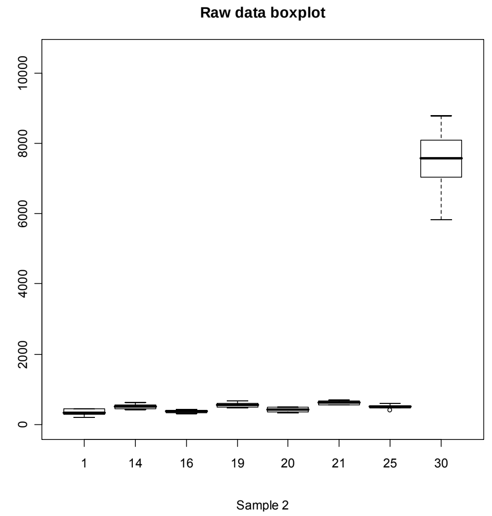
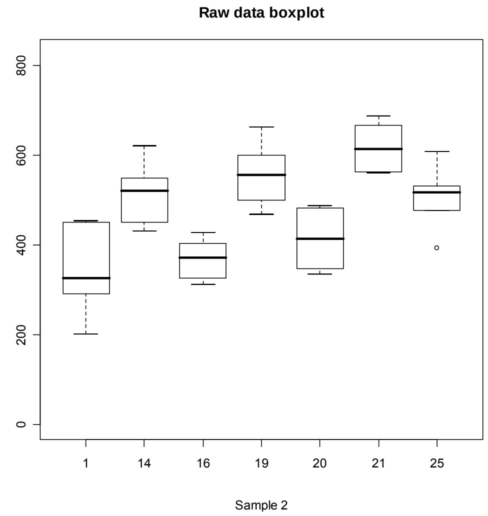
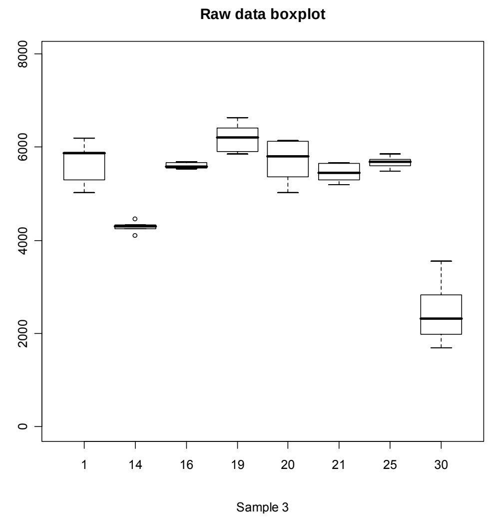
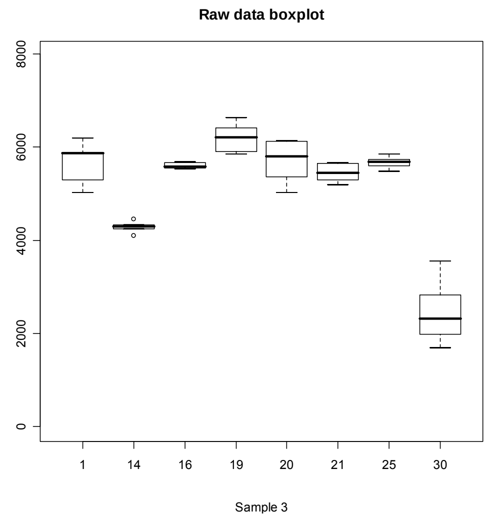
Table 62: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Magnesium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

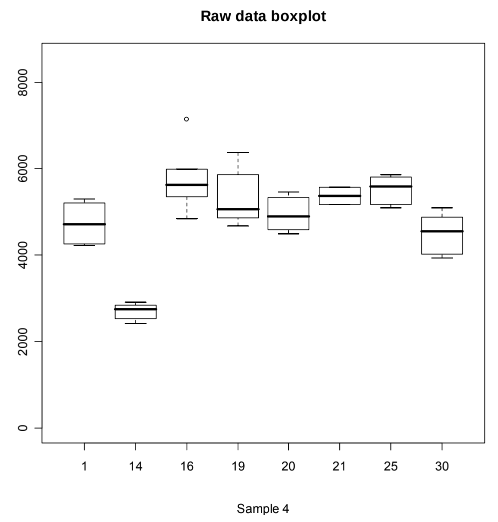
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	216044	7	36992	8	70085.27	32	288	144	24
2	134371260	7	98192243	8	46390856	32	288	144	24
3	149312149	7	5743203	8	9475838	32	288	144	24
4	13795112	7	899714	8	4931080	32	288	144	24
5	19544456	7	1494077	8	3761326	32	288	144	24

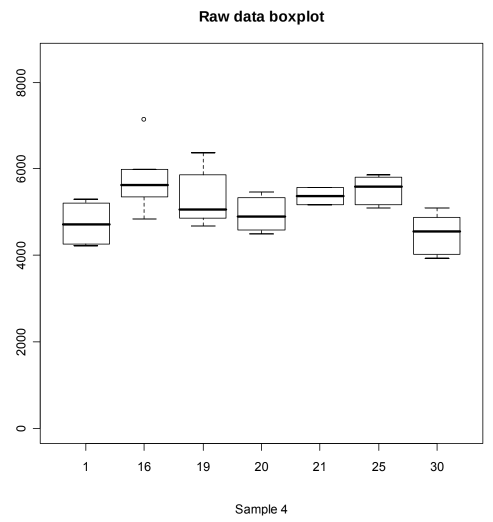
Table 63: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Magnesium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

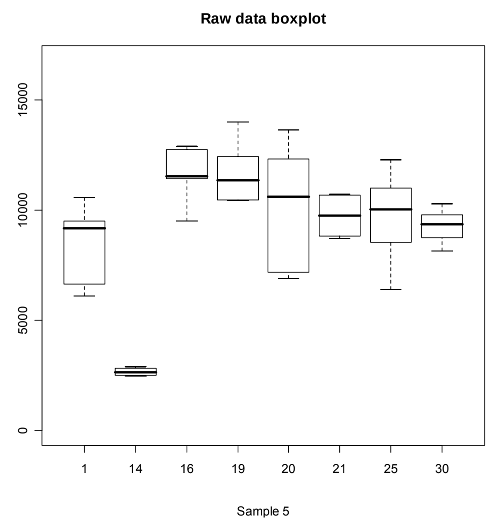
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	66	28	47	131.6	81	226.8
2	1074	1900	1204	3371.2	1613	4516.4
3	1853	375	544	1523.2	1932	5409.6
4	NA	NA	393	1100.4	393	1100.4
5	659	152	343	960.4	743	2080.4

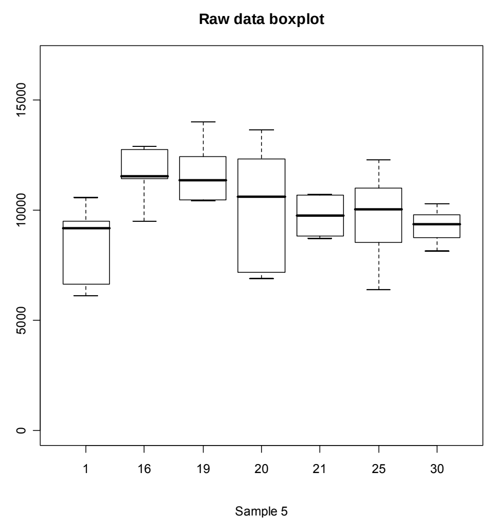
3.5.14 TS 15410 A - EN 13656 (aqua regia, HF, boric acid, micro-wave assisted digestion: Sodium)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	









Results are expressed in mg/kg d.m.

Table 64: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Sodium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	409	42
2	474	42
3	5136	48
4	5165	42
5	10112	42

Table 65: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Sodium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	574896	6	109490	9	98736.95	26	252	126	21
2	376789	6	112711.9	9	58609.17	26	252	126	21
3	62069135	7	2695133	8	2319611	32	288	144	24
4	7286446	6	567972.3	9	8566132	32	288	144	24
5	48936559	6	30605200	9	70912702	32	288	144	24

Table 66: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Sodium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	116	60	62	173.6	132	369.6
2	89	66	47	131.6	101	282.8
3	1192	297	269	753.2	1222	3421.6
4	NA	NA	517	1447.6	517	1447.6
5	792	666	1489	4169.2	1686	4720.8

3.5.15 TS 15410 A - EN 13656 (aqua regia, HF, boric acid, micro-wave assisted digestion: Phosphorus)

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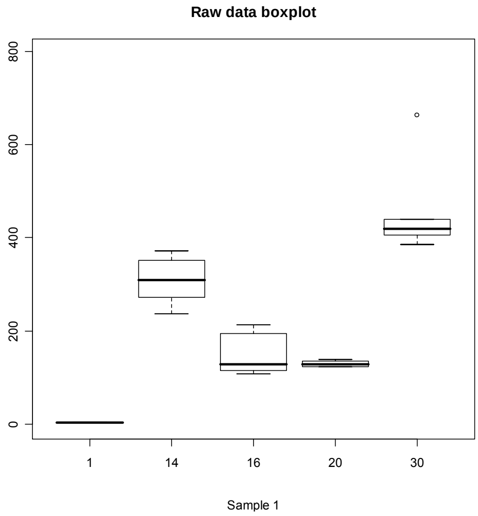	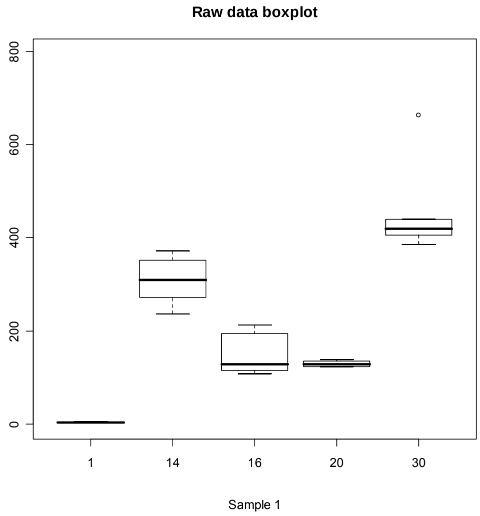
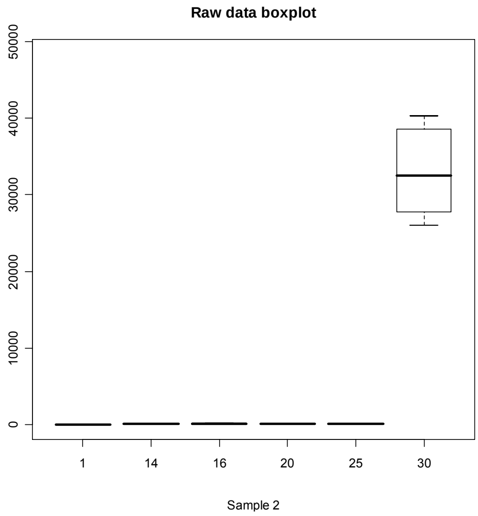	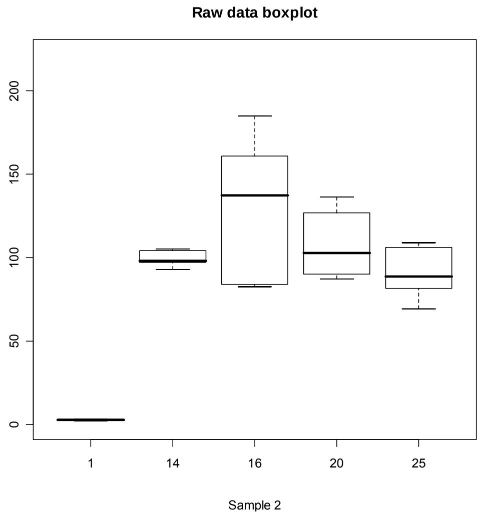
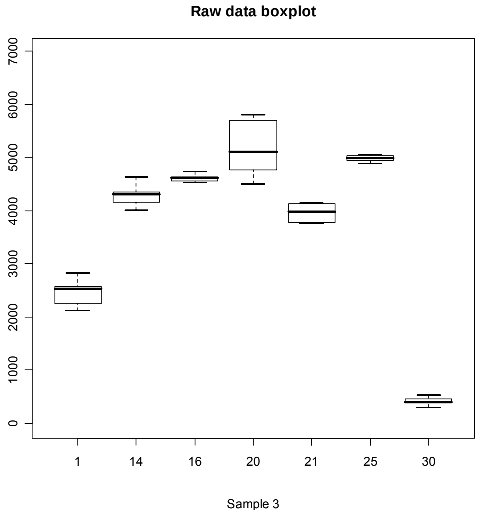	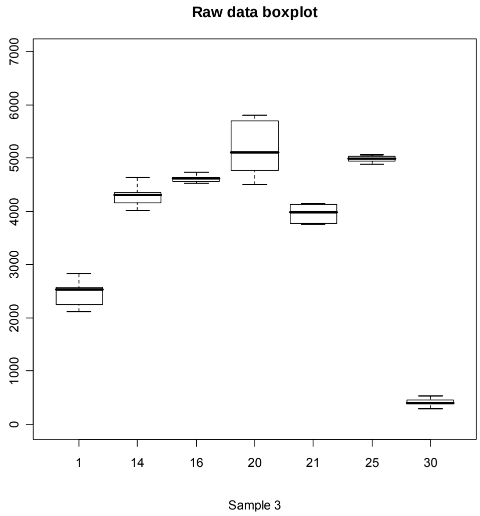

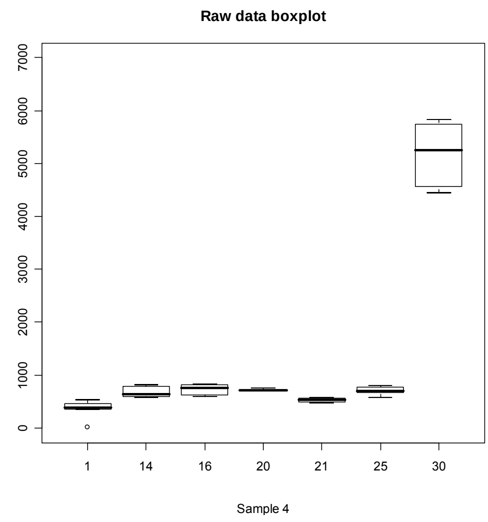

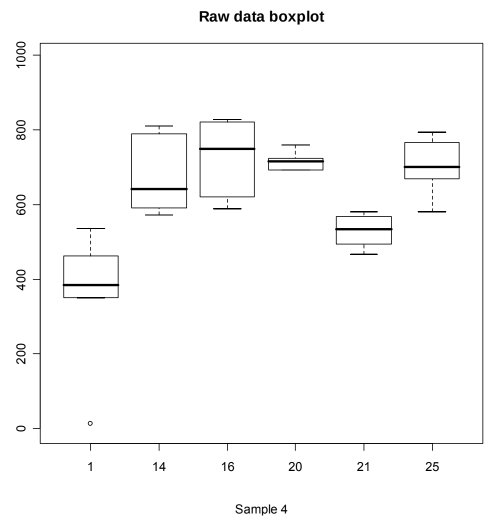

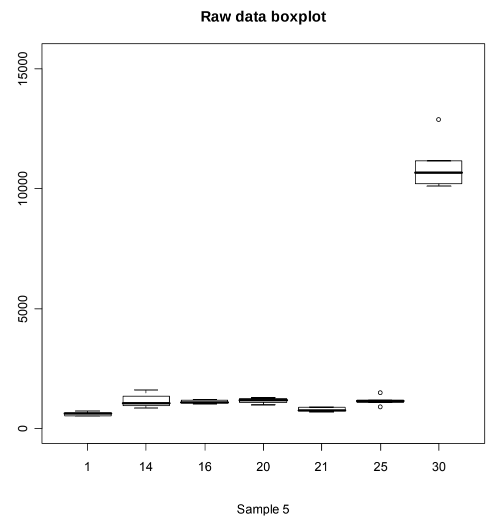

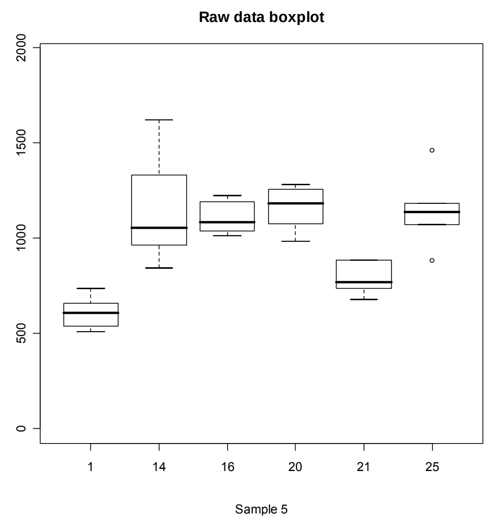

Results are expressed in mg/kg d.m.

Table 67: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Phosphorus) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	209	30
2	86	30
3	3701	42
4	618	36
5	991	36

Table 68: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Phosphorus) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS_{.Hj}$, ni_{Hj}) and for repeatability ($SS_{.rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	736712.3	4	16453.71	9	61286.92	16	180	90	15
2	57526.19	4	6910.412	9	5150.9	16	180	90	15
3	1.04E+08	6	456651	7	1656682	28	252	126	21
4	652398.3	5	58576.75	8	252815.3	28	252	126	21
5	1656797	5	89536.74	8	667330.6	28	252	126	21

Table 69: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Phosphorus) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	NA	NA	61.9	173.32	61.9	173.32
2	47	16.4	17.9	50.12	50.3	140.84
3	1699	45	243.2	680.96	1716.3	4805.64
4	NA	NA	95	266	95	266
5	NA	NA	154.4	432.32	154.4	432.32

3.5.16 TS 15410 A - EN 13656 (aqua regia, HF, boric acid, micro-wave assisted digestion: Silicon)

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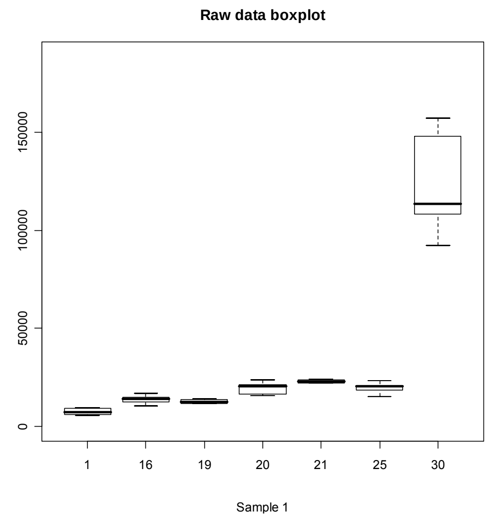

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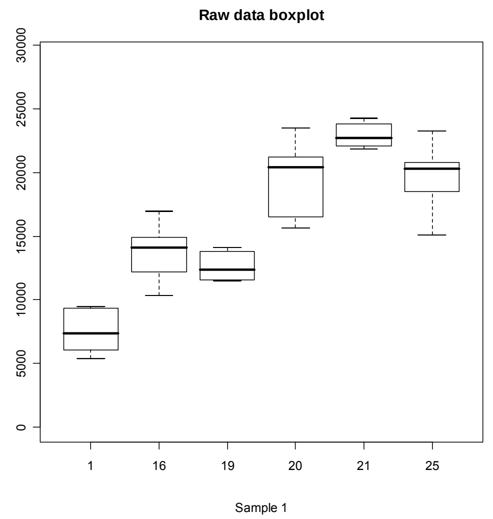

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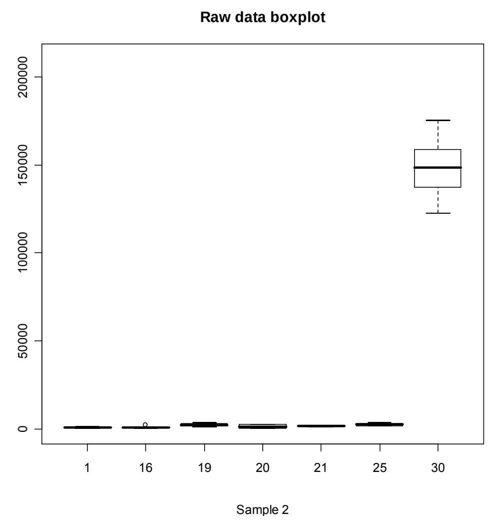

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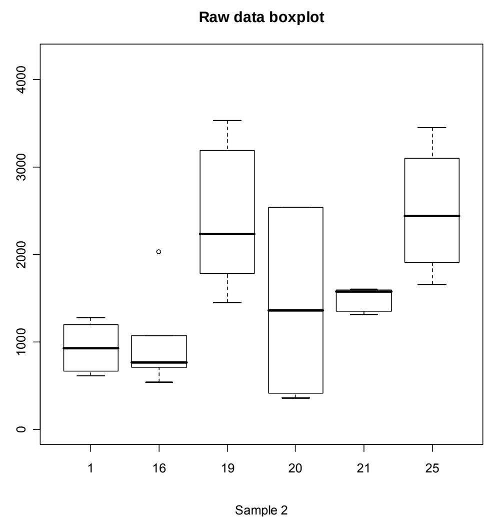

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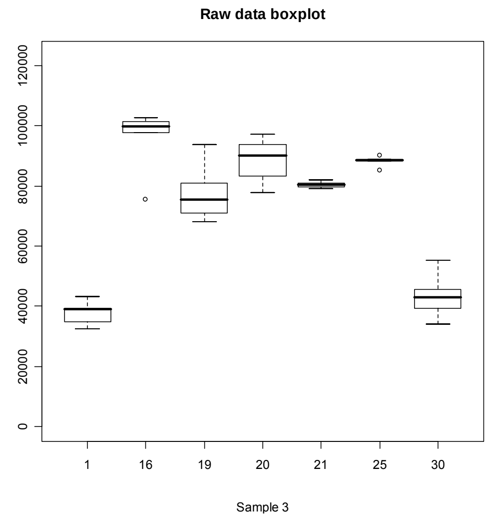

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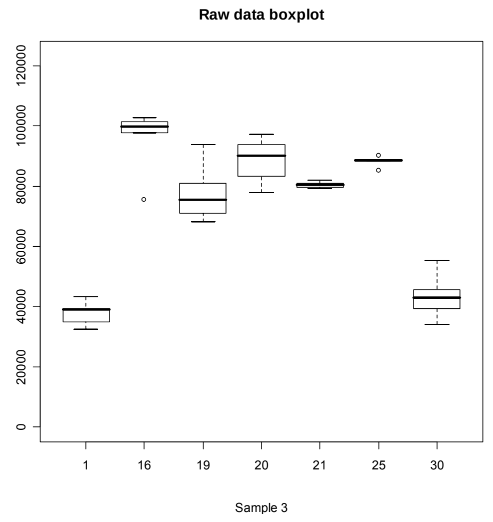

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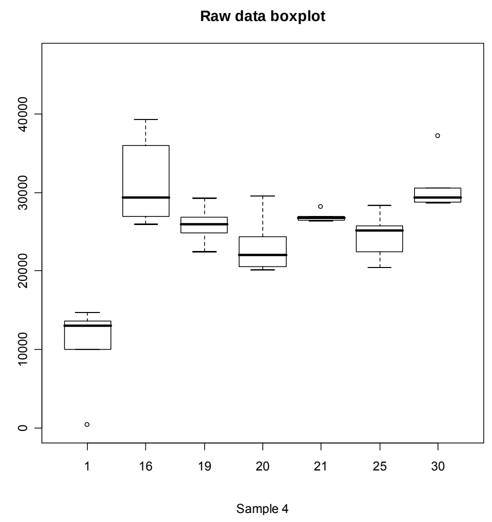
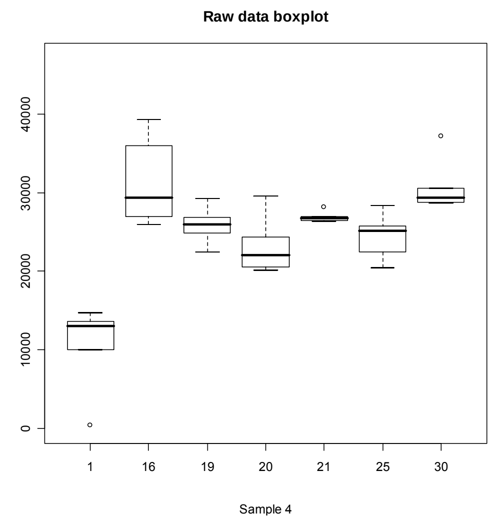
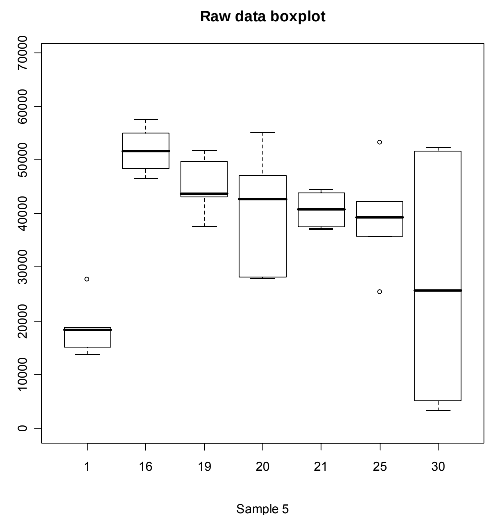
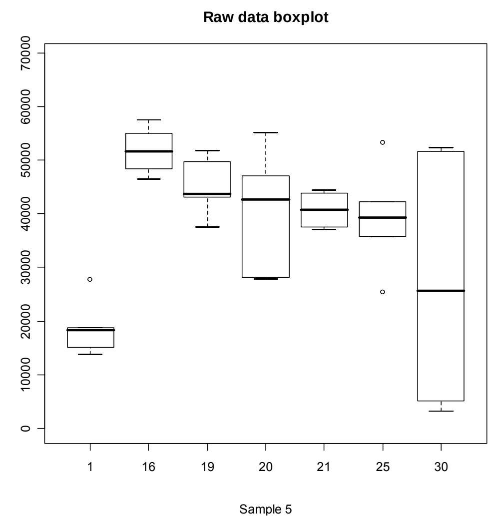








Results are expressed in mg/kg d.m.

Table 70: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Silicon) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	16266	35
2	1650	35
3	73228	42
4	24713	42
5	37587	42

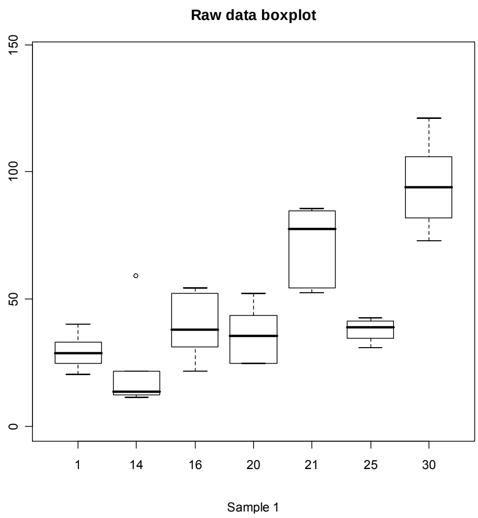
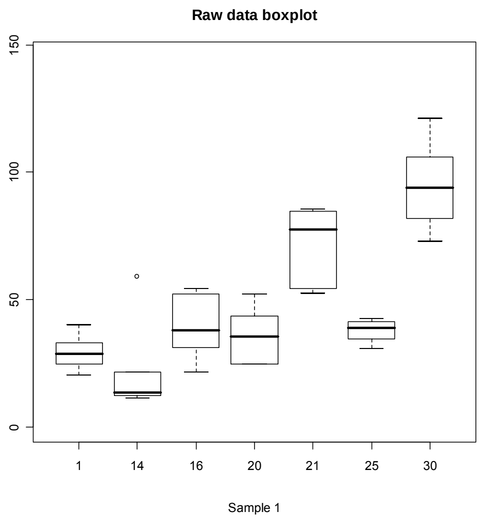
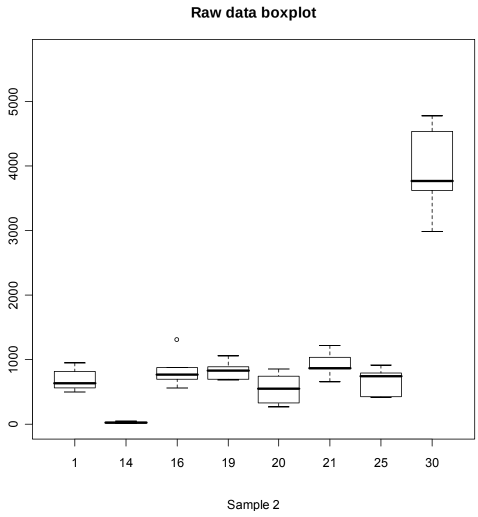
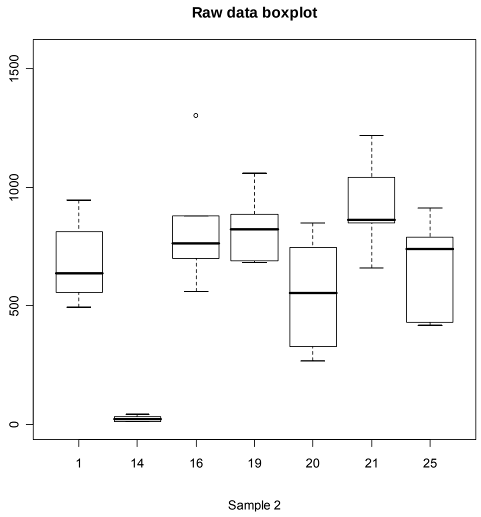
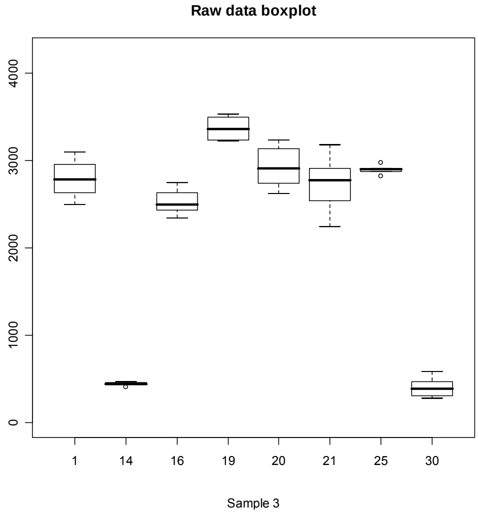
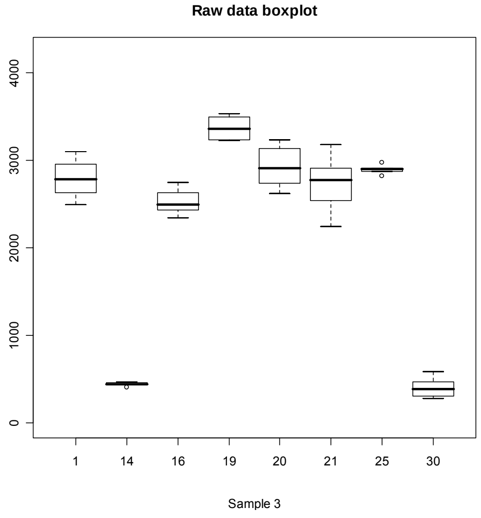
Table 71: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Silicon) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

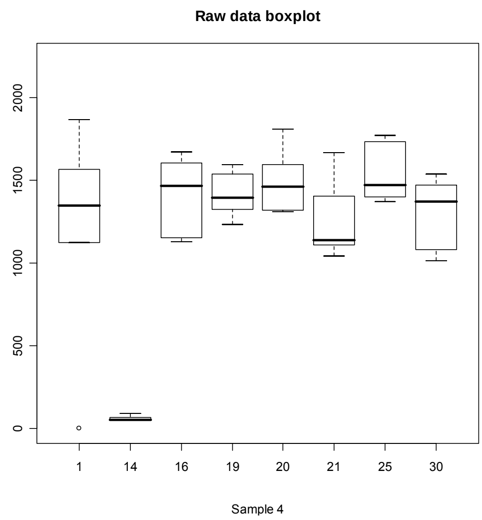
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	9.04E+08	5	64186202	8	71004151	21	205	103	17.6
2	13218821	5	11475002	8	2343616	21	205	103	17.6
3	1.92E+10	6	5.4E+08	7	1E+09	28	252	126	21
4	1.67E+09	6	34938121	7	4.37E+08	28	252	126	21
5	4.44E+09	6	3.48E+09	7	1.09E+09	28	252	126	21

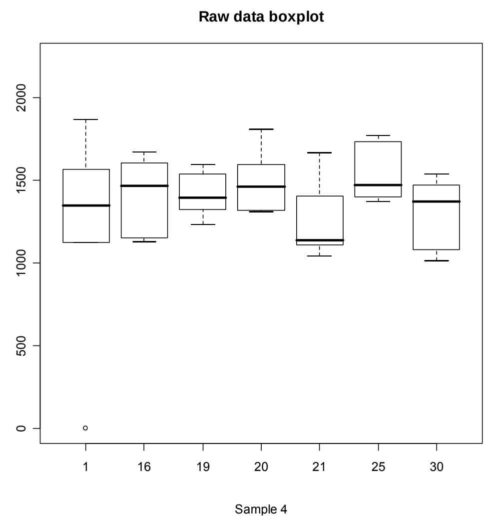
Table 72: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Silicon) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

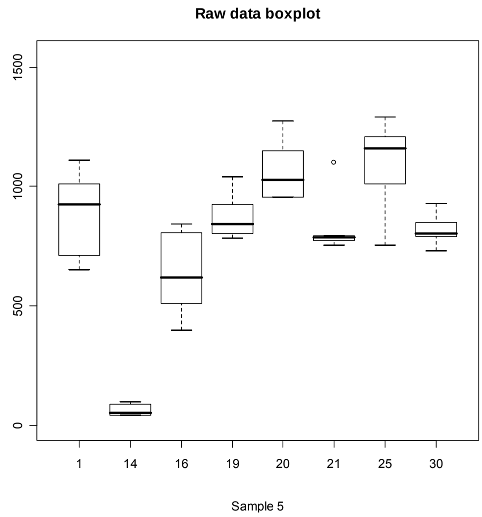
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	5420	1461	1839	5149.2	5723	16024.4
2	359	780	334	935.2	490	1372
3	22811	3714	5979	16741.2	23582	66029.6
4	NA	NA	3952	11065.6	3952	11065.6
5	6362	12351	6236	17460.8	8909	24945.2

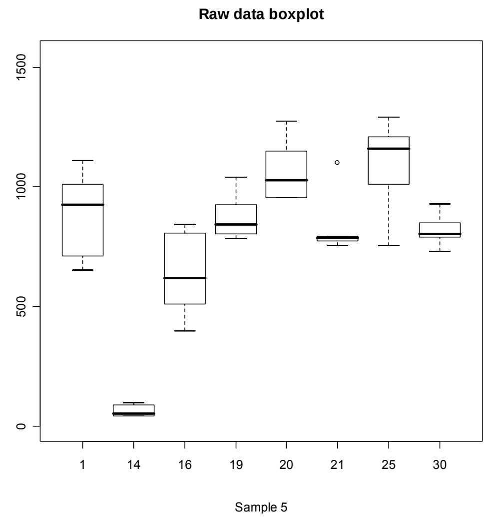
3.5.17 TS 15410 A - EN 13656 (aqua regia, HF, boric acid, micro-wave assisted digestion: Titanium)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	









Results are expressed in mg/kg d.m.

Table 73: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Silicon: Titanium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	47	42
2	643	42
3	2262	48
4	1374	42
5	784	48

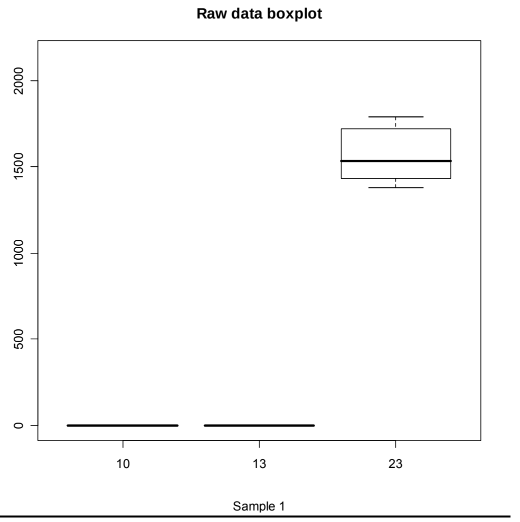
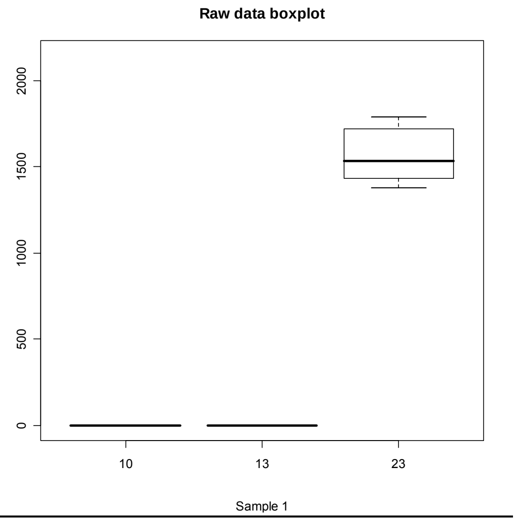
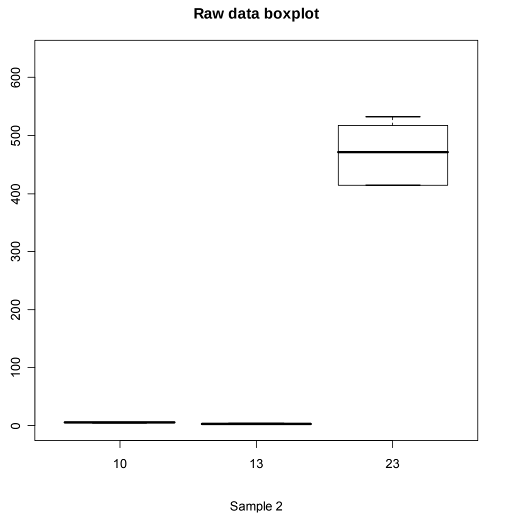
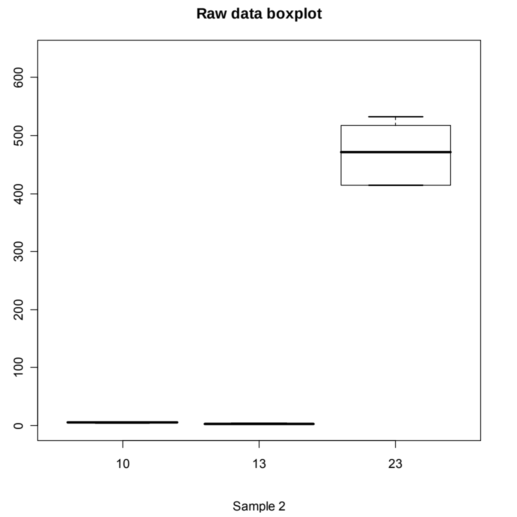
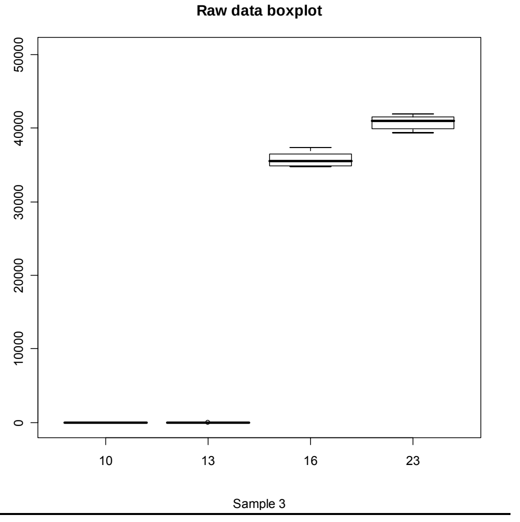
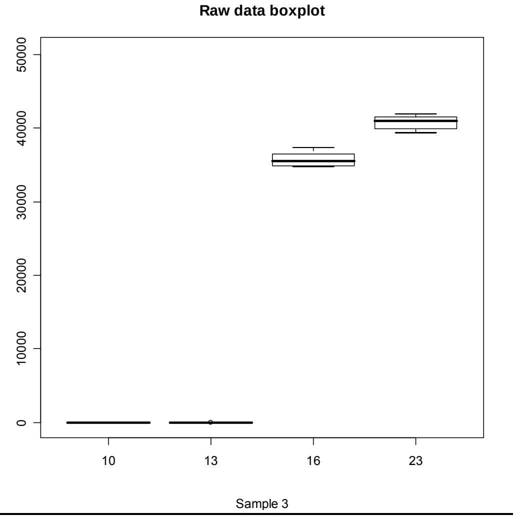
Table 74: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Silicon: Titanium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

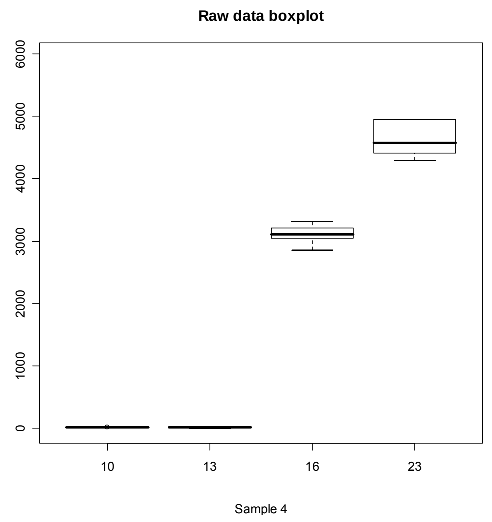
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	24823	6	2122.4	9	4001.5	26	252	126	21
2	3216120	6	733678.7	9	506631.2	26	252	126	21
3	56590761	7	610586	8	706873.9	32	288	144	24
4	546347.3	6	207794.1	9	3167053	32	288	144	24
5	4448069	7	155929	8	564772.7	32	288	144	24

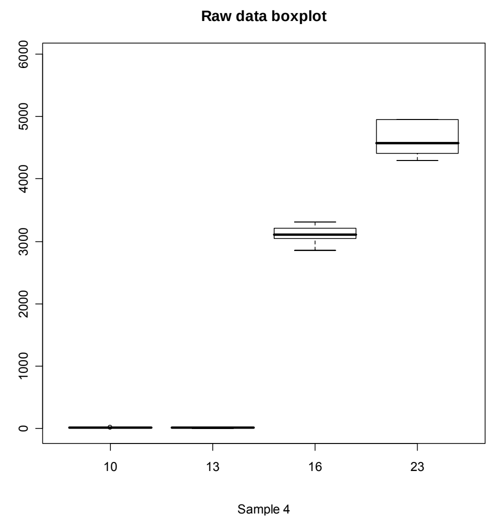
Table 75: TS 15410 A (aqua regia, HF, boric acid, micro-wave assisted digestion: Silicon: Titanium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

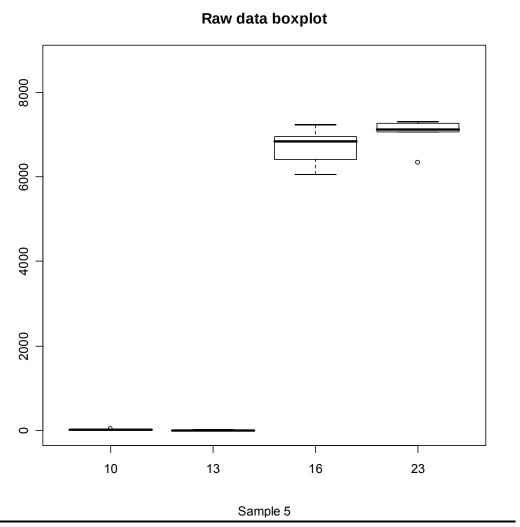
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	25	6	12	33.6	28	78.4
2	270	163	140	392	304	851.2
3	1155	134	149	417.2	1165	3262
4	NA	NA	315	882	315	882
5	320	25	133	372.4	347	971.6

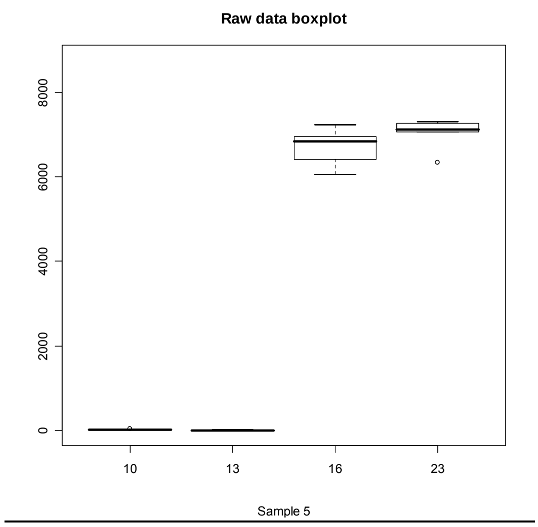
3.5.18 TS 15410 B (Ashing (ASTM D6722 and XRF): Aluminium)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	









N.B. Data set to small for meaningful outlier testing

Results are expressed in mg/kg d.m.

Table 76: TS 15410 B (Ashing (ASTM D6722 and XRF): Aluminium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	626	15
2	204	14
3	19132	24
4	1936	24
5	3446	24

Table 77: TS 15410 B (Ashing (ASTM D6722 and XRF): Aluminium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj}) .

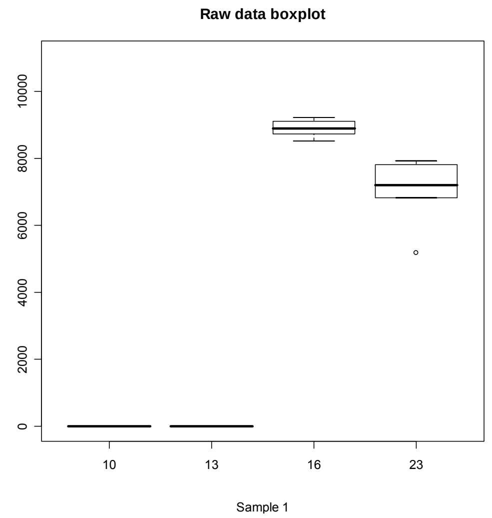
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	8796090	2	136504.2	4	13346.67	8	81	45	9
2	743687.6	2	14018.65	5	717.34	6	76	38	7
3	8.85E+09	3	5783100	4	4697960	16	144	72	12
4	96100231	3	353016.4	4	151487.1	16	144	72	12
5	2.83E+08	3	486802.9	4	1012914	16	144	72	12

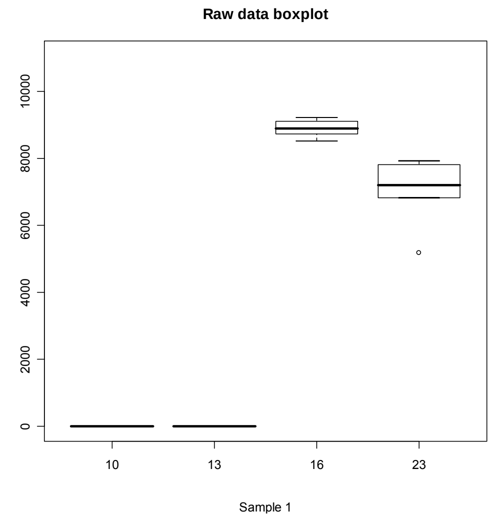
Table 78: TS 15410 B (Ashing (ASTM D6722 and XRF): Aluminium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

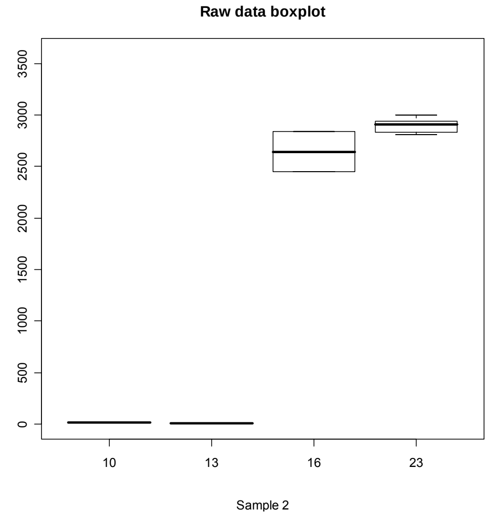
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	950	147	41	114.8	951	2662.8
2	293	44	11	30.8	293	820.4
3	22165	620	542	1517.6	22172	62081.6
4	2307	162	97	271.6	2309	6465.2
5	3964	140	252	705.6	3972	11121.6

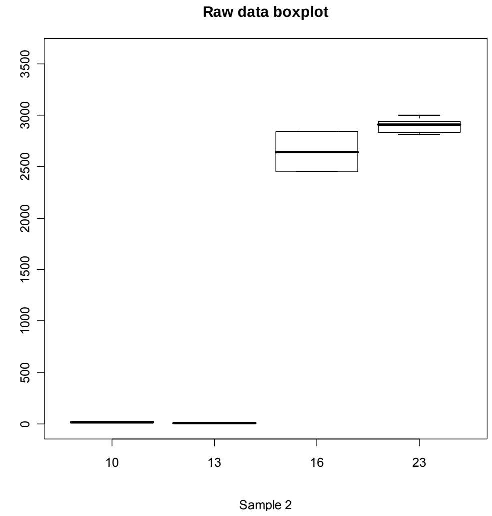
3.5.19 TS 15410 B (Ashing (ASTM D6722 + XRF): Calcium)

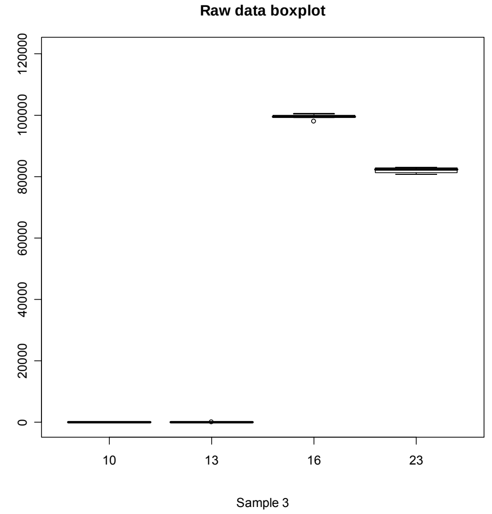
Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*

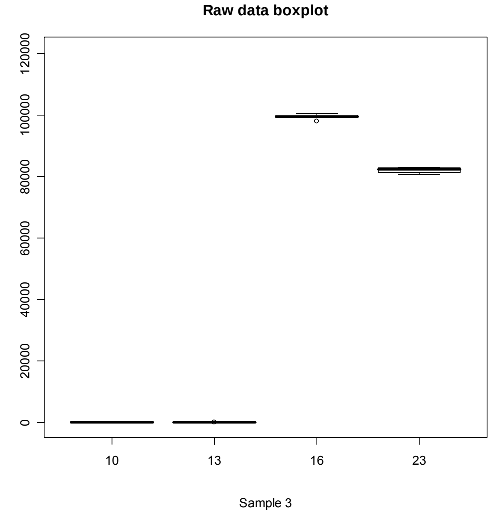












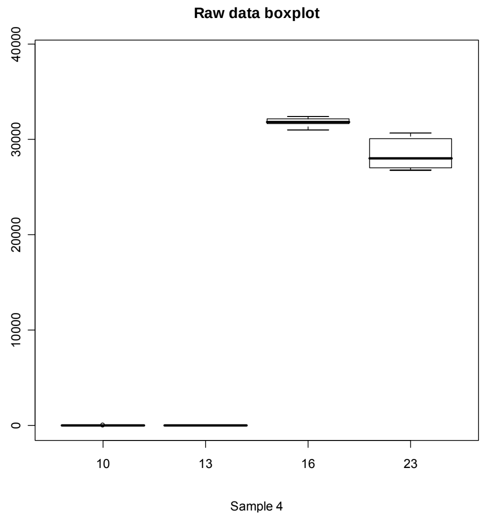
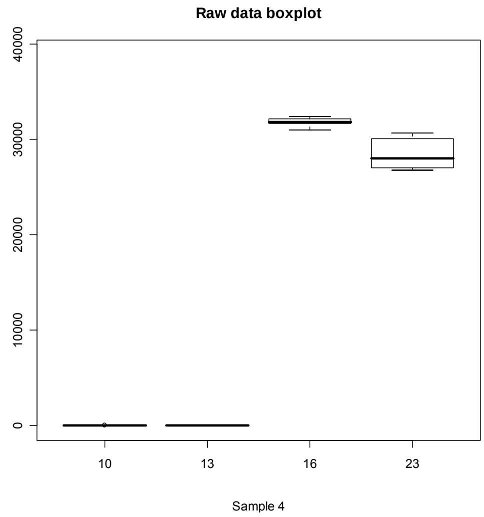
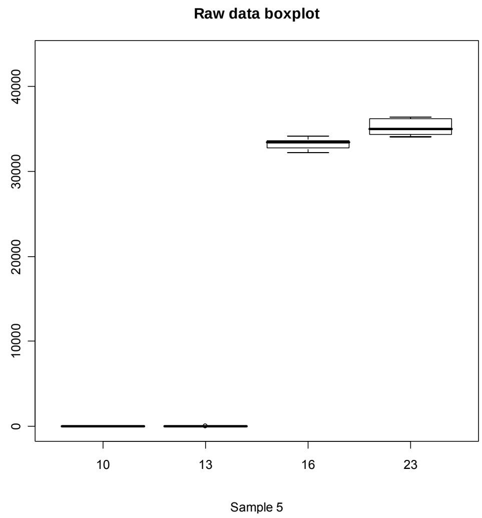
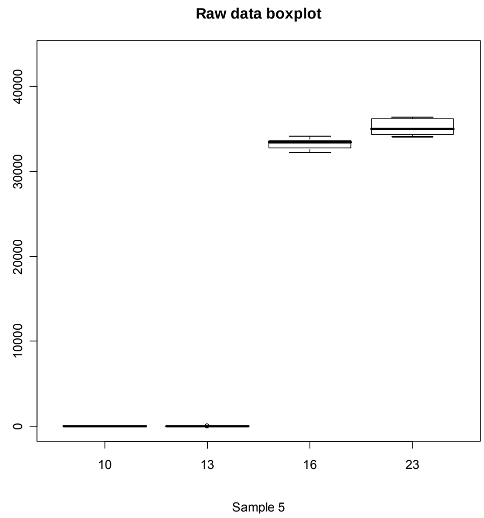
	
	
	N.B. Data set to small for meaningful outlier testing *Results are expressed in mg/kg d.m.*

Table 79: TS 15410 B (Ashing (ASTM D6722 + XRF): Calcium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	4545	21
2	1423	16
3	45368	24
4	15076	24
5	17113	24

Table 80: TS 15410 B (Ashing (ASTM D6722 + XRF): Calcium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	3.35E+08	3	3462440	3	2090907	14	117	63	12
2	31929229	3	76276.17	4	23466.67	8	80	40	8
3	5.03E+10	3	5364729	4	3144986	16	144	72	12
4	5.47E+09	3	13426993	4	2183622	16	144	72	12
5	7.02E+09	3	5189427	4	1558624	16	144	72	12

Table 81: TS 15410 B (Ashing (ASTM D6722 + XRF): Calcium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	4636	579	386	1080.8	4652	13025.6
2	1702	90	54	151.2	1703	4768.4
3	52843	618	443	1240.4	52845	147966
4	17423	1036	369	1033.2	17427	48795.6
5	19749	632	312	873.6	19752	55305.6

3.5.20 TS 15410 B (Ashing (ASTM D6722 + XRF): Iron)

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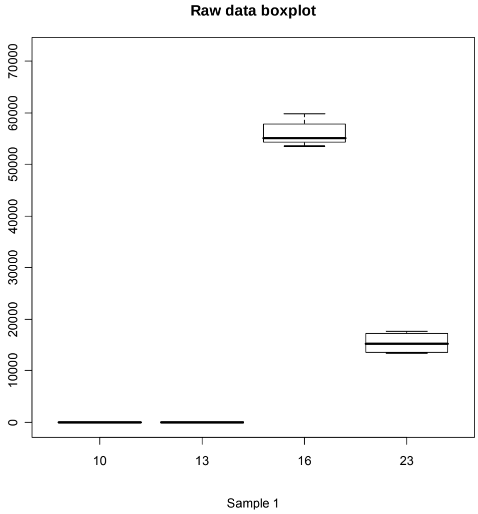
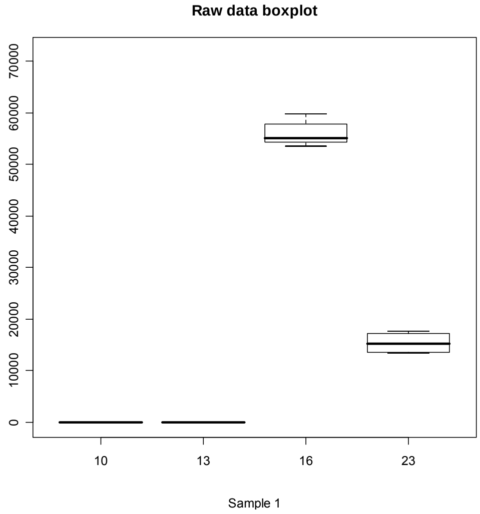
<tr><td>
</td><td>
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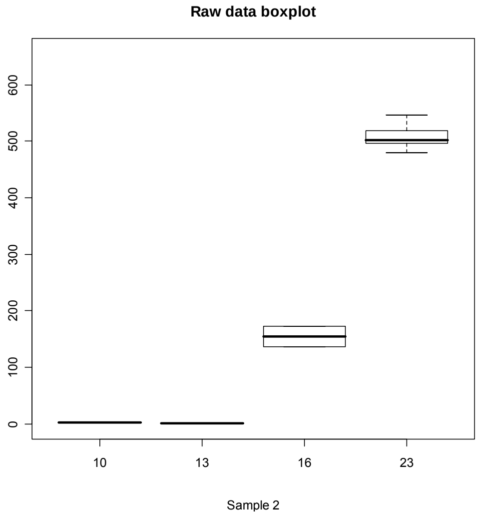
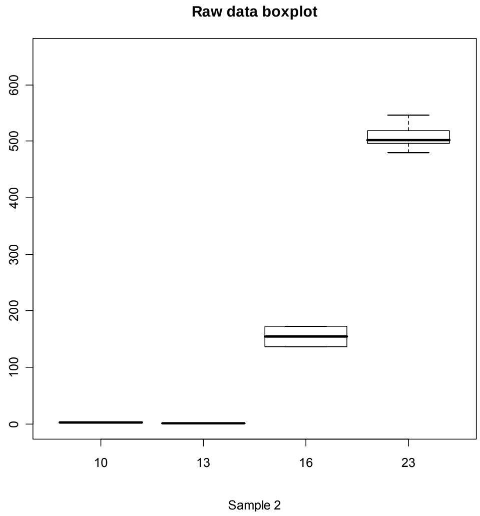
<tr><td>
</td><td>
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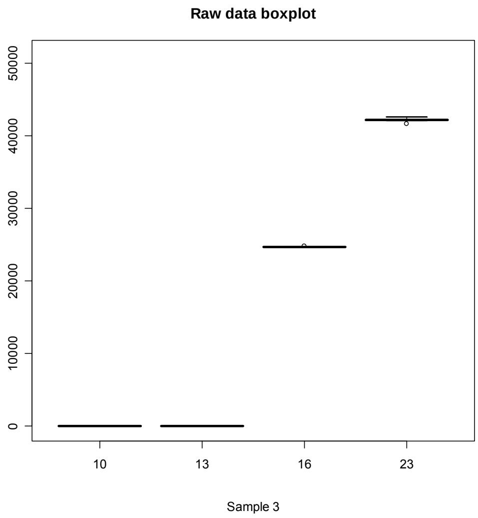
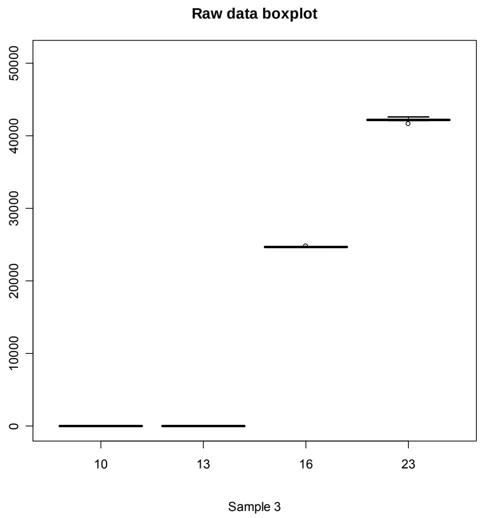
<tr><td>
</td><td>
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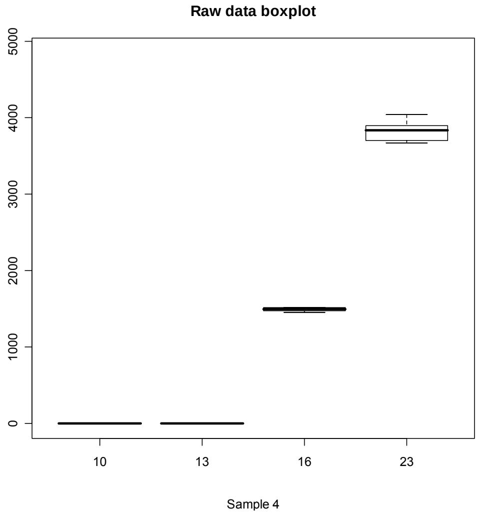
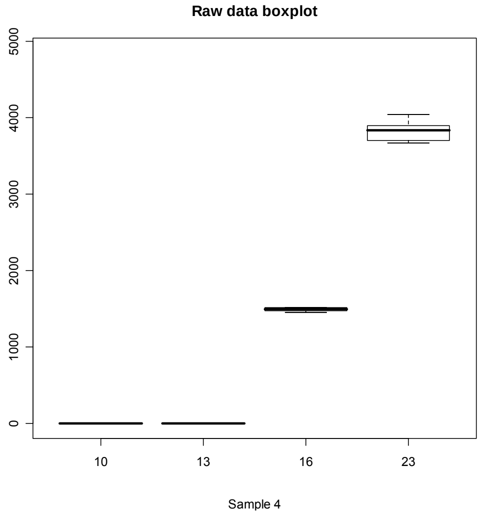
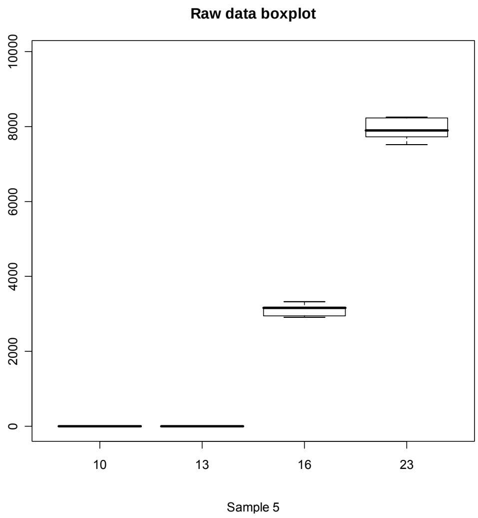
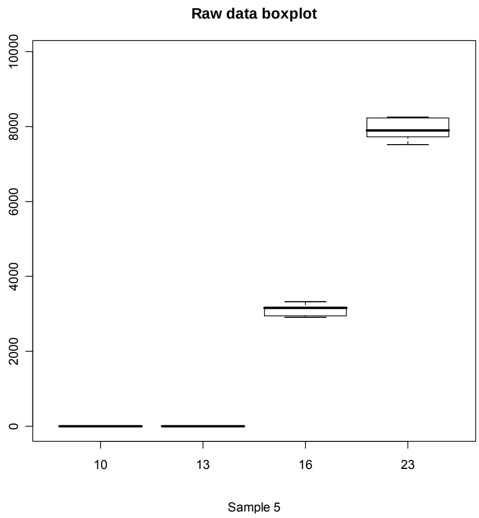
	
	
	N.B. Data set to small for meaningful outlier testing *Results are expressed in mg/kg d.m.*

Table 82: TS 15410 B (Ashing (ASTM D6722 + XRF): Iron) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	20374	21
2	211	16
3	16710	24
4	1331	24
5	2758	24

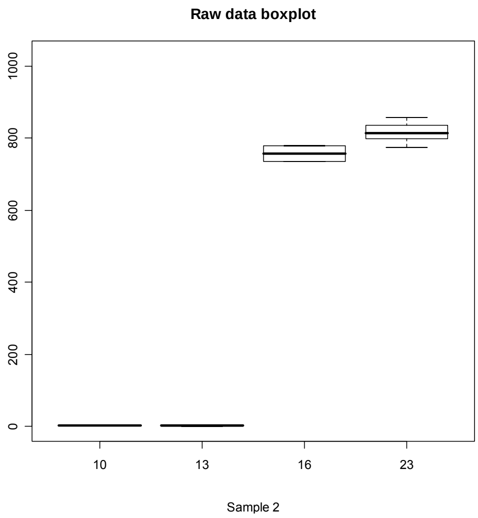
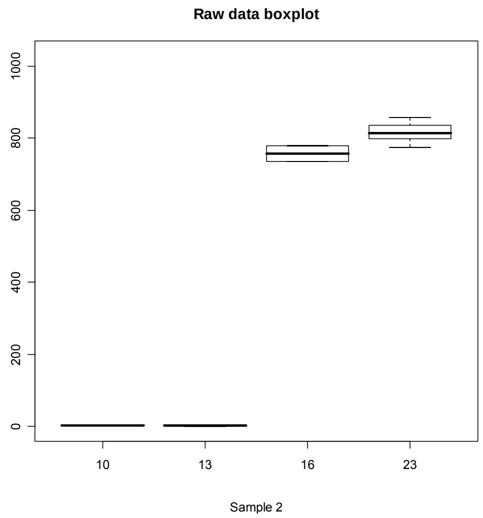
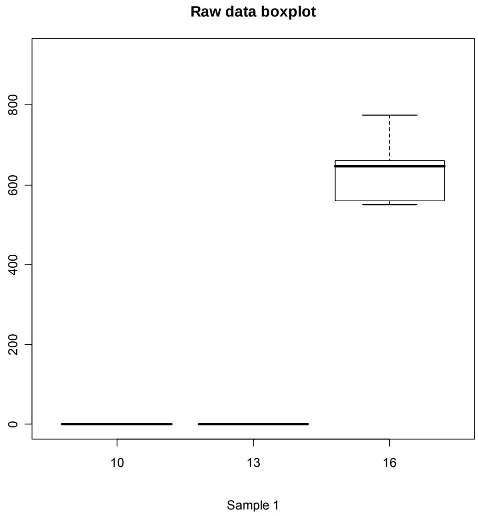
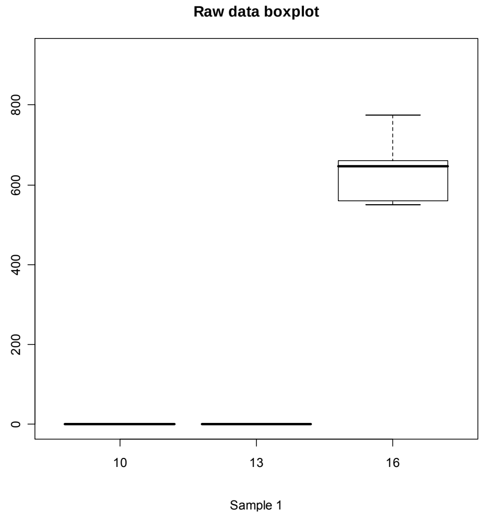
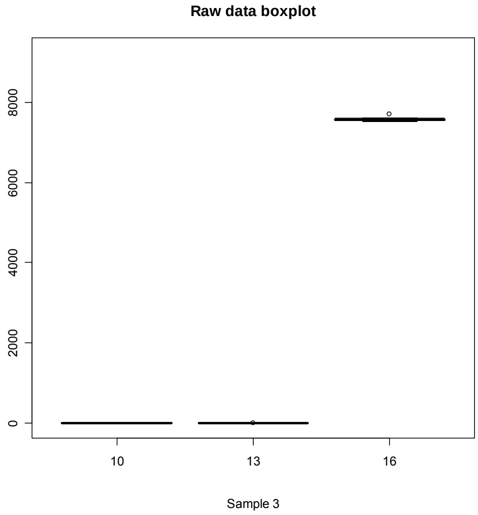
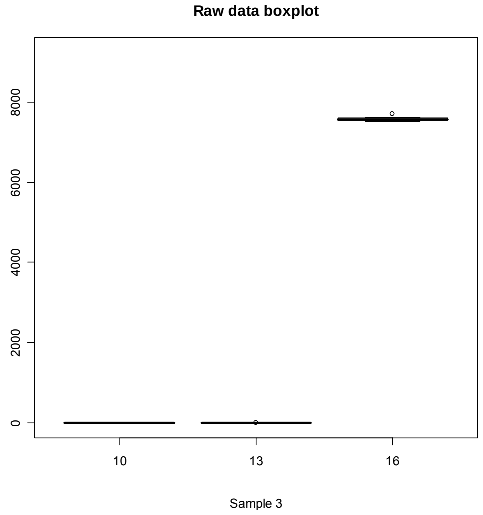
Table 83: TS 15410 B (Ashing (ASTM D6722 + XRF): Iron) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

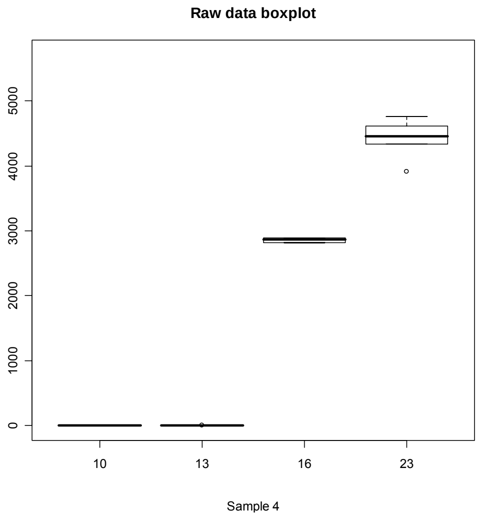
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	1.14E+10	3	20181416	3	28256558	14	117	63	12
2	882034.1	3	2354.466	4	946.6667	8	80	40	8
3	7.62E+09	3	149636.8	4	400846.3	16	144	72	12
4	58777057	3	37489.99	4	56330.38	16	144	72	12
5	2.51E+08	3	393623.5	4	144416.2	16	144	72	12

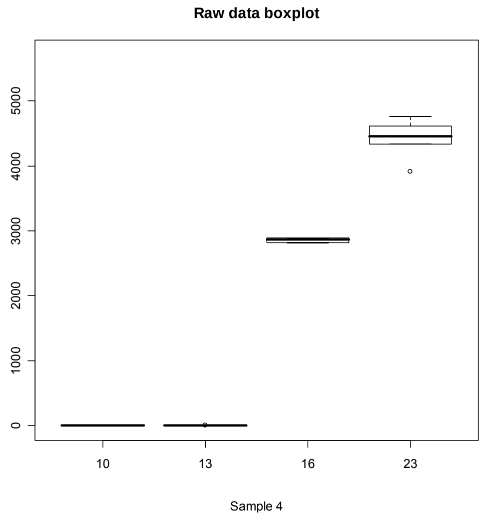
Table 84: TS 15410 B (Ashing (ASTM D6722 + XRF): Iron) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

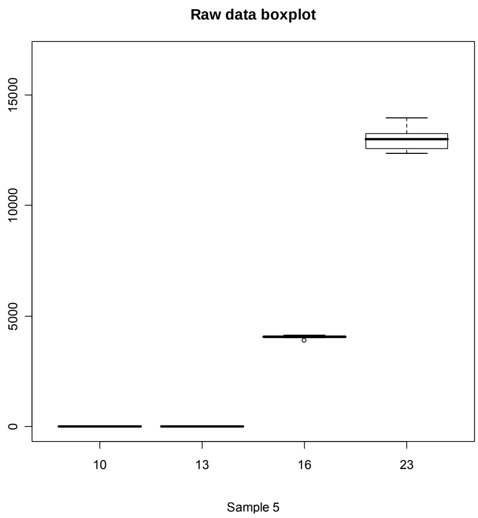
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	27207	1253	1421	3978.8	27245	76286
2	283	15	11	30.8	283	792.4
3	20569	64	158	442.4	20570	57596
4	1807	44	59	165.2	1808	5062.4
5	3735	173	95	266	3736	10460.8

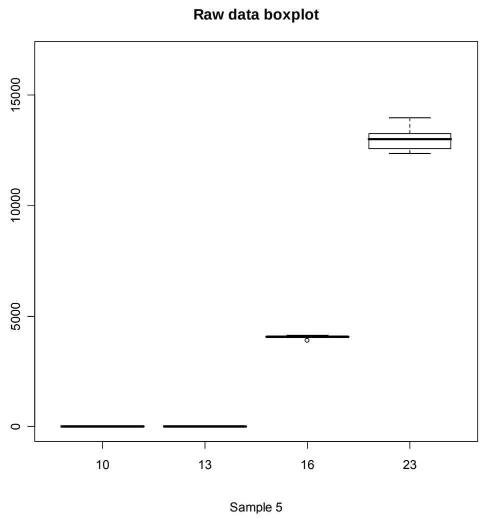
3.5.21 TS 15410 B (Ashing (ASTM D6722 + XRF): Potassium)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	









N.B. Data set to small for meaningful outlier testing

Results are expressed in mg/kg d.m.

Table 85: TS 15410 B (Ashing (ASTM D6722 + XRF): Potassium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	256	15
2	401	16
3	2529	18
4	1821	24
5	4268	24

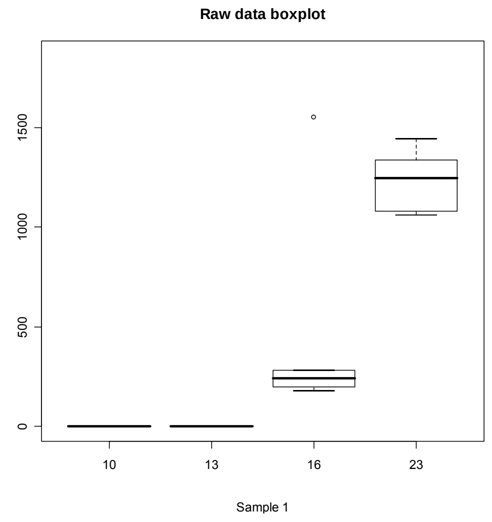
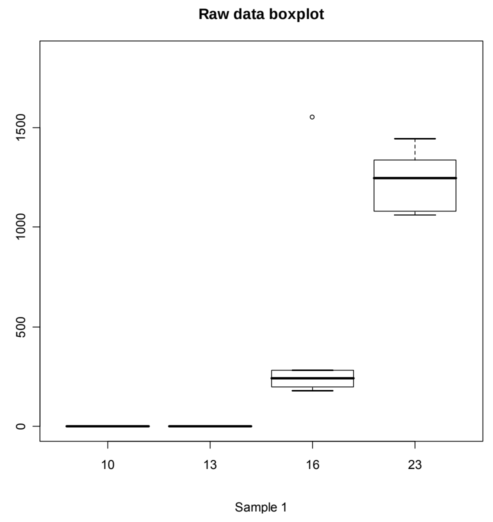
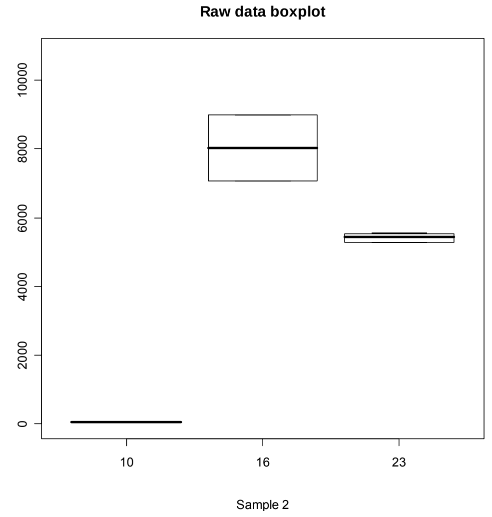
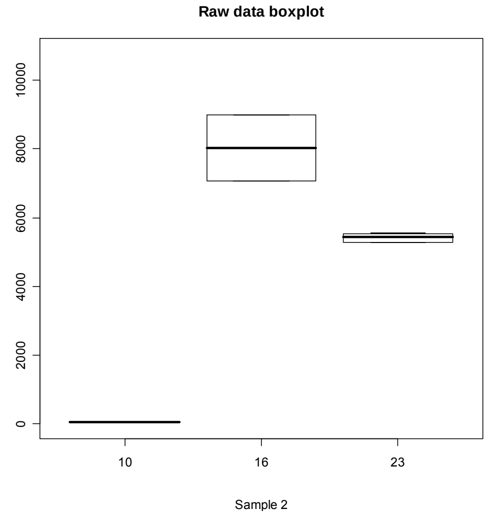
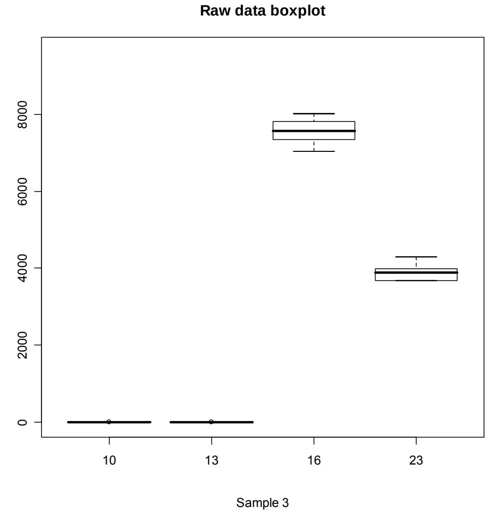
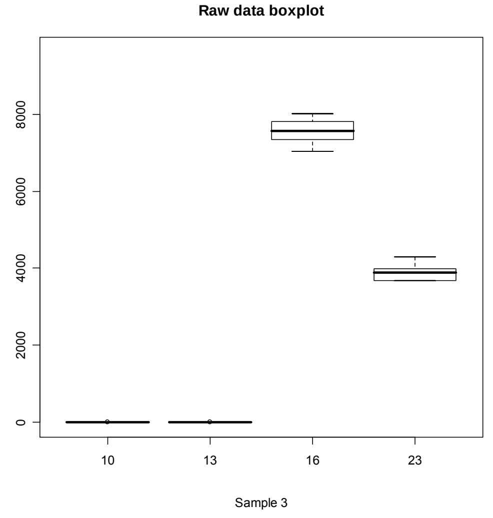
Table 86: TS 15410 B (Ashing (ASTM D6722 + XRF): Potassium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

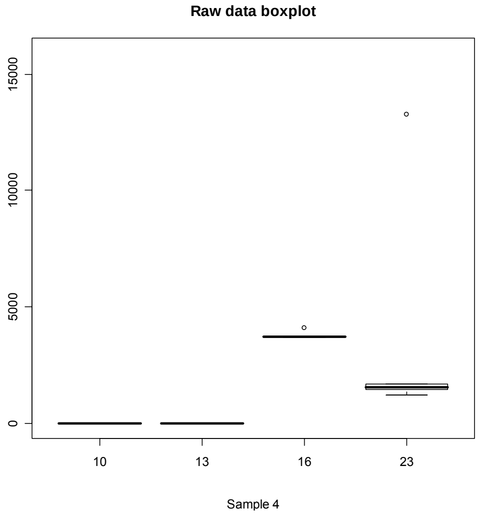
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	1470699	2	777.7281	4	32451.67	8	81	45	9
2	2556425	3	3827.169	4	1371.333	8	80	40	8
3	2.3E+08	2	6209.876	5	12045.26	16	144	72	12
4	86748539	3	44711.45	4	391122.7	16	144	72	12
5	6.78E+08	3	1170795	4	472707.3	16	144	72	12

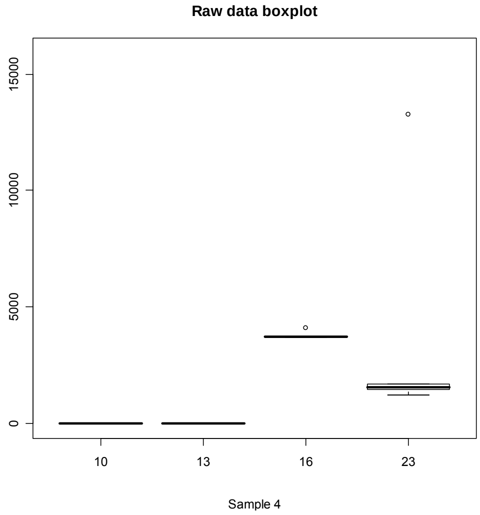
Table 87: TS 15410 B (Ashing (ASTM D6722 + XRF): Potassium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

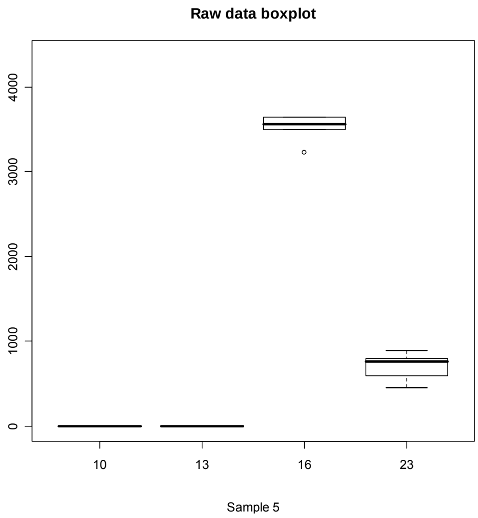
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	NA	NA	64	179.2	64	179.2
2	482	20	13	36.4	482	1349.6
3	3574	14	27	75.6	3574	10007.2
4	NA	NA	156	436.8	156	436.8
5	6135	296	172	481.6	6137	17183.6

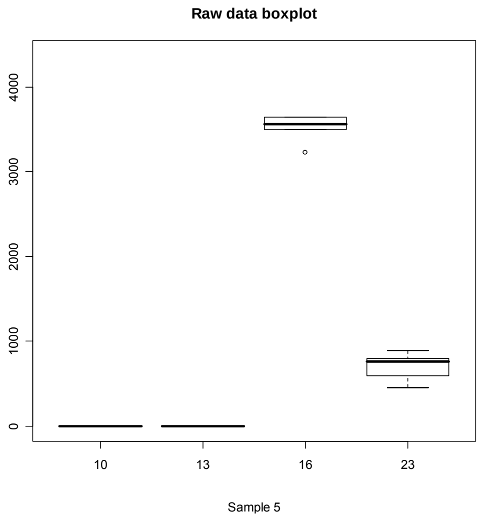
3.5.22 TS 15410 B (Ashing (ASTM D6722 + XRF): Magnesium)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	









N.B. Data set to small for meaningful outlier testing

Results are expressed in mg/kg d.m.

Table 88: TS 15410 B (Ashing (ASTM D6722 + XRF): Magnesium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	481	21
2	3490	14
3	2866	24
4	1805	24
5	1058	24

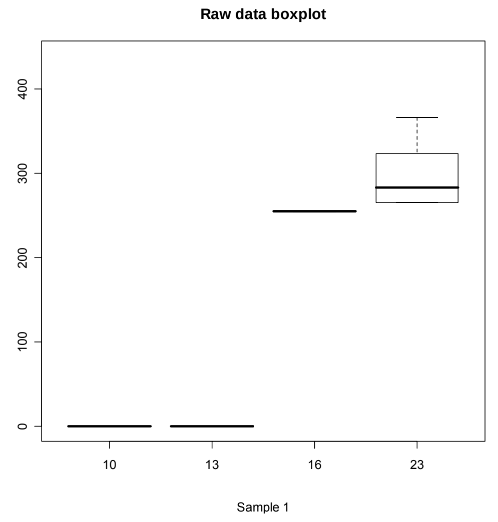
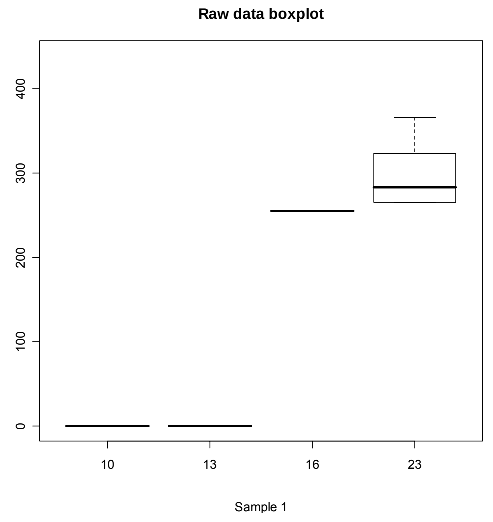
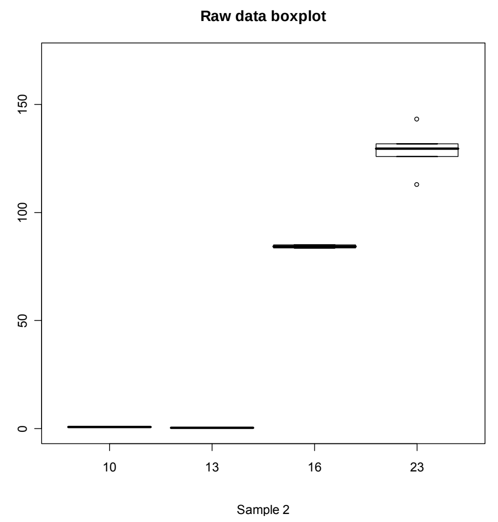
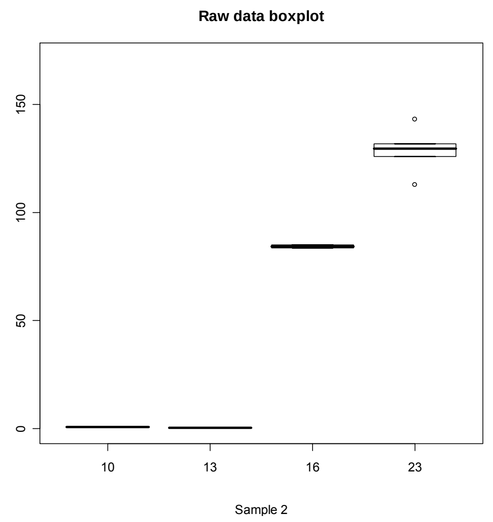
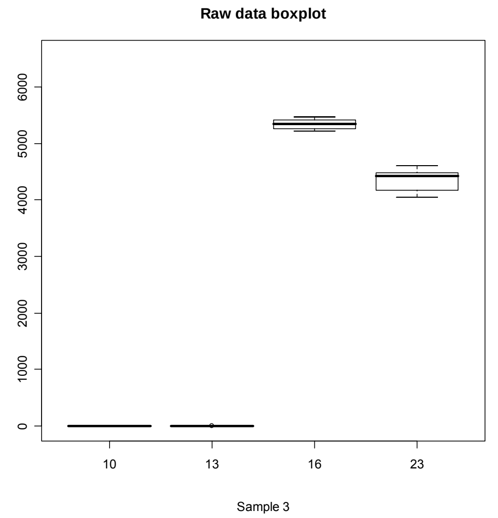
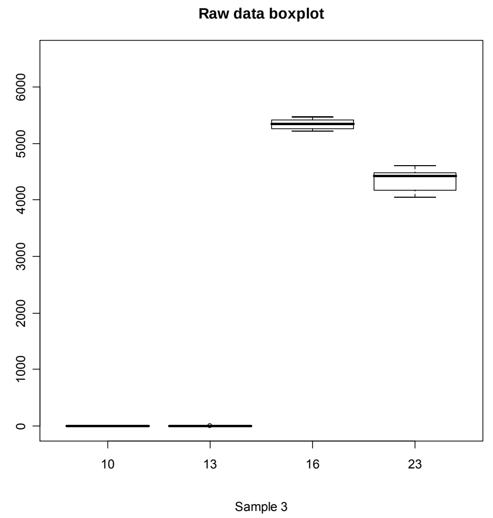
Table 89: TS 15410 B (Ashing (ASTM D6722 + XRF): Magnesium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

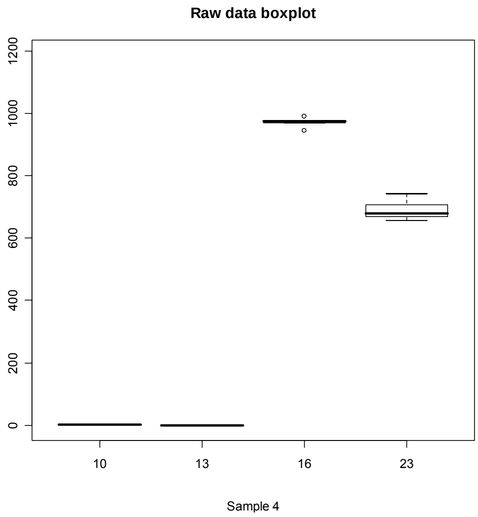
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	5495627	3	465322	3	1108439	14	117	63	12
2	1.35E+08	2	1828765	5	72722.67	12	112	56	10
3	2.37E+08	3	580986.5	4	355146	16	144	72	12
4	78209276	3	20573886	4	94758701	16	144	72	12
5	50457659	3	119968.6	4	125338.6	16	144	72	12

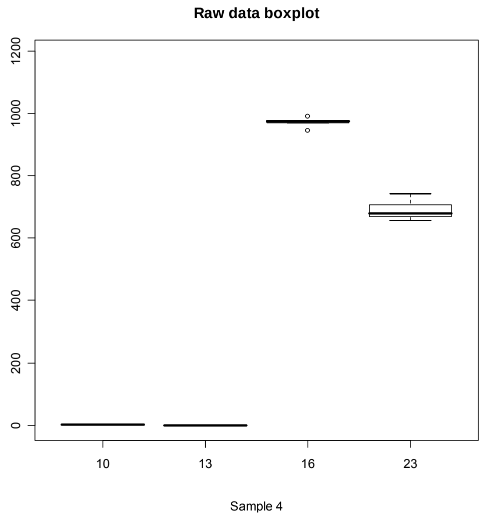
Table 90: TS 15410 B (Ashing (ASTM D6722 + XRF): Magnesium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

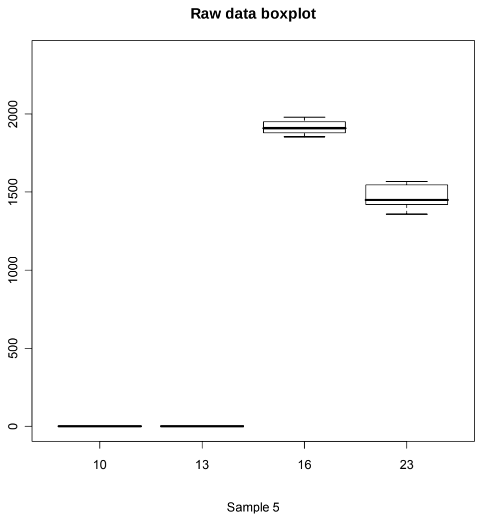
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	571	159	281	786.8	637	1783.6
2	3047	424	78	218.4	3048	8534.4
3	3623	203	149	417.2	3626	10152.8
4	NA	NA	2434	6815.2	2434	6815.2
5	1673	86	89	249.2	1675	4690

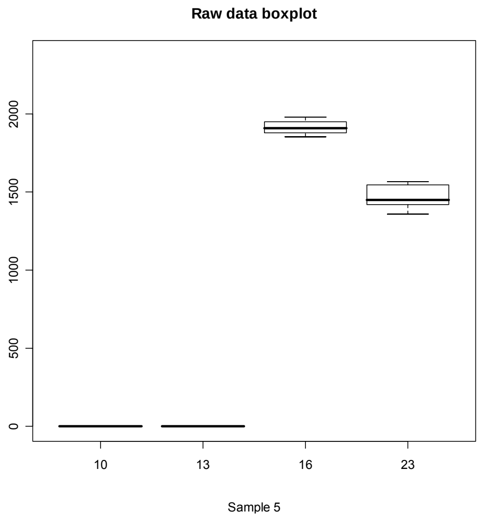
3.5.23 TS 15410 B (Ashing (ASTM D6722 + XRF): Phosphorus)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	









N.B. Data set to small for meaningful outlier testing

Results are expressed in mg/kg d.m.

Table 91: TS 15410 B (Ashing (ASTM D6722 + XRF): Phosphorus) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	158	21
2	59	16
3	2426	24
4	415	24
5	845	24

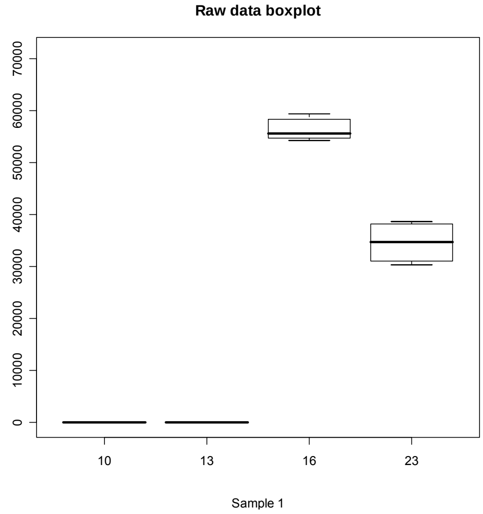
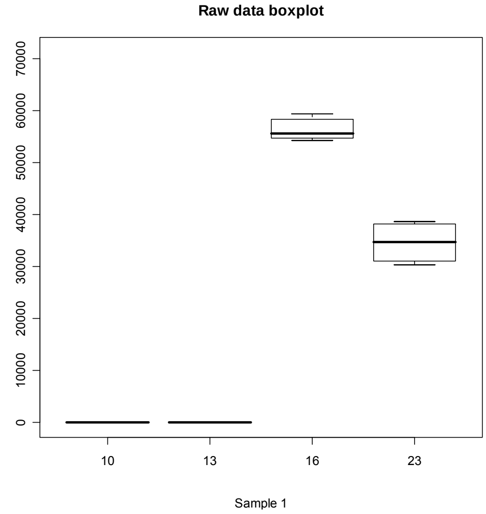
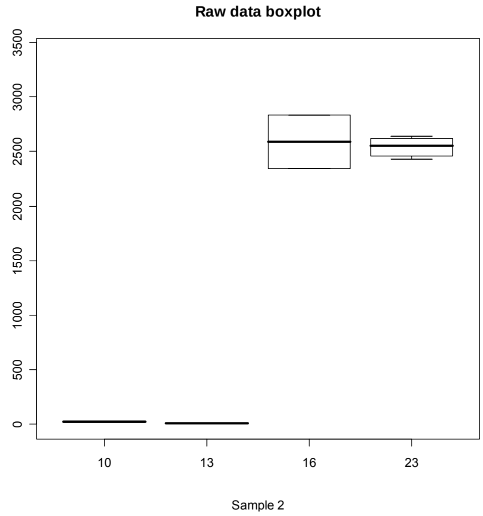
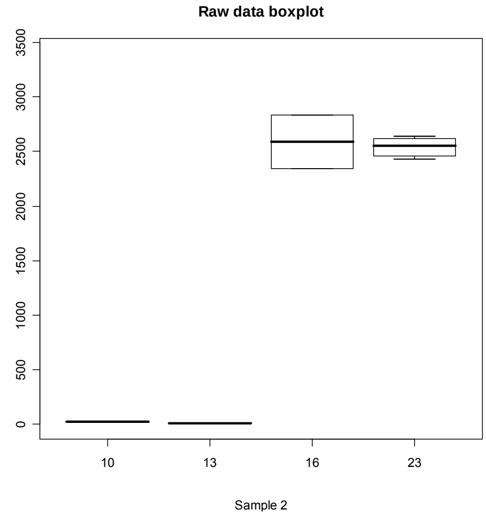
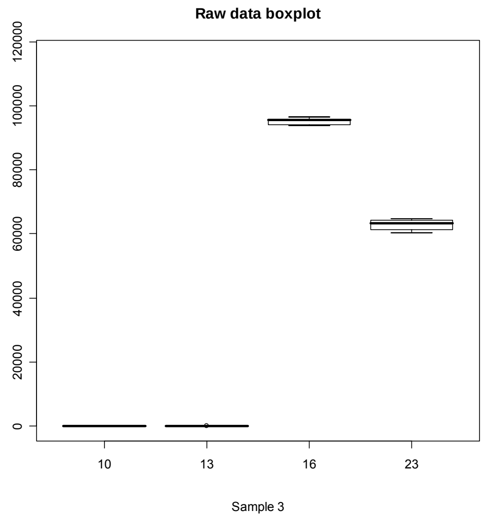
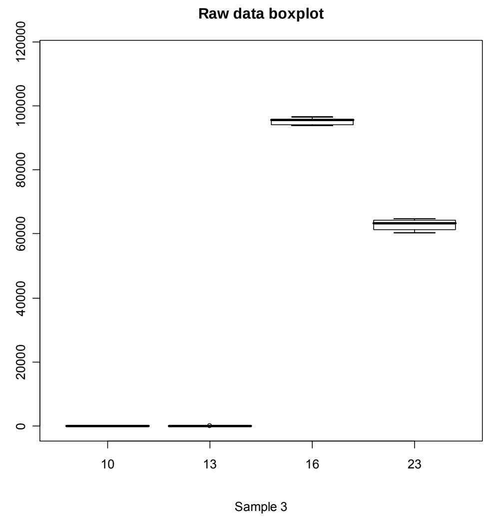
Table 92: TS 15410 B (Ashing (ASTM D6722 + XRF): Phosphorus) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

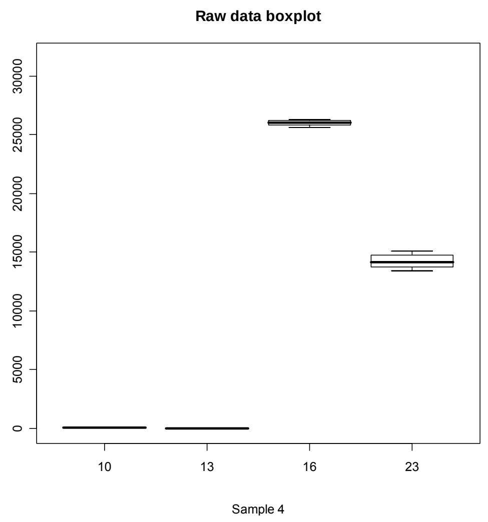
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	397113.3	3	6340.519	3	2186	14	117	63	12
2	57815.82	3	105.1123	4	366.6668	8	80	40	8
3	1.44E+08	3	130162.9	4	126096.5	16	144	72	12
4	4361560	3	996.9596	4	5000.794	16	144	72	12
5	17705345	3	26201.76	4	17180.34	16	144	72	12

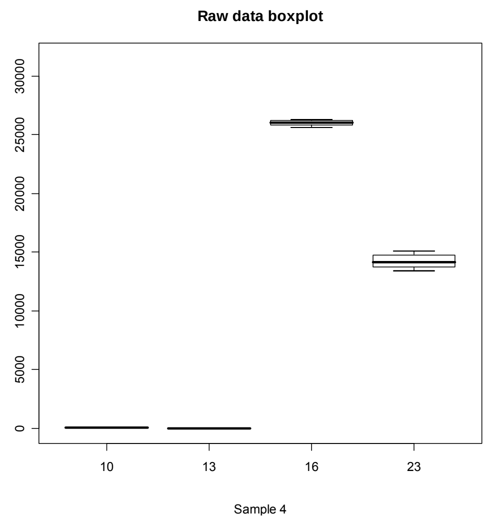
Table 93: TS 15410 B (Ashing (ASTM D6722 + XRF): Phosphorus) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

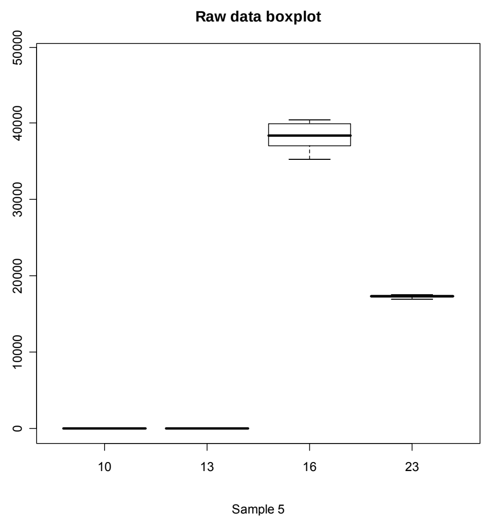
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	159	26	12	33.6	160	448
2	NA	NA	7	19.6	7	19.6
3	2827	91	89	249.2	2829	7921.2
4	NA	NA	18	50.4	18	50.4
5	991	43	33	92.4	992	2777.6

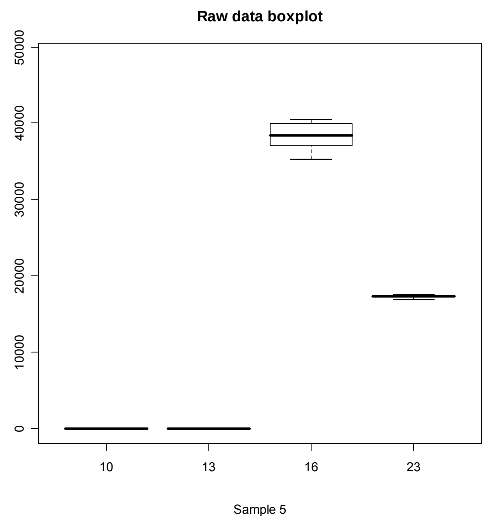
3.5.24 TS 15410 B(Ashing (ASTM D6722 + XRF): Silicon)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	









N.B. Data set to small for meaningful outlier testing

Results are expressed in mg/kg d.m.

Table 94: TS 15410 B (Ashing (ASTM D6722 + XRF): Silicon) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	26016	21
2	1286	16
3	39553	24
4	10062	24
5	13913	24

Table 95: TS 15410 B(Ashing (ASTM D6722 + XRF): Silicon) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

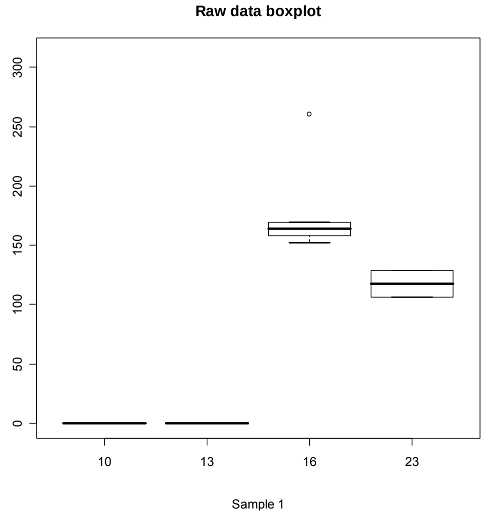
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	1.21E+10	3	1.02E+08	3	5039613	14	117	63	12
2	25786807	3	158245.1	4	2309.35	8	80	40	8
3	4.06E+10	3	12819199	4	7737947	16	144	72	12
4	2.83E+09	3	1803870	4	595262	16	144	72	12
5	5.94E+09	3	19132774	4	2464531	16	144	72	12

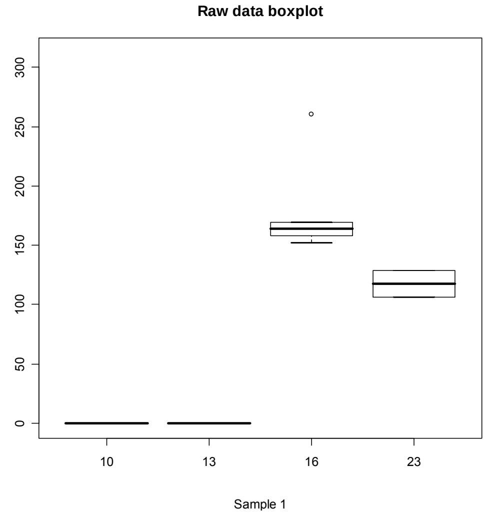
Table 96: TS 15410 B(Ashing (ASTM D6722 + XRF): Silicon) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

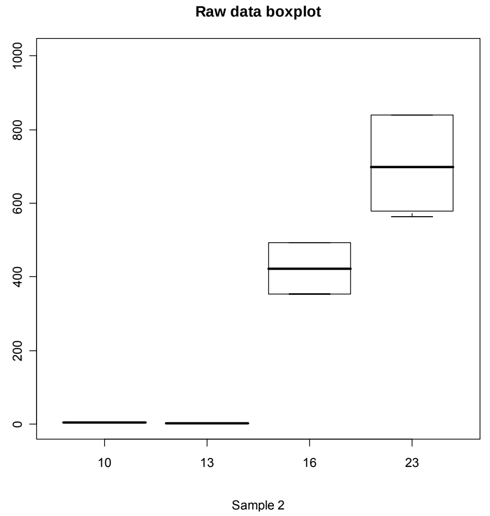
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	27829	3341	600	1680	27835	77938
2	1528	140	17	47.6	1528	4278.4
3	47494	952	695	1946	47499	132997.2
4	12540	371	193	540.4	12542	35117.6
5	18148	1242	392	1097.6	18152	50825.6

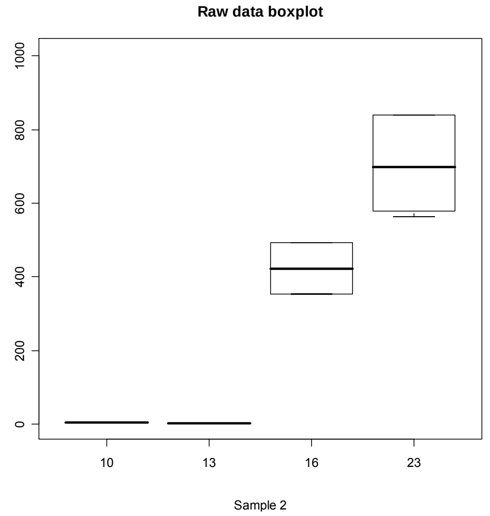
3.5.25 TS 15410 B (Ashing (ASTM D6722 + XRF): Titanium)

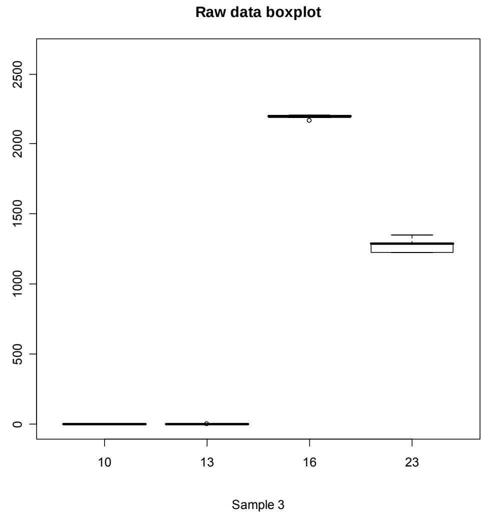
Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*

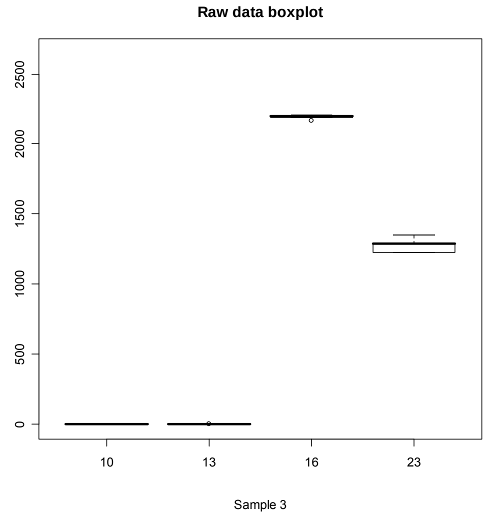


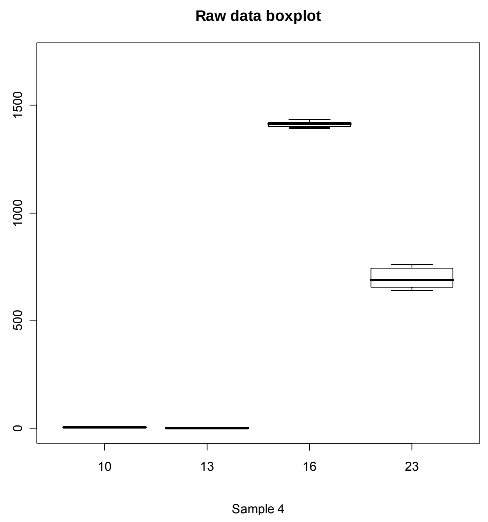


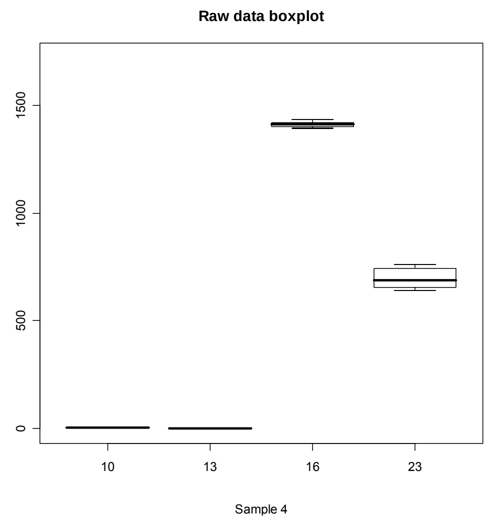


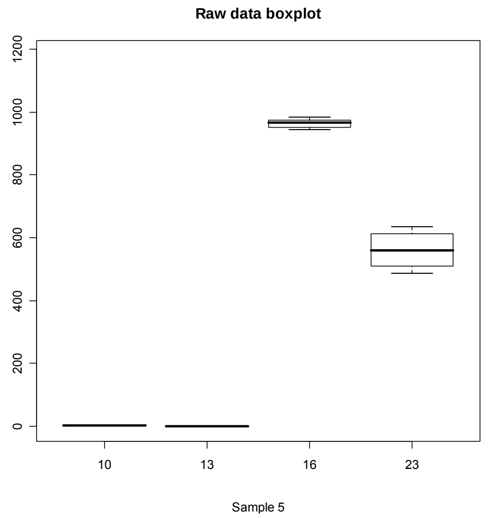


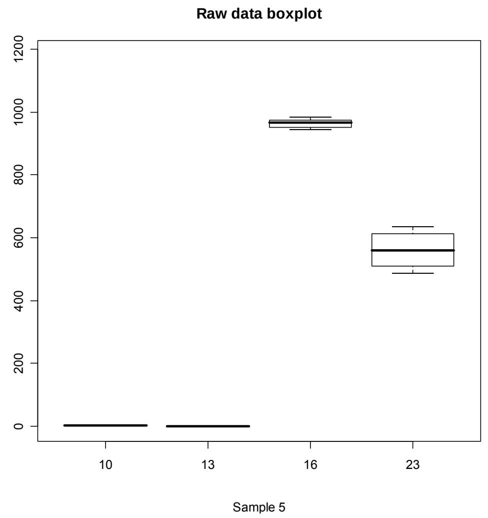












N.B. Data set to small for meaningful outlier testing

Results are expressed in mg/kg d.m.

Table 97: TS 15410 B (Ashing (ASTM D6722 + XRF): Titanium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	84	21
2	318	16
3	868	24
4	527	24
5	382	24

Table 98: TS 15410 B (Ashing (ASTM D6722 + XRF): Titanium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

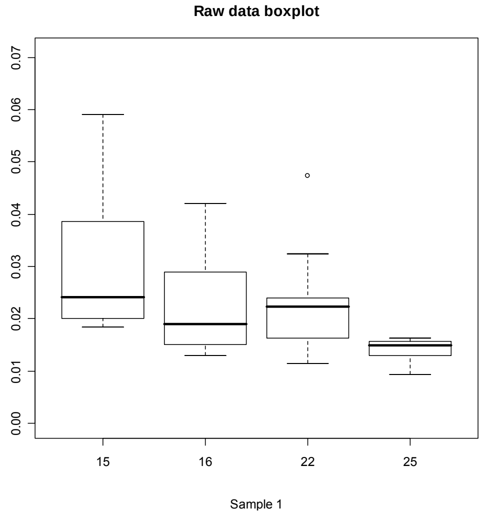
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	123021.8	3	3631	3	5514	14	117	63	12
2	1702340	3	108227	4	593	8	80	40	8
3	20556086	3	856	4	10782	16	144	72	12
4	8175078	3	10768	4	2098	16	144	72	12
5	3973179	3	17064	4	2196	16	144	72	12

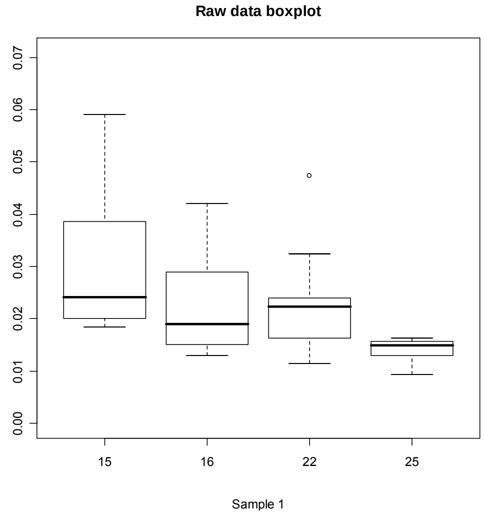
Table 99: TS 15410 B (Ashing (ASTM D6722 + XRF): Titanium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

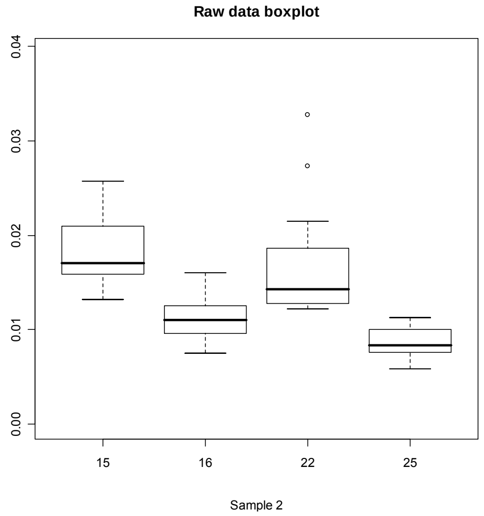
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	88	16	20	56	90	252
2	385	116	9	25.2	385	1078
3	0	NA	26	72.8	26	72.8
4	674	29	11	30.8	674	1887.2
5	469	37	12	33.6	469	1313.2

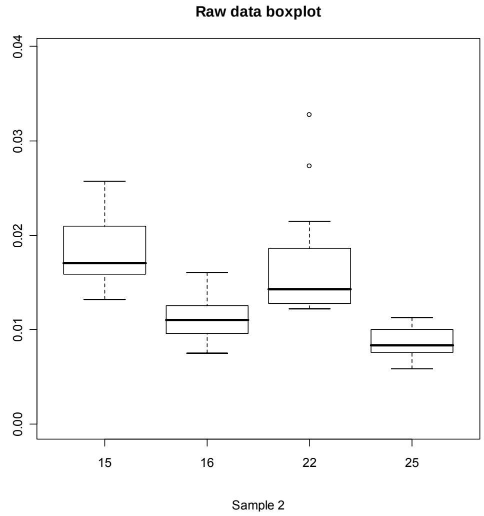
3.5.26 TS 15411 (Mercury by automatic analyzer)

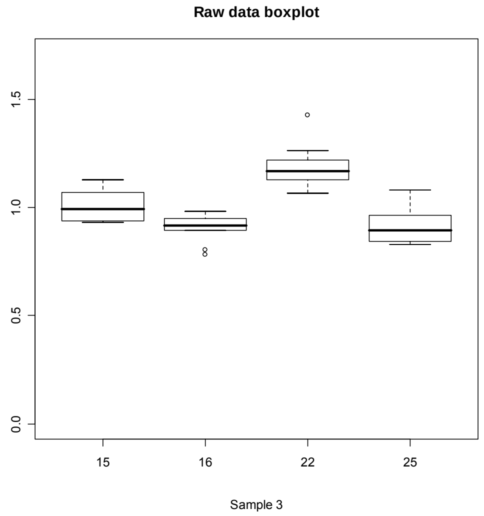
Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*

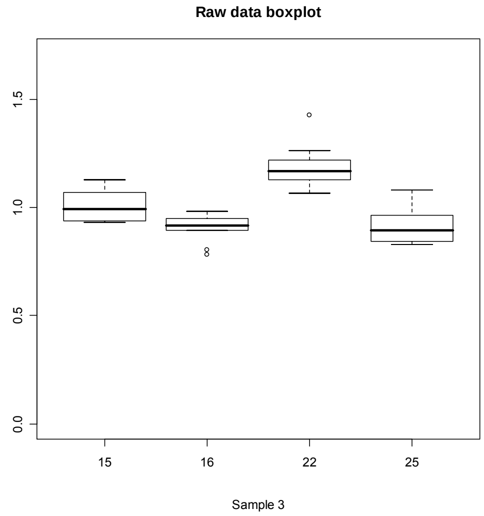


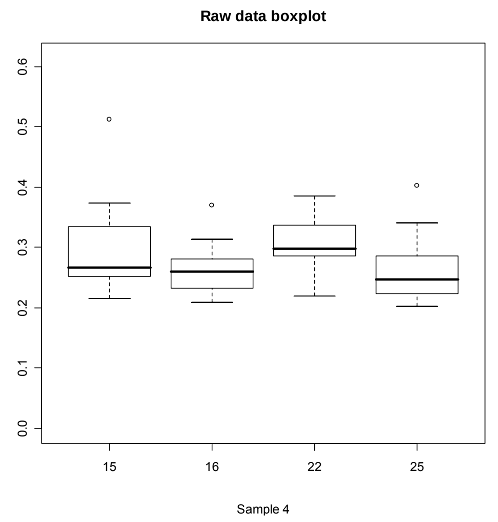


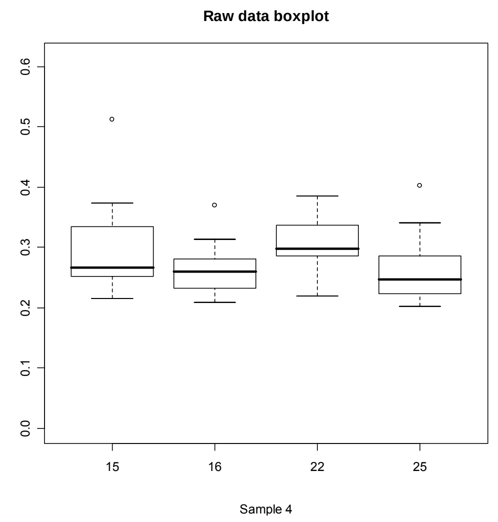


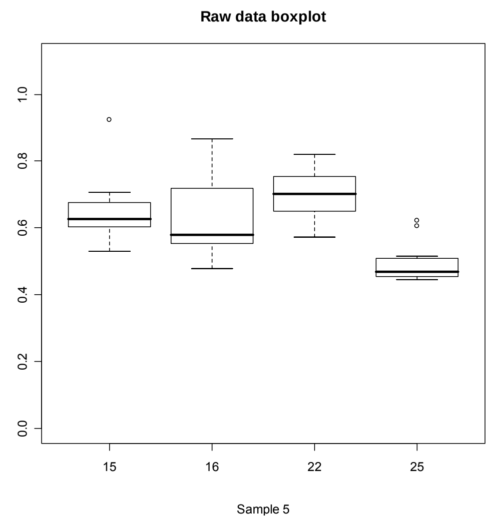


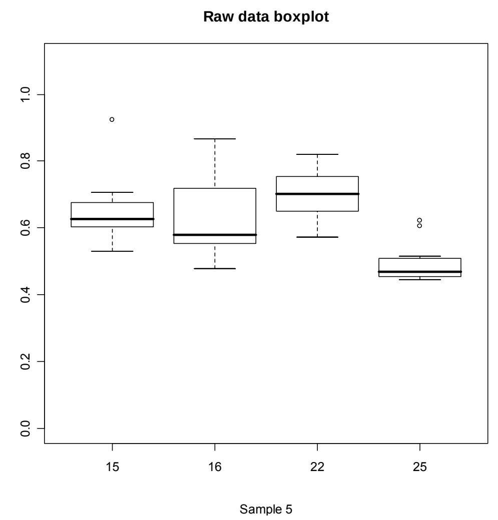












N.B. Data set to small for meaningful outlier testing

Results are expressed in mg/kg d.m.

Table 100: TS 15411 (Mercury by automatic analyzer) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	0.02	48
2	0.01	48
3	1.00	48
4	0.28	48
5	0.62	48

Table 101: TS 15411 (Mercury by automatic analyzer) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	0.002	3	0.0004	4	0.004	40	576	288	24
2	0.001	3	0.0002	4	0.001	40	576	288	24
3	0.595	3	0.0381	4	0.246	40	576	288	24
4	0.018	3	0.0337	4	0.126	40	576	288	24
5	0.277	3	0.0691	4	0.303	40	576	288	24

Table 102: TS 15411 (Mercury by automatic analyzer) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	r	s_{Rj}	R
1	NA	NA	0.01	0.028	0.01	0.028
2	0.004	0.003	0.004	0.0112	0.006	0.0168
3	0.125	0.024	0.078	0.2184	0.148	0.4144
4	0	0.03	0.056	0.1568	0.056	0.1568
5	0.079	0.04	0.087	0.2436	0.118	0.3304

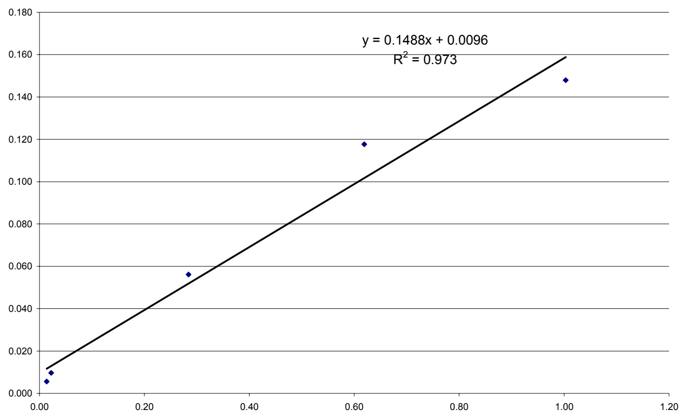
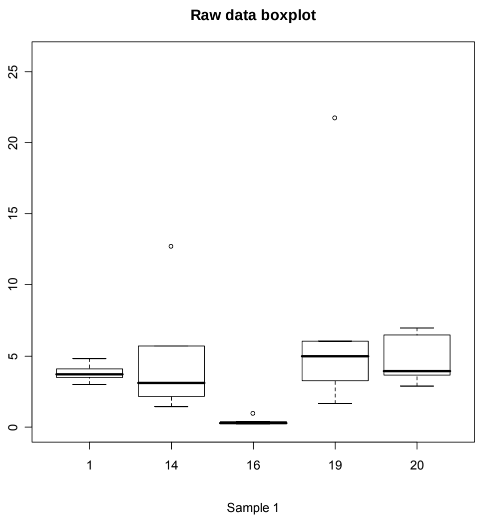
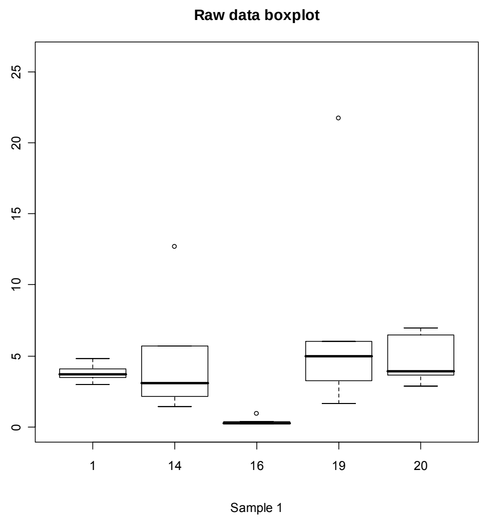
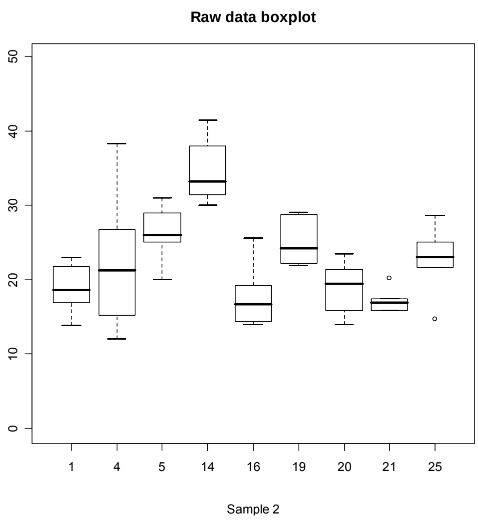
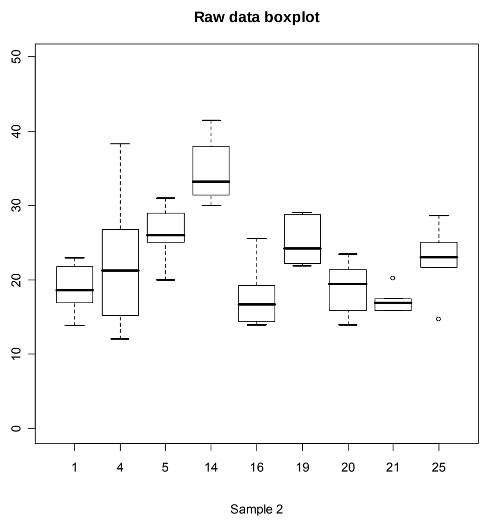
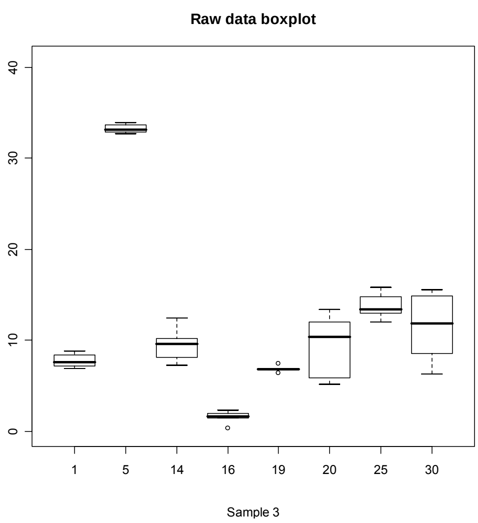
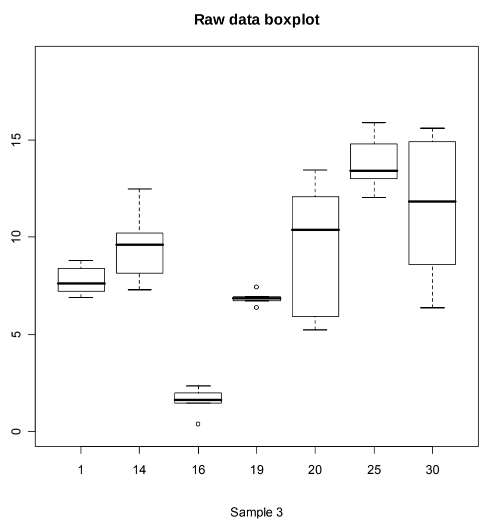


Figure 24: TS 15411 (Mercury by automatic analyzer) - Concentration dependence (in mg/kg d.m.) of reproducibility.

3.5.27 TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Arsenic)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	

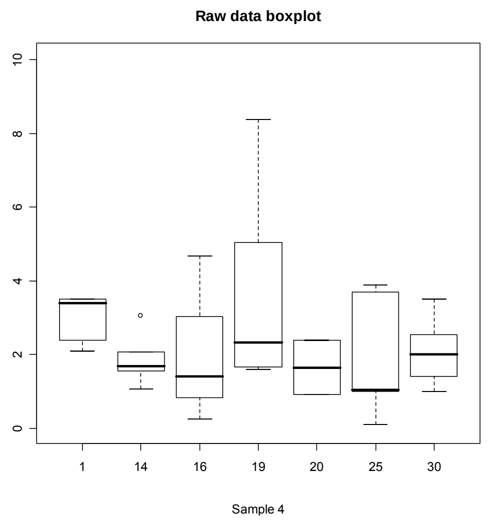
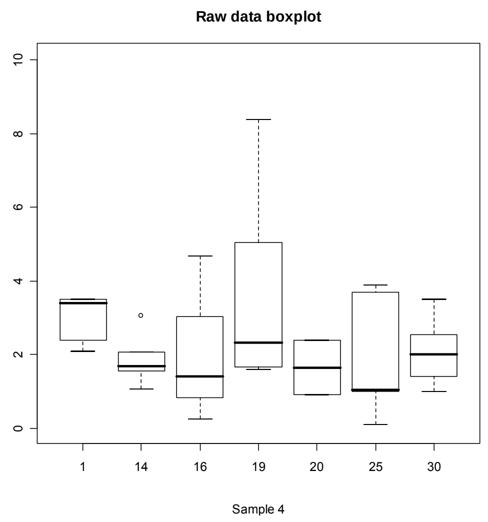
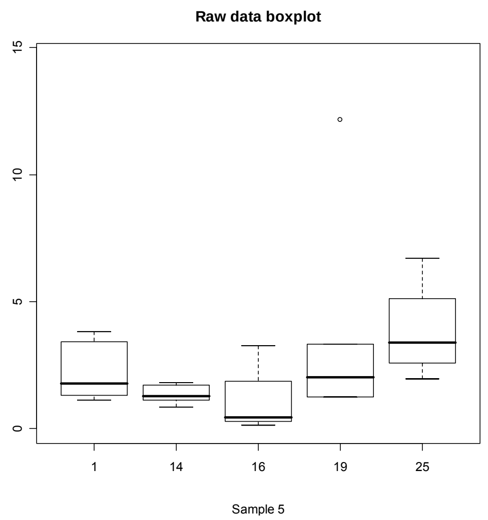
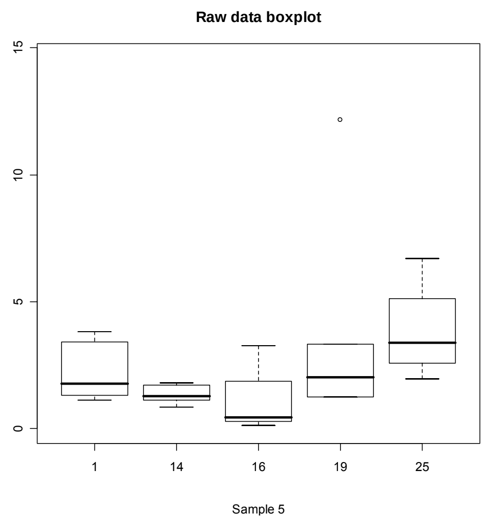
	
	
	Results are expressed in mg/kg d.m.

Table 103: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Arsenic) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	4.3	28
2	22.6	54
3	8.6	42
4	2.4	32
5	2.5	25

Table 104: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Arsenic) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	125.6	4	85.4	13	286.7	10	158	80	14.2
2	1449.6	8	556.6	9	482.8	36	324	162	27
3	541.1	6	49.2	11	103.7	36	324	162	27
4	14.3	6	7.0	11	60.4	26	232	118	22.467
5	28.7	4	29.5	13	85.3	29	255	129	23.867

Table 105: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Arsenic) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	0	NA	5.4	15.12	5.4	15.12
2	4.5	4	3.7	10.36	5.8	16.24
3	3.3	0.8	1.7	4.76	3.7	10.36
4	0	NA	1.5	4.2	1.5	4.2
5	0	NA	1.7	4.76	1.7	4.76

3.5.28 TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Barium)

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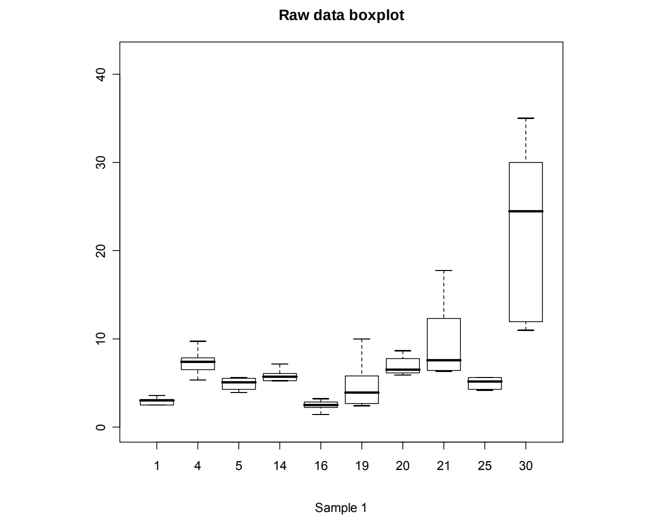
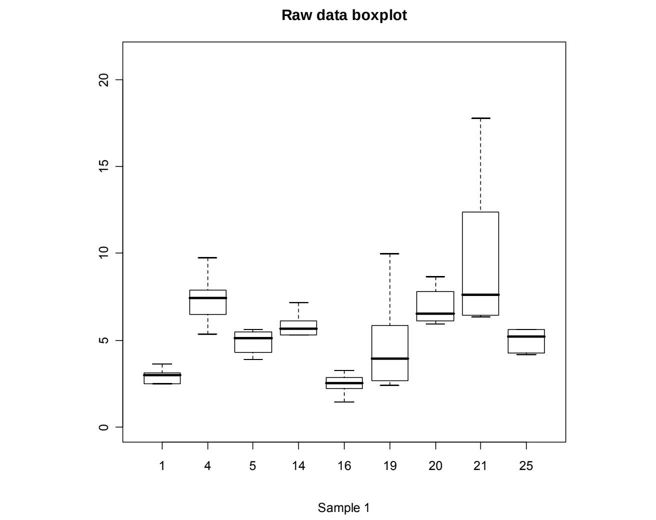
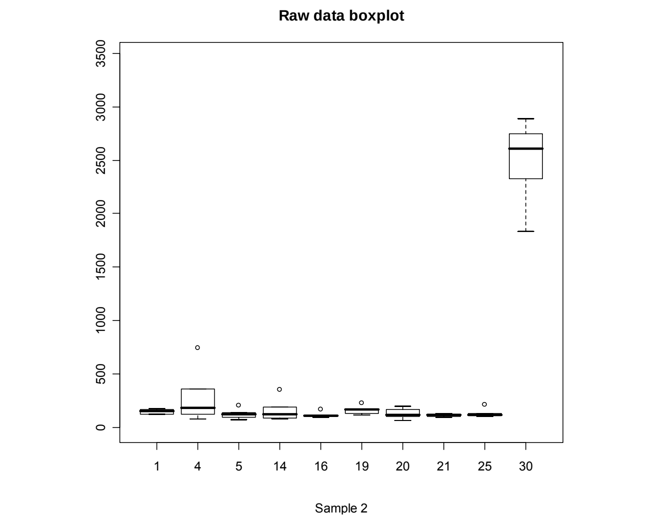
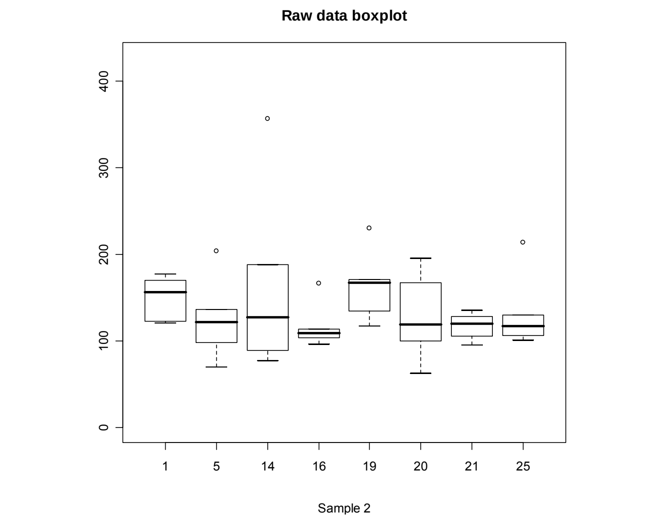
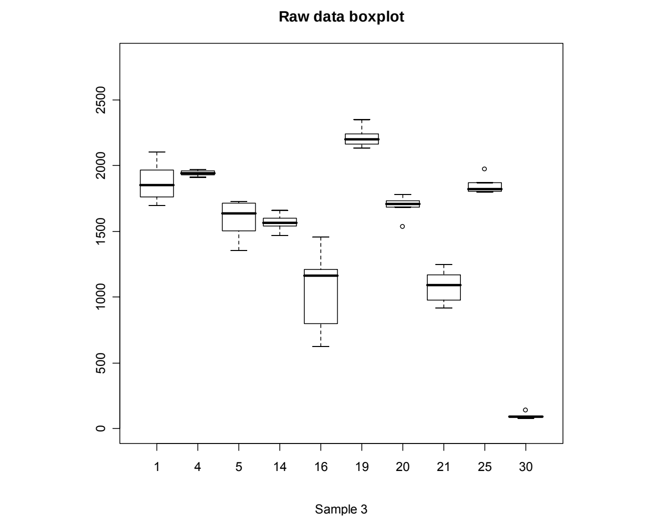
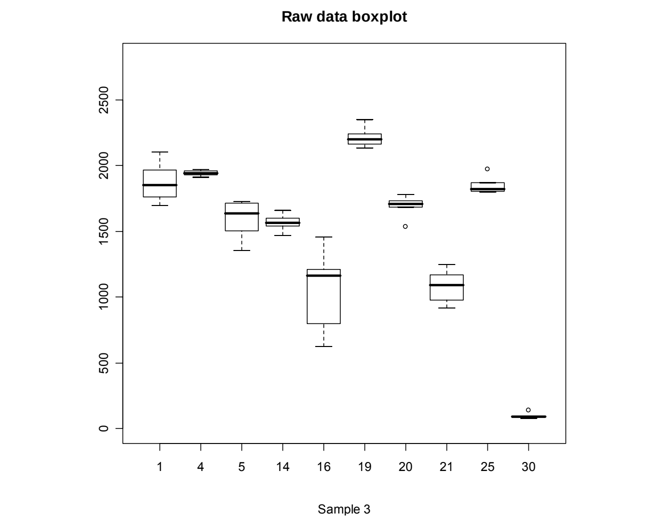
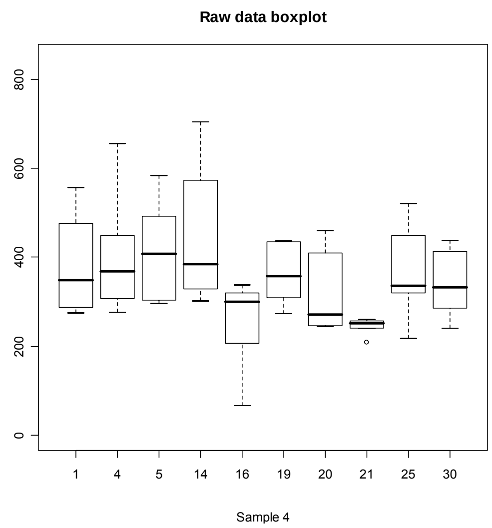
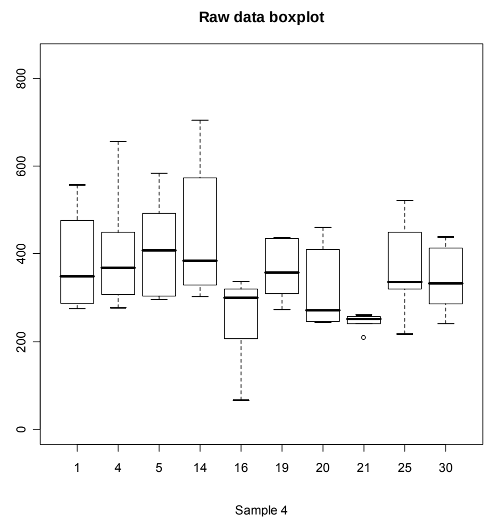
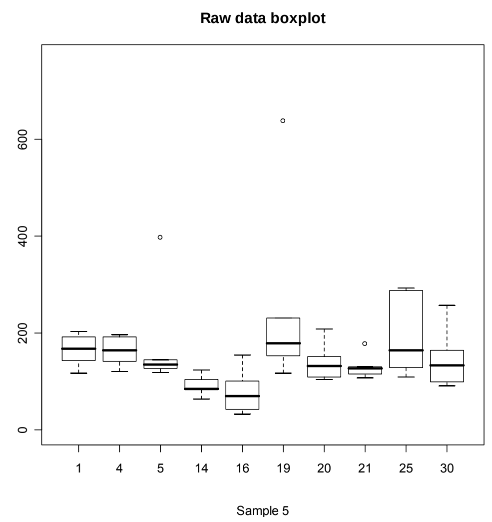
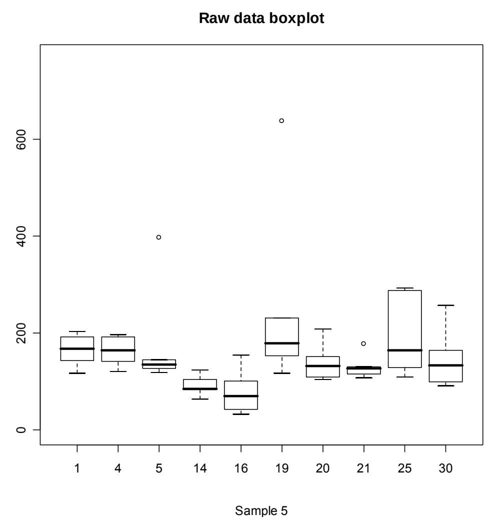
	
	
	

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*









Results are expressed in mg/kg d.m.

Table 106: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Barium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	6	54
2	152	54
3	1498	60
4	352	58
5	153	60

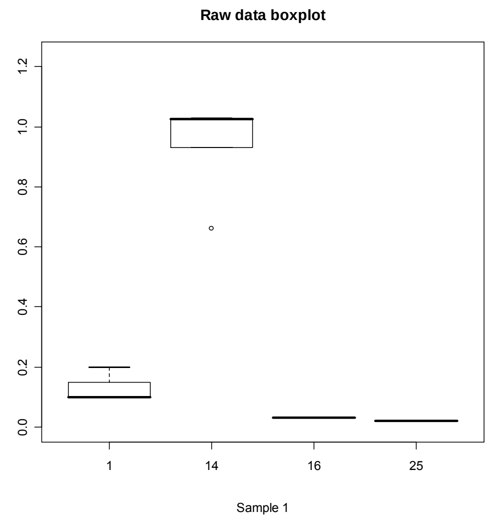
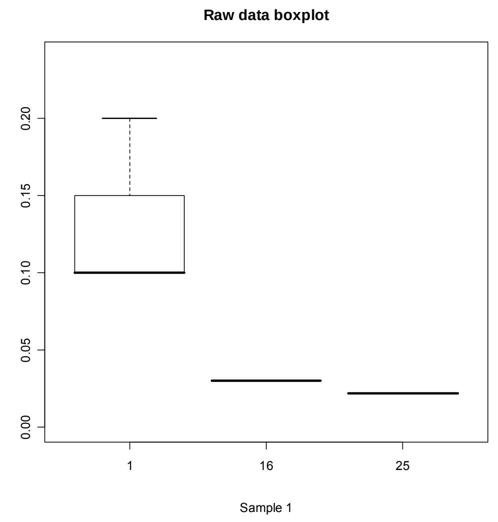
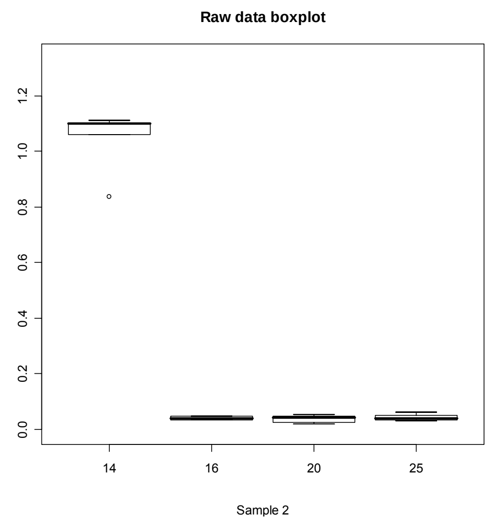
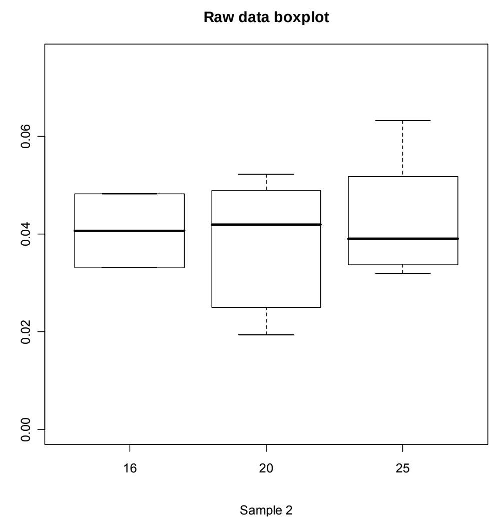
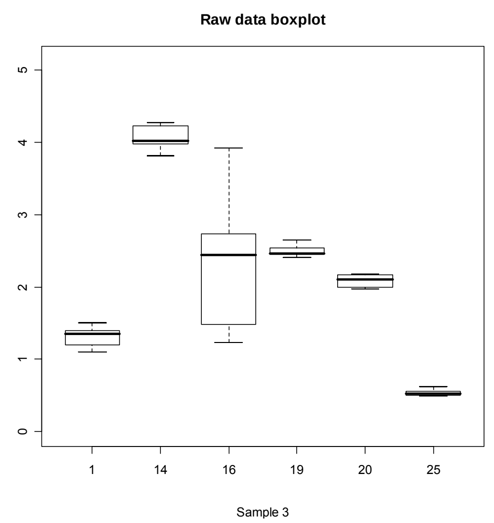
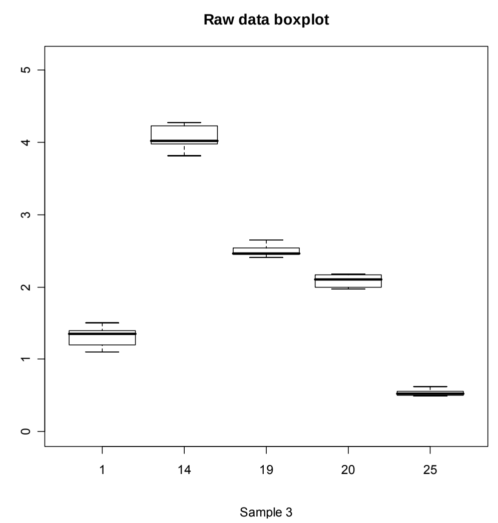
Table 107: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Barium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

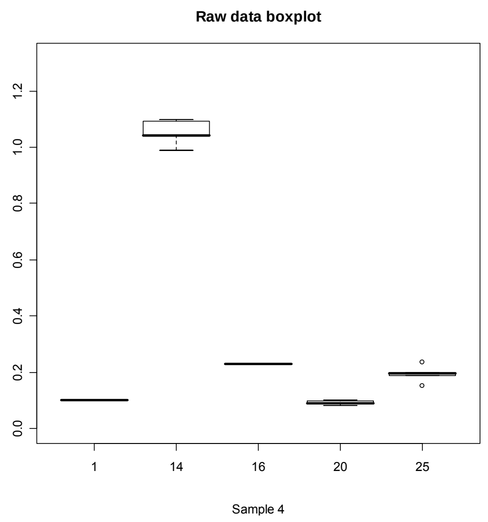
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	240	8	30	11	141	34	324	162	27
2	122029	8	133674	11	273526	34	324	162	27
3	20105082	9	343574	10	529886	40	360	180	30
4	230812	9	136316	10	419378	38	340	172	29.5
5	131178	9	60384	10	270413	40	360	180	30

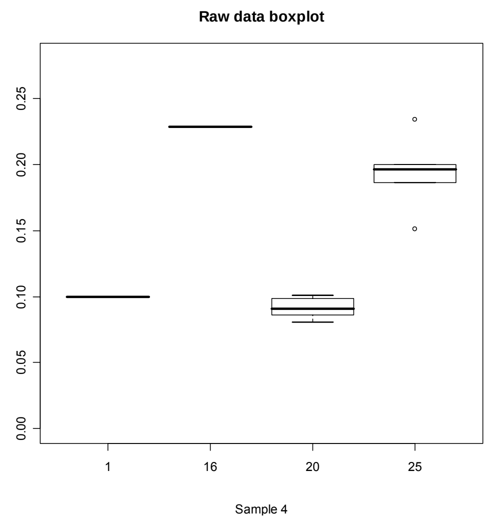
Table 108: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Barium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

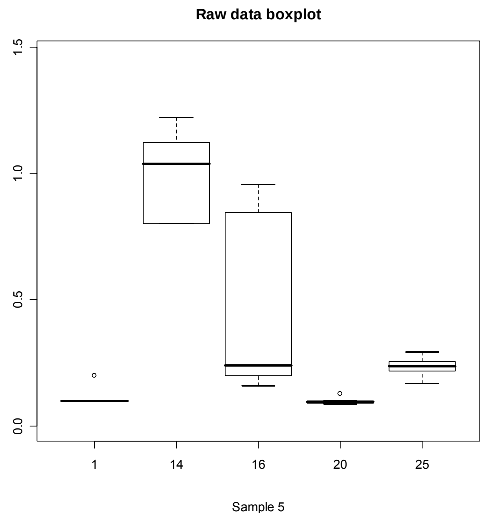
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	NA	NA	2	5.6	2	5.6
2	19	41	90	252	92	257.6
3	605	84	115	322	616	1724.8
4	45	30	105	294	114	319.2
5	NA	NA	82	229.6	82	229.6

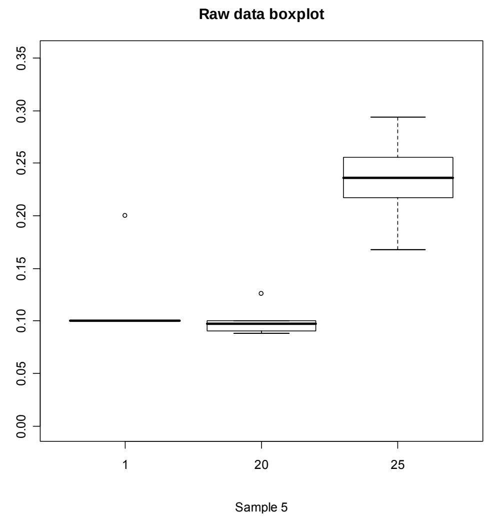
3.5.29 TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Beryllium)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	









Results are expressed in mg/kg d.m.

Table 109: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Beryllium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	0.56	11
2	0.34	20
3	2.14	36
4	0.36	24
5	0.38	29

Table 110: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Beryllium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	2.0	3	0.0365	5	0.08	2	47	29	8
2	4.3	3	0.0153	7	0.04	21	184	94	17
3	42.7	5	0.2175	6	4.79	24	216	108	18
4	3.8	4	0.0007	6	0.01	19	170	86	15.6
5	3.4	4	0.5897	7	0.17	23	205	103	17.6

Table 111: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Beryllium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	NA	NA	0.197	0.5516	0.197	0.5516
2	0.404	0.007	0.045	0.126	0.406	1.1368
3	NA	NA	0.447	1.2516	0.447	1.2516
4	NA	NA	0.024	0.0672	0.024	0.0672
5	0.316	0.176	0.087	0.2436	0.327	0.9156

3.5.30 TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Cadmium)

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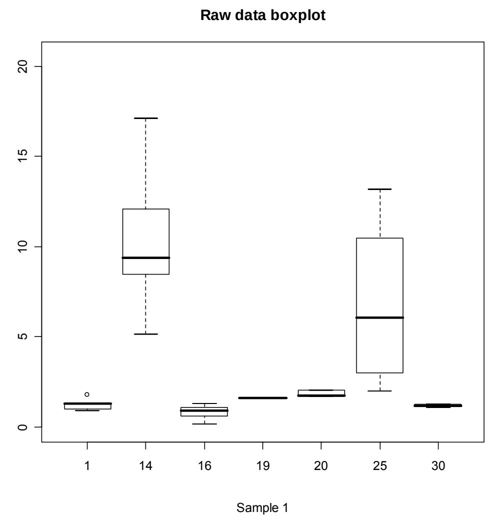
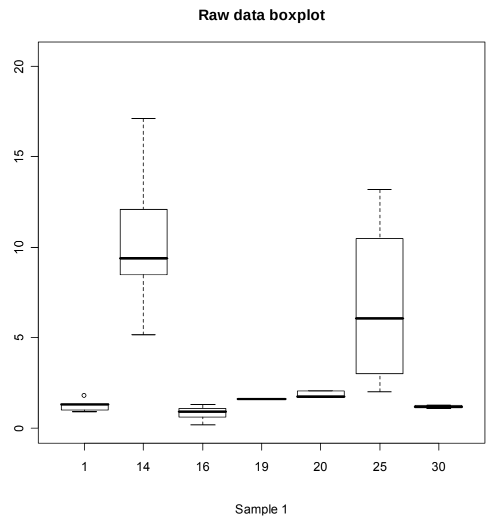
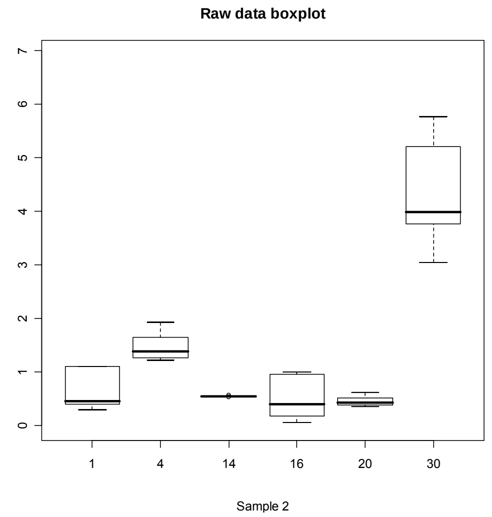
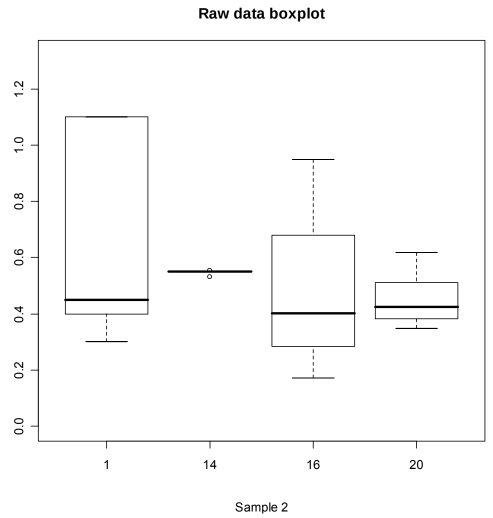
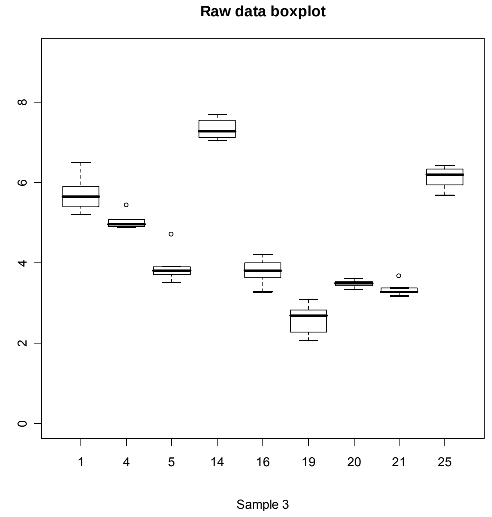
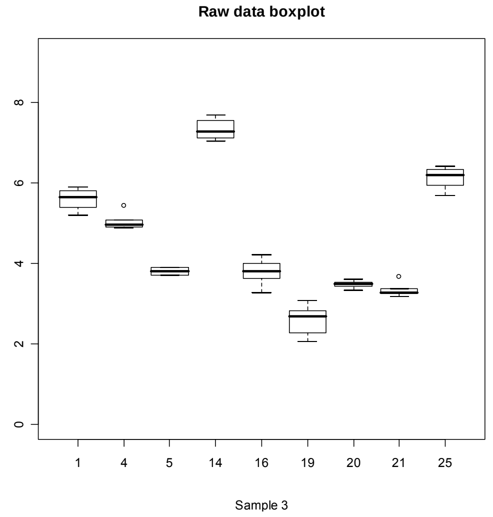
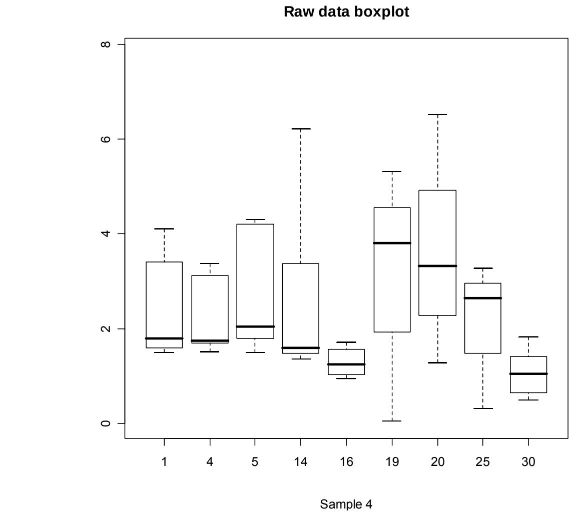
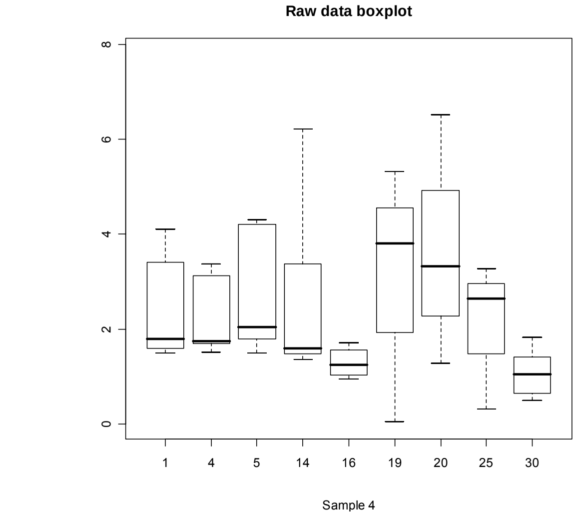
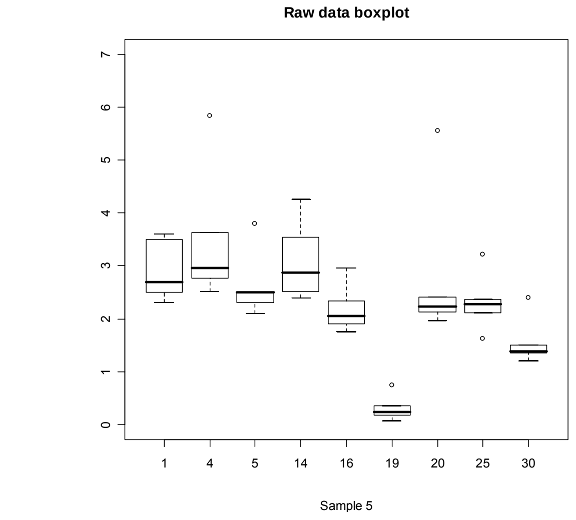
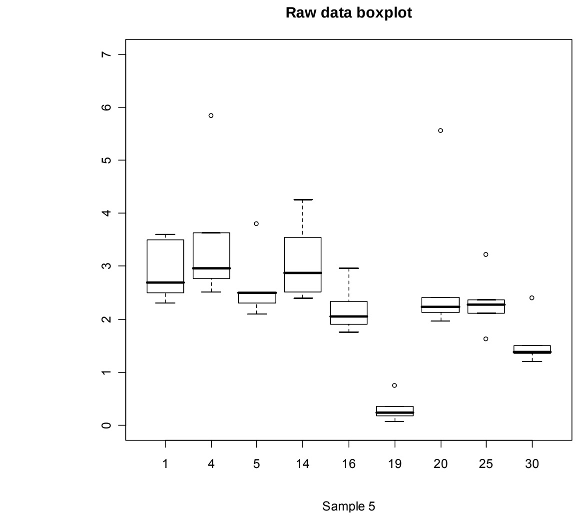
	
	
	









Results are expressed in mg/kg d.m.

Table 112: TS 15411 A - (aqua regia, HF, boric acid and micro-wave assisted digestion: Cadmium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	3.6	37
2	0.7	30
3	4.6	54
4	2.3	47
5	2.3	54

Table 113: TS 15411 A - (aqua regia, HF, boric acid and micro-wave assisted digestion: Cadmium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

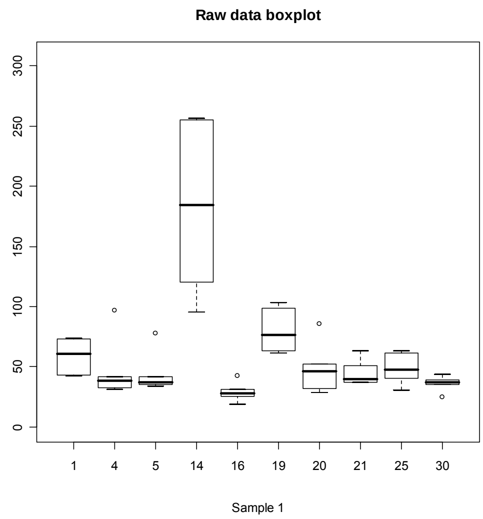
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	463.57	6	34.71	10	155.91	20	217	109	19
2	4.30	4	0.60	13	1.27	24	252	126	21
3	114.92	8	0.69	9	3.59	36	324	162	27
4	28.68	8	22.50	8	52.27	30	259	135	25.267
5	42.75	8	5.48	9	20.95	36	324	162	27

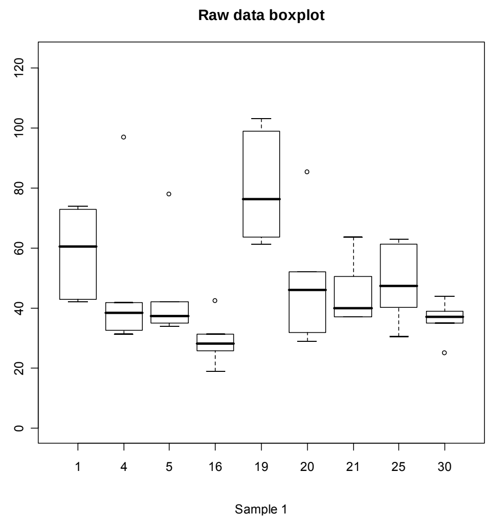
Table 114: TS 15411 A - (aqua regia, HF, boric acid and micro-wave assisted digestion: Cadmium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

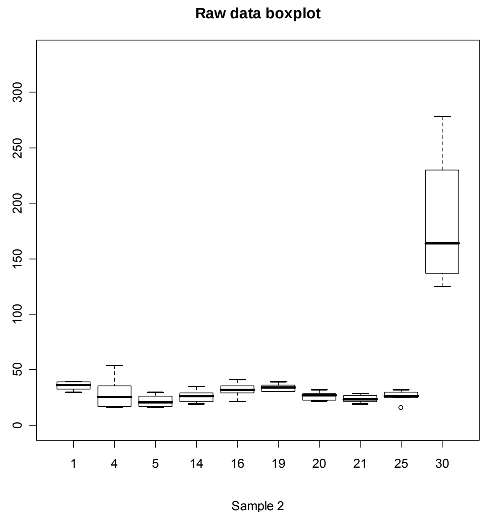
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	NA	NA	2.79	7.812	2.79	7.812
2	NA	NA	0.23	0.644	0.23	0.644
3	NA	NA	0.32	0.896	0.32	0.896
4	0.38	0.63	1.32	3.696	1.37	3.836
5	0.89	0.1	0.76	2.128	1.17	3.276

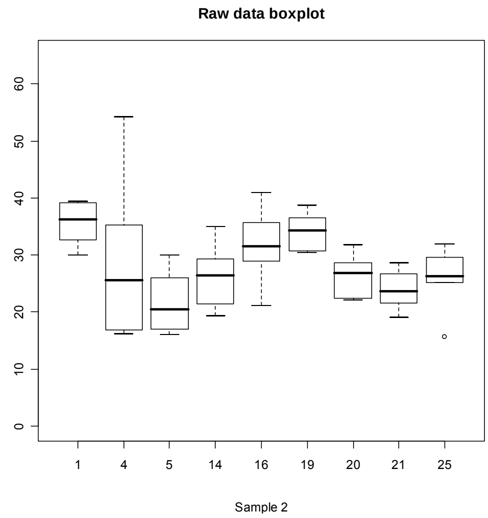
3.5.31 TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Chromium)

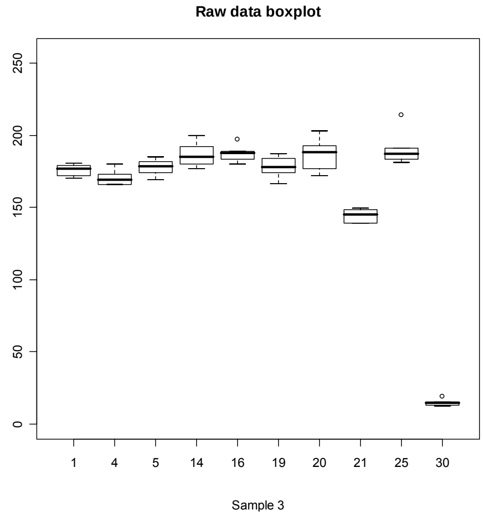
Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*

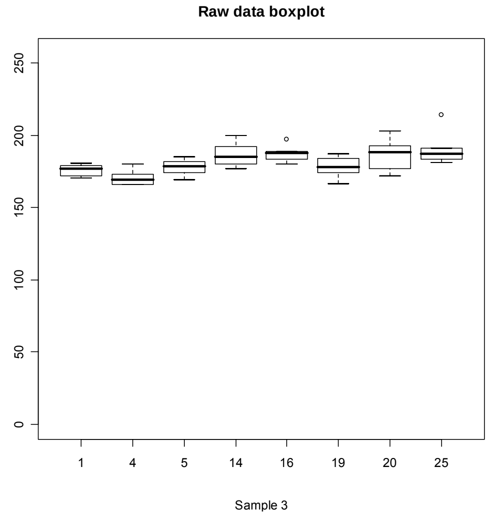


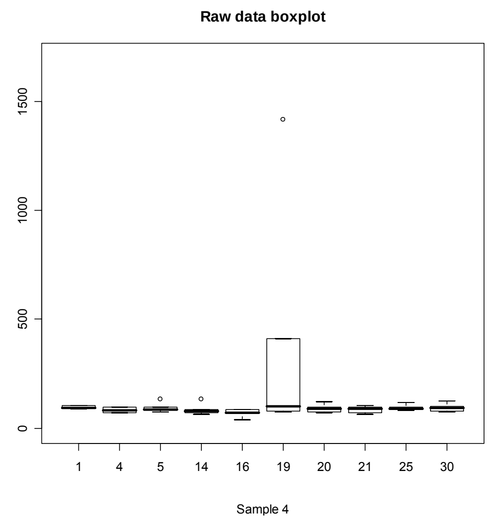


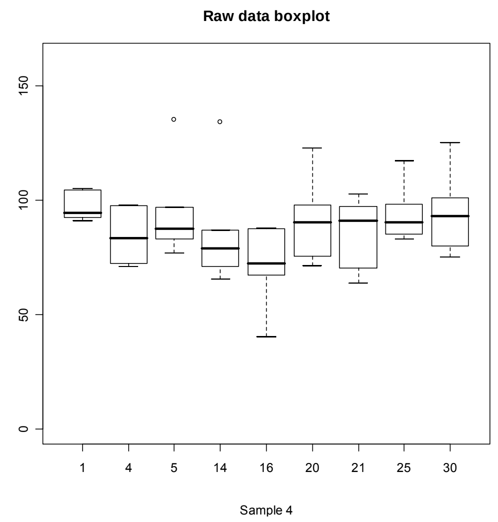


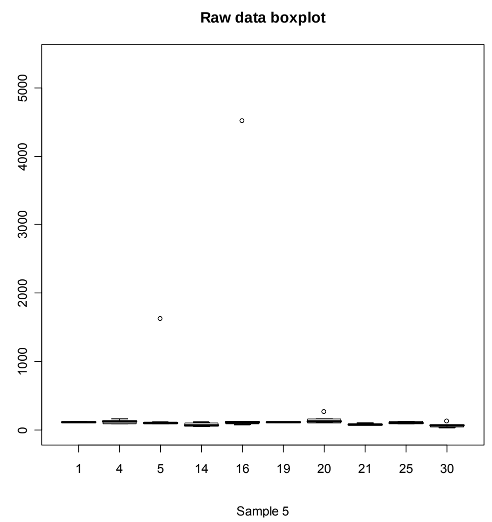


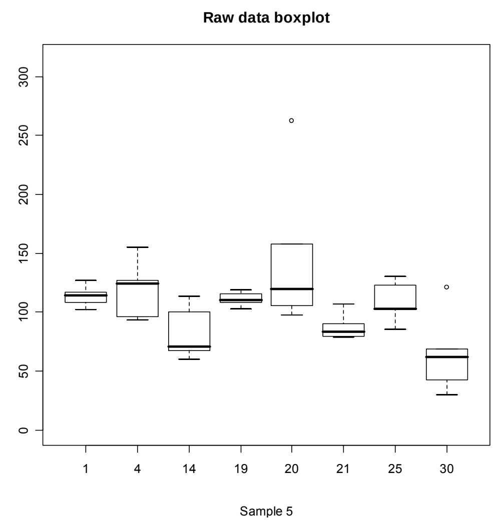












Results are expressed in mg/kg d.m.

Table 115: TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Chromium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	48	54
2	28	54
3	178	54
4	89	53
5	132	54

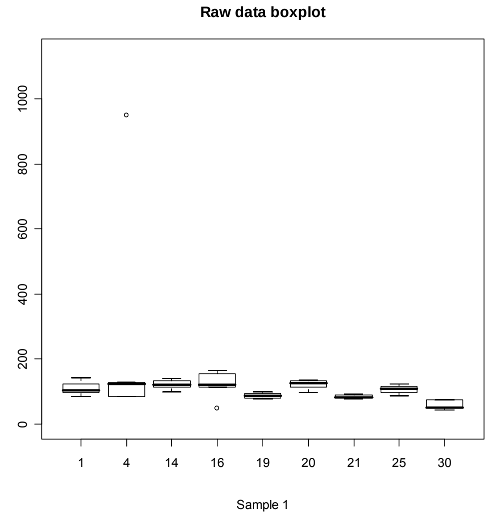
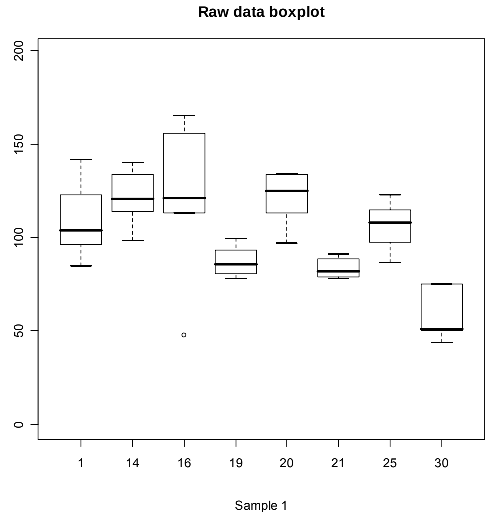
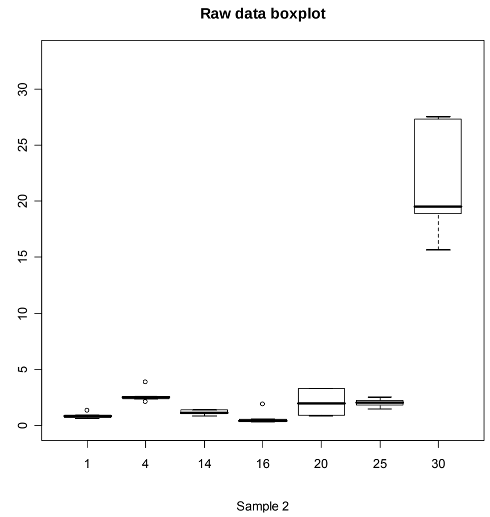
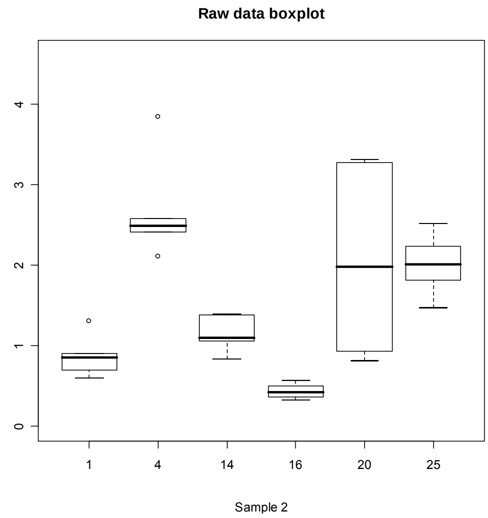
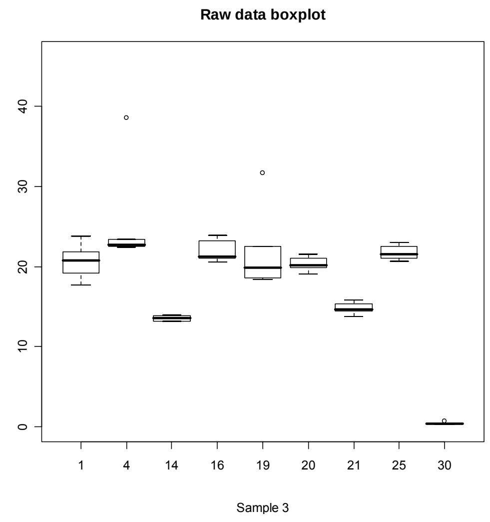
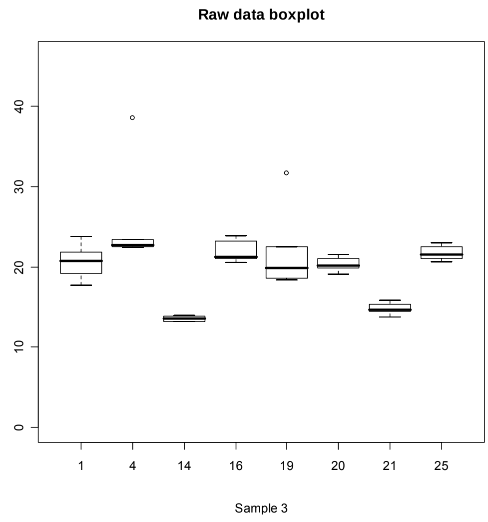
Table 116: TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Chromium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS_{.Hj}$, ni_{Hj}) and for repeatability ($SS_{.rj}$, ni_{rj})

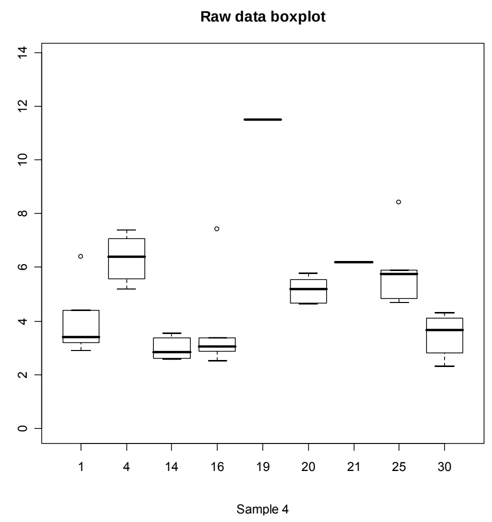
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	9957	8	2774	9	8279	36	324	162	27
2	1061	8	1265	9	850	36	324	162	27
3	9469	8	419	9	2218	36	324	162	27
4	3027	8	2687	9	10438	35	313	157	26.6
5	362290	8	409378	9	1564967	36	324	162	27

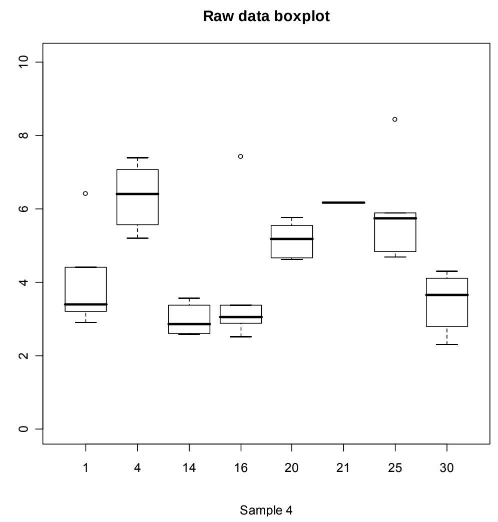
Table 117: TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Chromium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

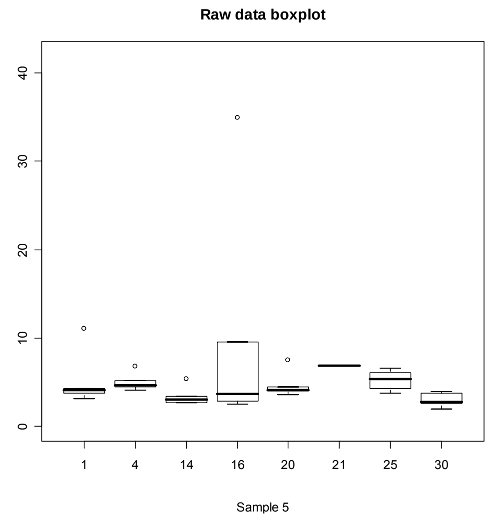
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	12	5	15	42	20	56
2	NA	6	5	14	5	14
3	NA	NA	8	22.4	8	22.4
4	4	0	17	47.6	18	50.4
5	NA	26	208	582.4	208	582.4

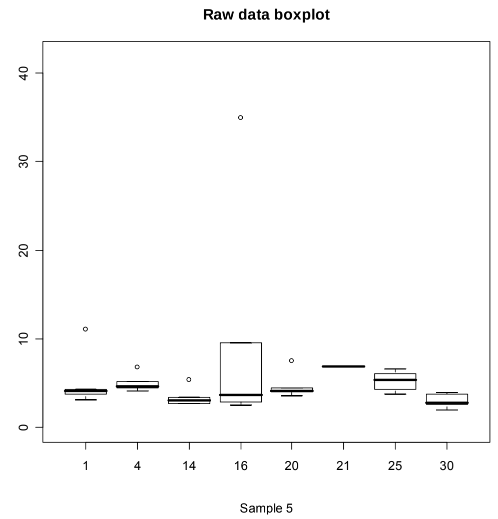
3.5.32 TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Cobalt)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	









Results are expressed in mg/kg d.m.

Table 118: TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Cobalt) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	100.9	48
2	1.5	35
3	20.0	48
4	4.6	42
5	5.1	43

Table 119: TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Cobalt) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	22121	7	2971	8	12256	32	288	144	24
2	18	5	7	10	4	31	277	139	23.6
3	645	7	69	8	310	32	288	144	24
4	62	7	13	7	29	27	242	122	21.6
5	168	7	238	7	640	28	253	127	22

Table 120: TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Cobalt) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	NA	NA	19.57	54.796	19.57	54.796
2	0.53	0.51	0.36	1.008	0.64	1.792
3	NA	NA	3.11	8.708	3.11	8.708
4	1.16	0.54	1.04	2.912	1.56	4.368
5	0	1.93	4.78	13.384	4.78	13.384

3.5.33 TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Copper)

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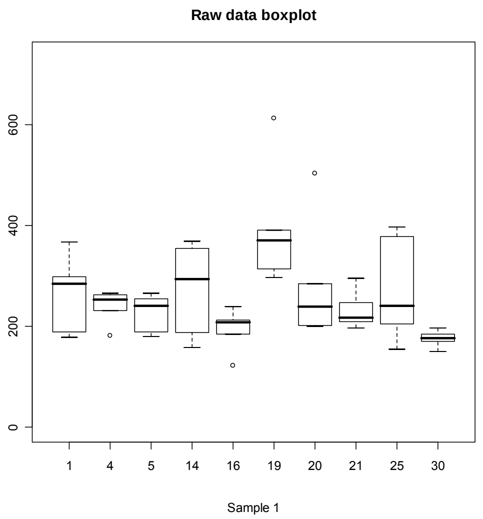	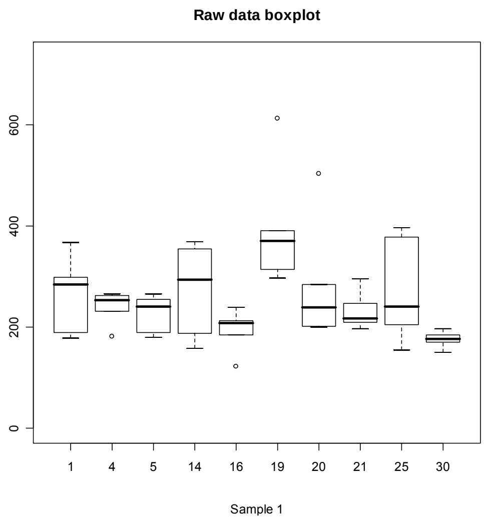
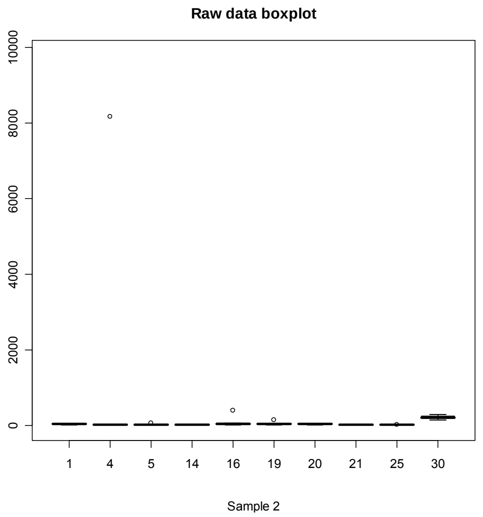	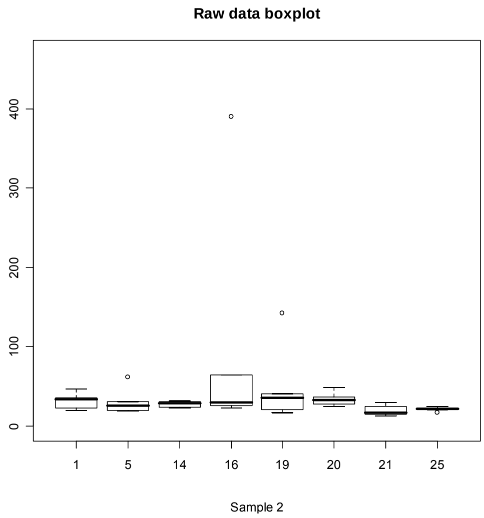
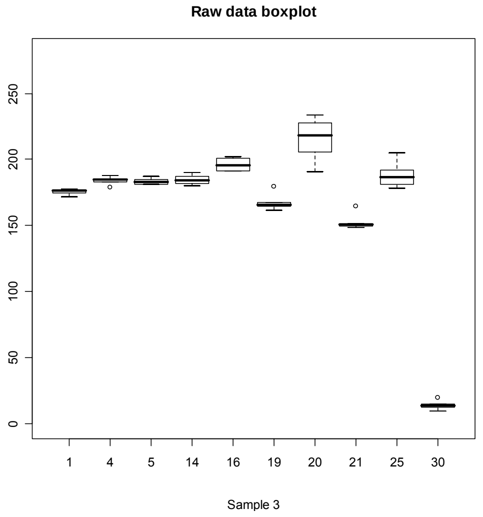	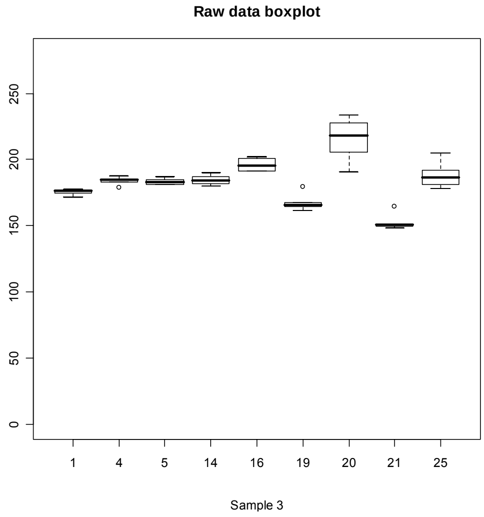

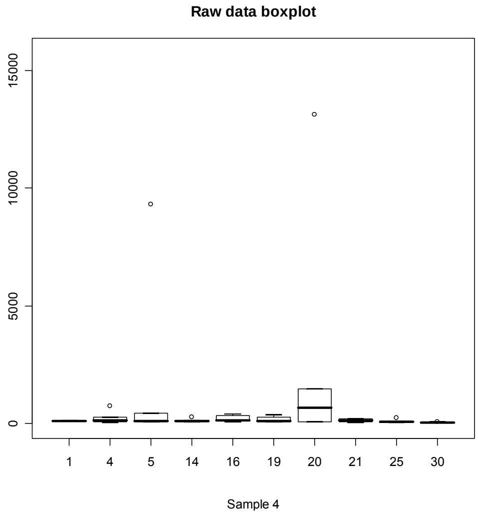

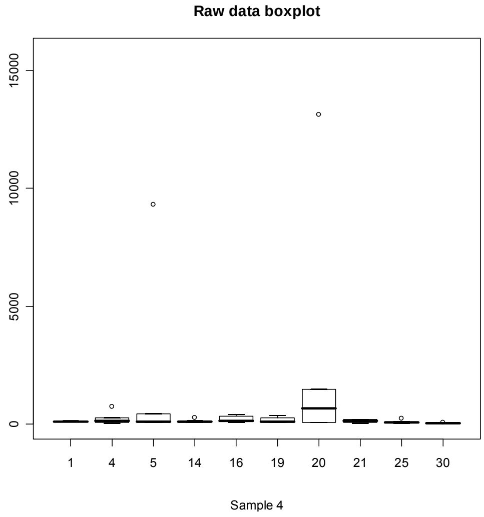

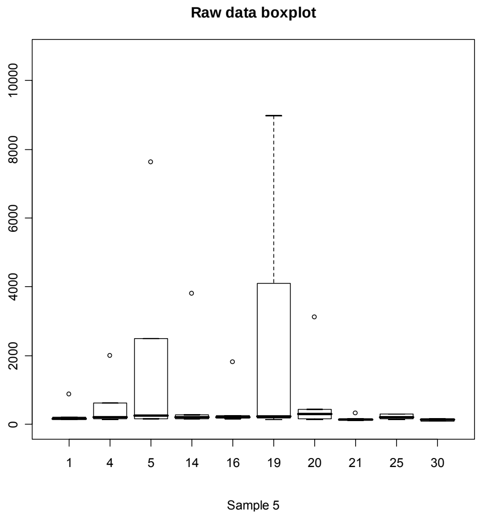

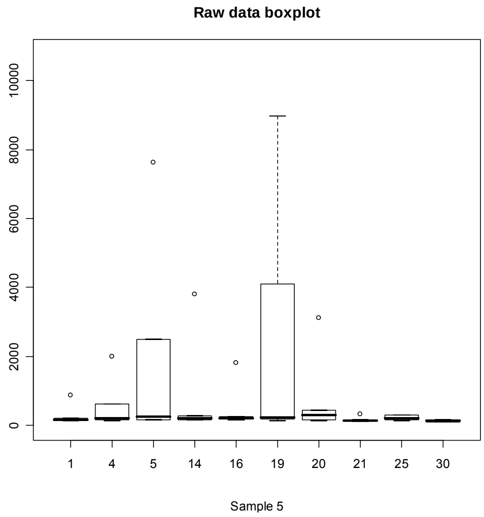

Results are expressed in mg/kg d.m.

Table 121: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Copper) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	255	60
2	58	54
3	183	54
4	564	57
5	748	60

Table 122: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Copper) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	188833.4	9	36231.71	10	237385.8	40	360	180	30
2	192742	8	22712.67	11	107510.8	40	360	180	30
3	14946.73	8	500.4431	11	1847.015	40	360	180	30
4	42764074	9	45146641	10	1.58E+08	37	333	167	28.67
5	29624528	9	12513657	10	1.21E+08	40	360	180	30

Table 123: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Copper) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	NA	NA	77	215.6	77	215.6
2	NA	NA	51.8	145.04	51.8	145.04
3	NA	NA	6.8	19.04	6.8	19.04
4	203.2	292	2067.2	5788.16	2077.1	5815.88
5	NA	NA	1736.2	4861.36	1736.2	4861.36

3.5.34 TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Mercury)

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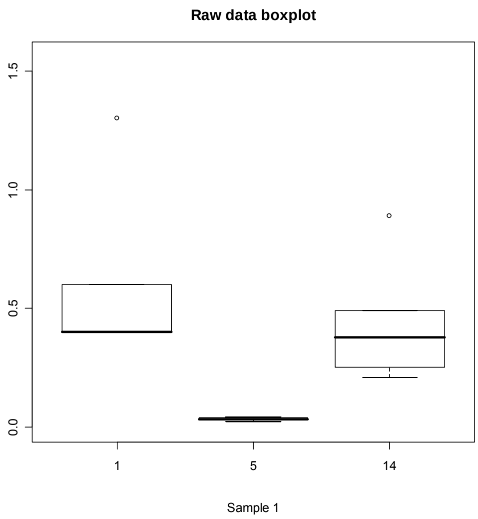
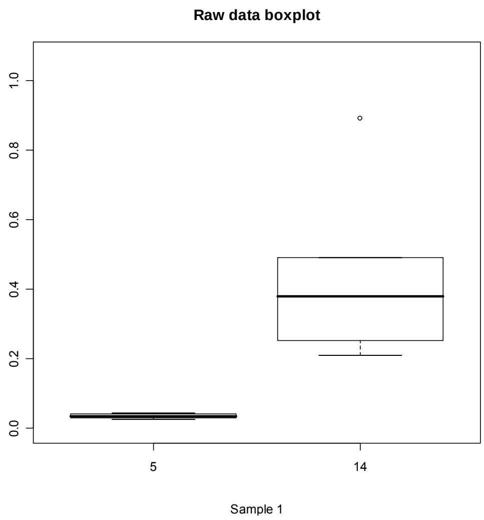
<tr><td>
</td><td>
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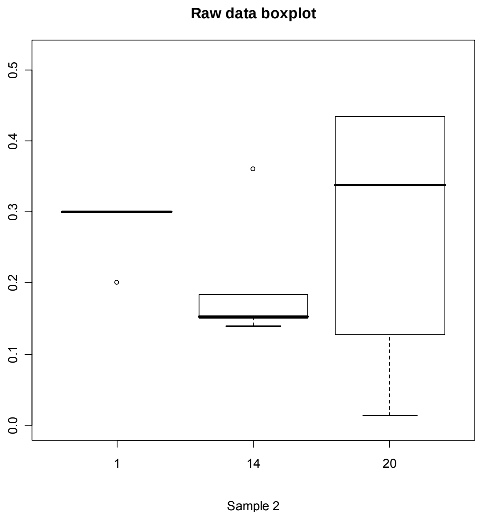
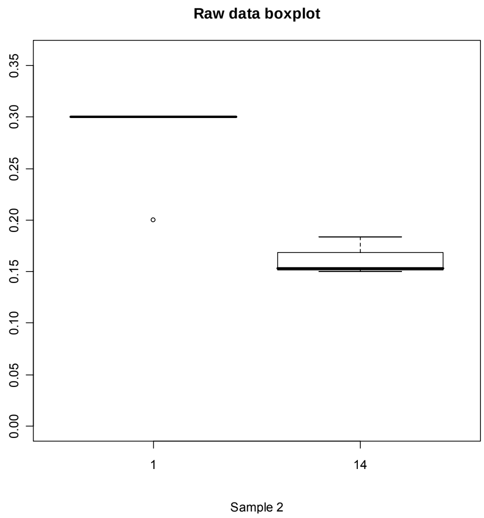
<tr><td>
</td><td>
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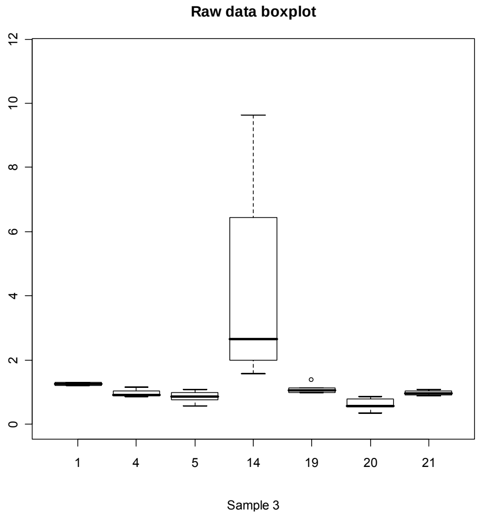
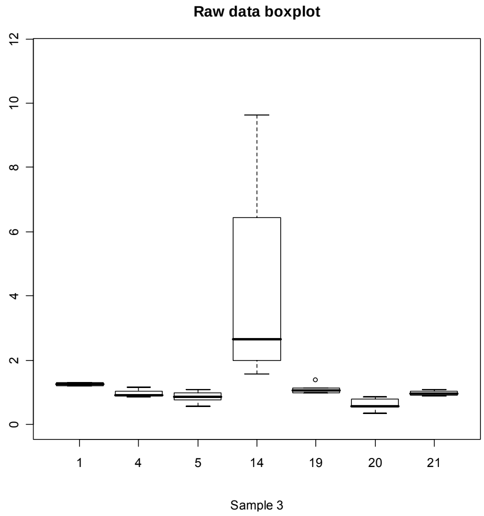
<tr><td>
</td><td>
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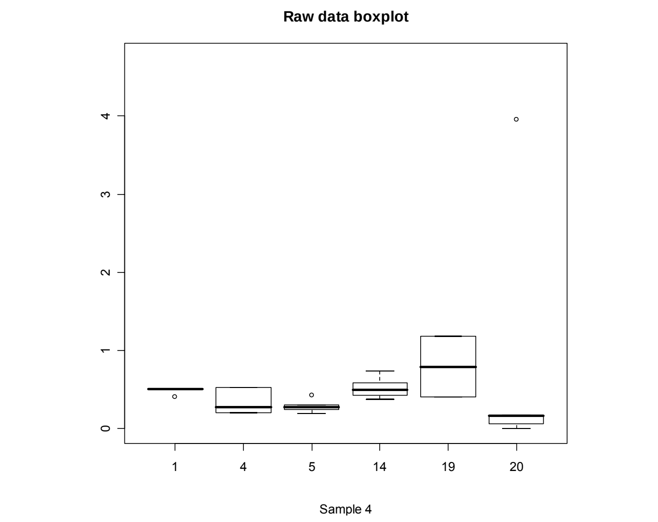
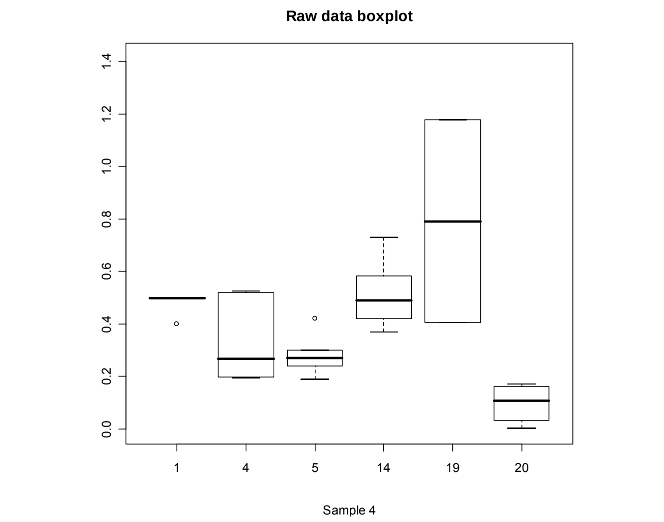
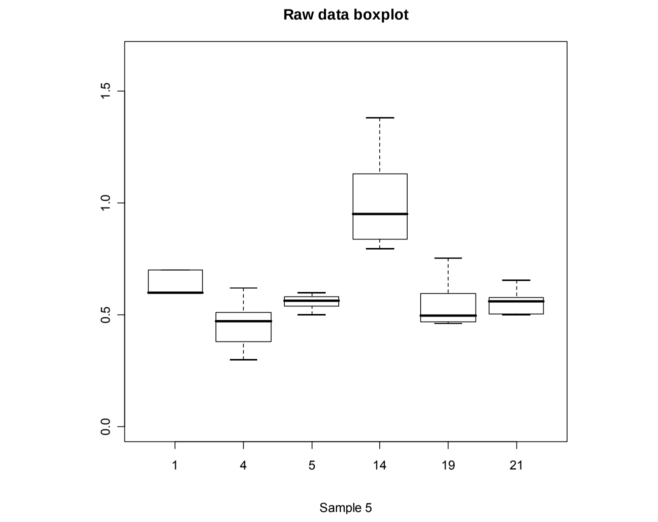
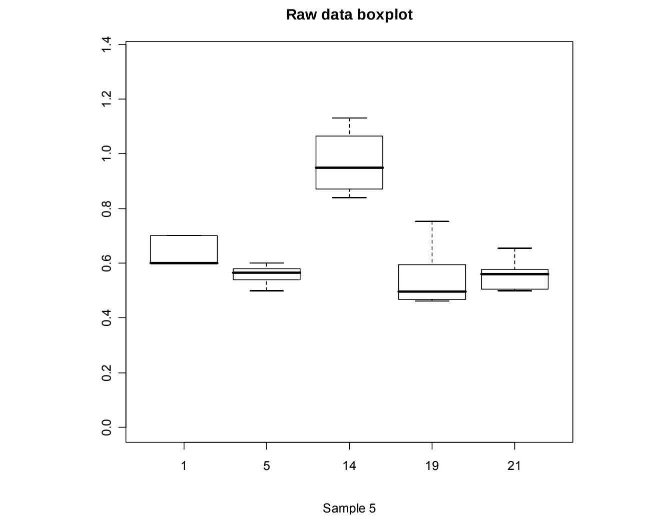








Results are expressed in mg/kg d.m.

Table 124: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Mercury) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	0.35	18
2	0.25	16
3	1.41	42
4	0.49	31
5	0.63	36

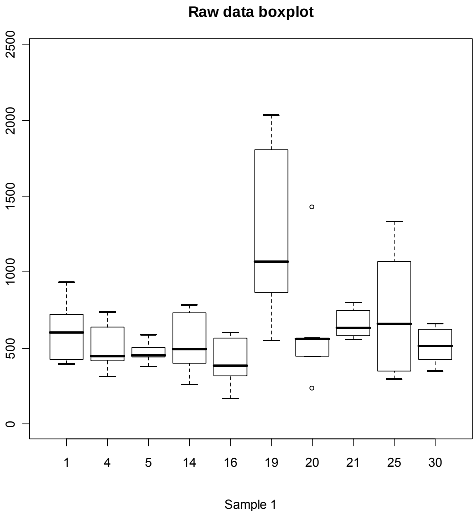
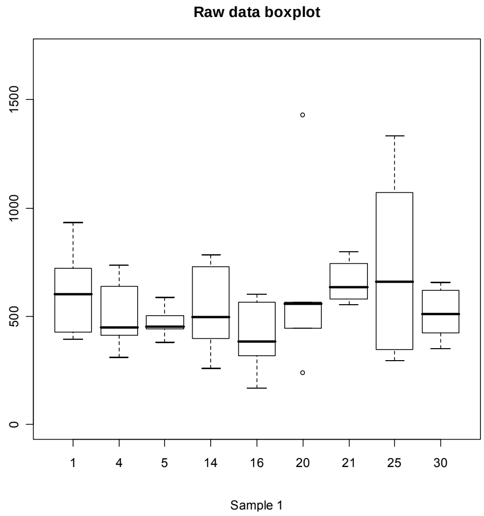
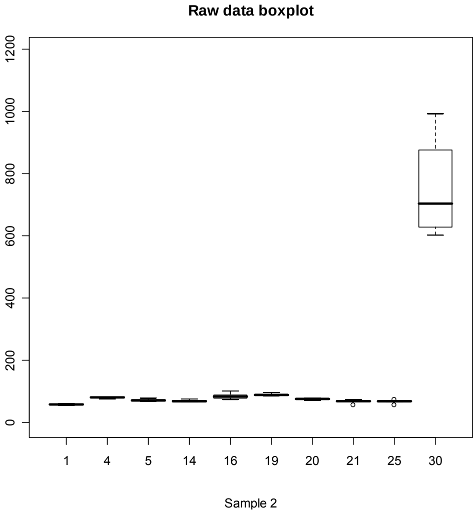
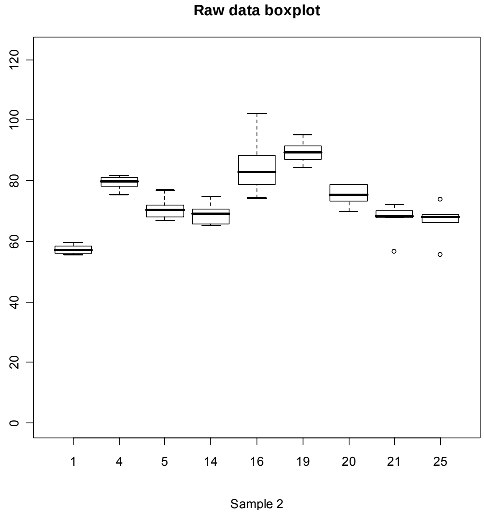
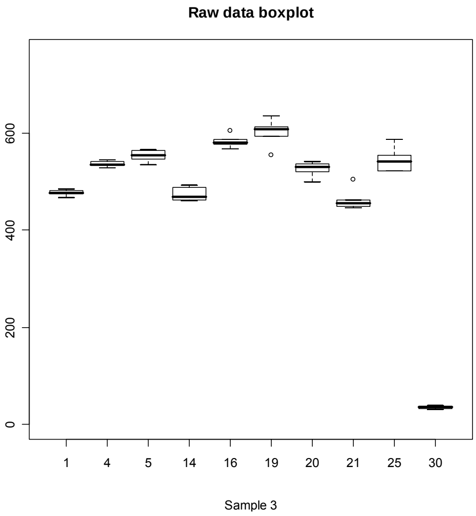
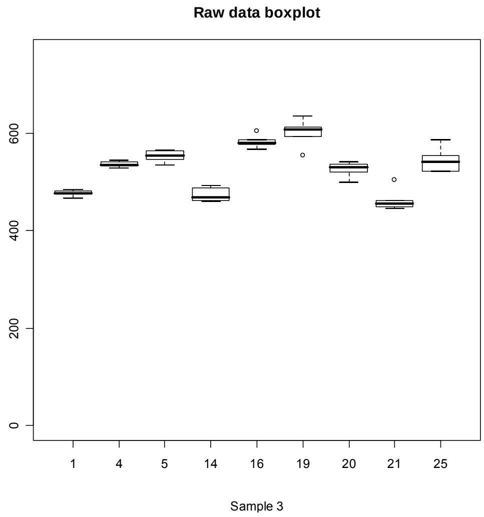
Table 125: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Mercury) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

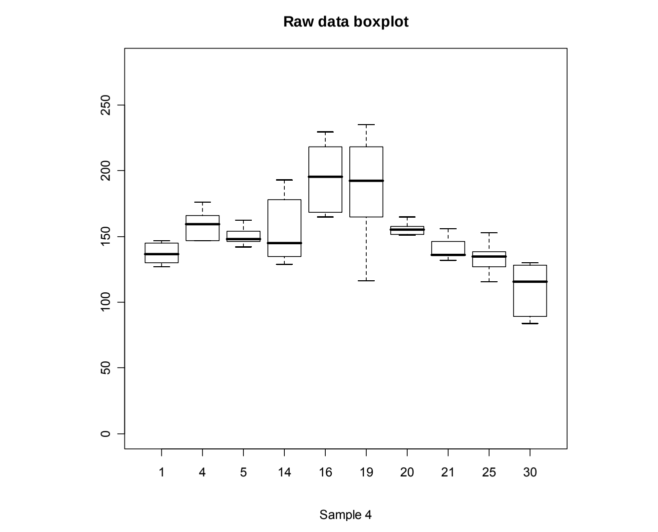
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	0.97	2	0.21	11	0.76	4	108	54	9
2	0.03	2	0.10	11	0.06	2	88	46	8.5
3	53.94	6	4.15	7	47.58	28	252	126	21
4	1.01	5	2.76	7	10.10	24	209	107	19.6
5	1.14	5	0.04	8	0.35	28	252	126	21

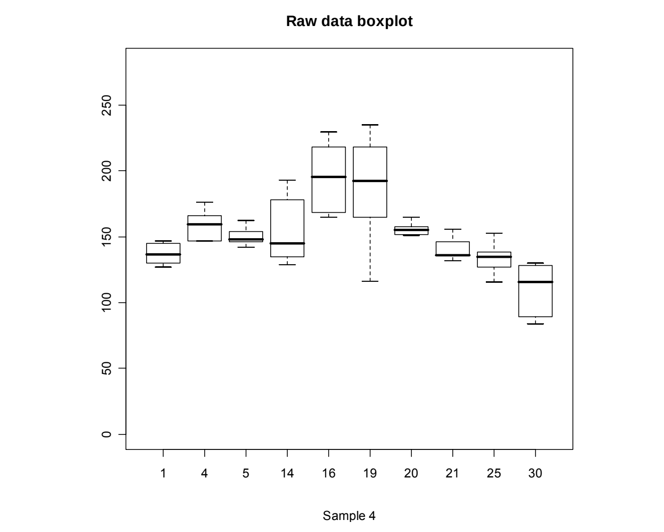
Table 126: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Mercury) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

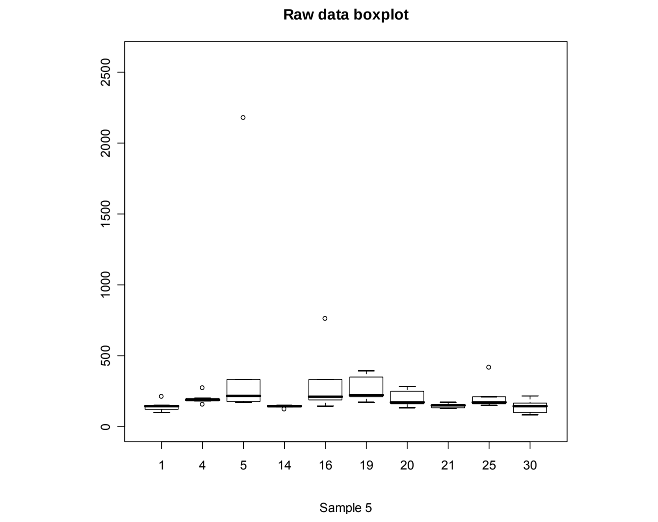
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	NA	NA	0.44	1.232	0.44	1.232
2	NA	NA	0.18	0.504	0.18	0.504
3	NA	NA	1.3	3.64	1.3	3.64
4	NA	NA	0.65	1.82	0.65	1.82
5	NA	NA	0.11	0.308	0.11	0.308

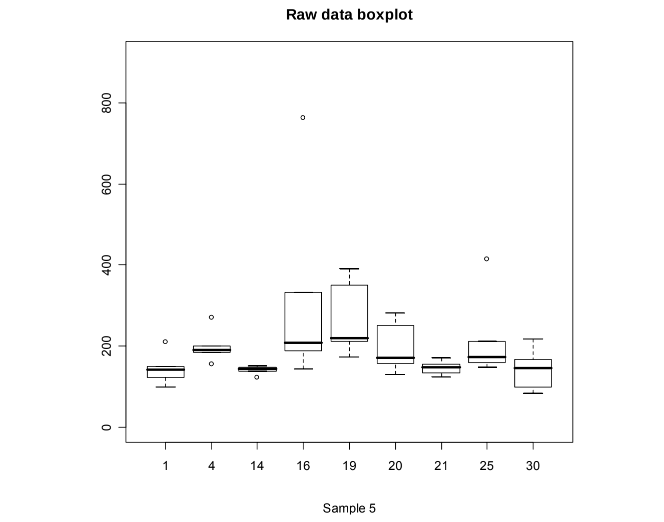
3.5.35 TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Manganese)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	









Results are expressed in mg/kg d.m.

Table 127: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Manganese) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	560	54
2	73	54
3	529	54
4	152	60
5	195	54

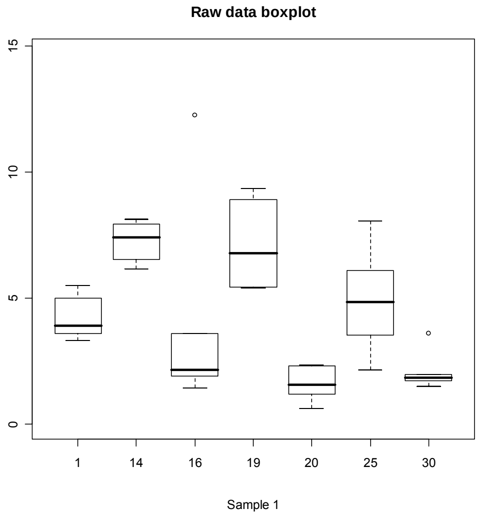
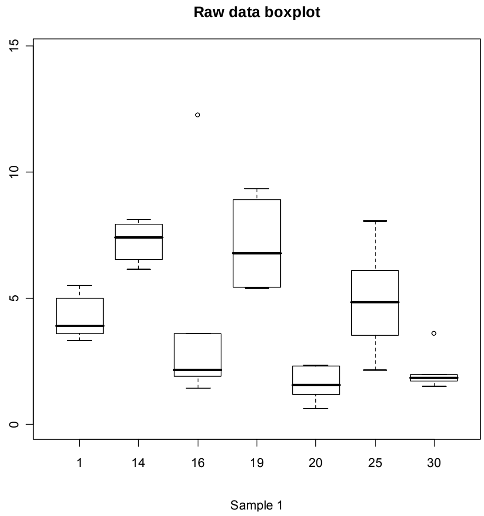
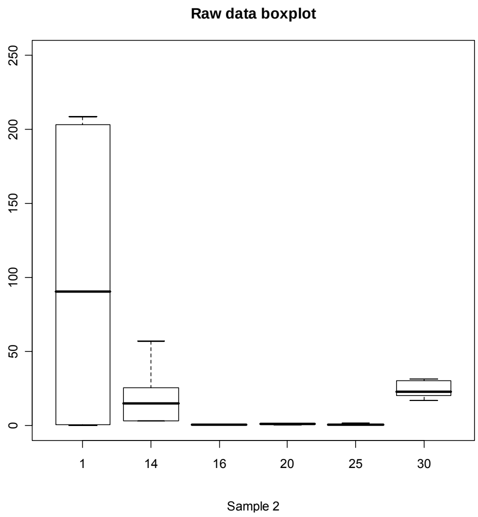
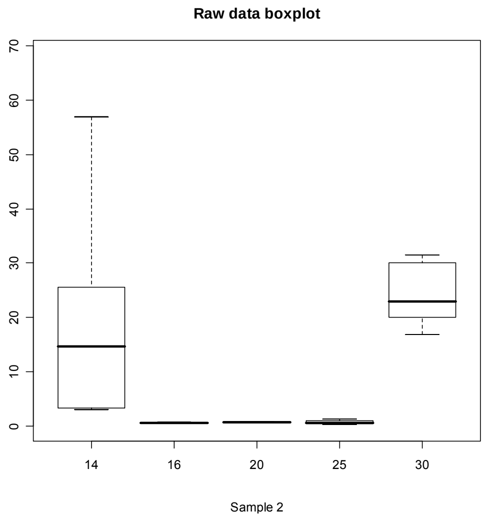
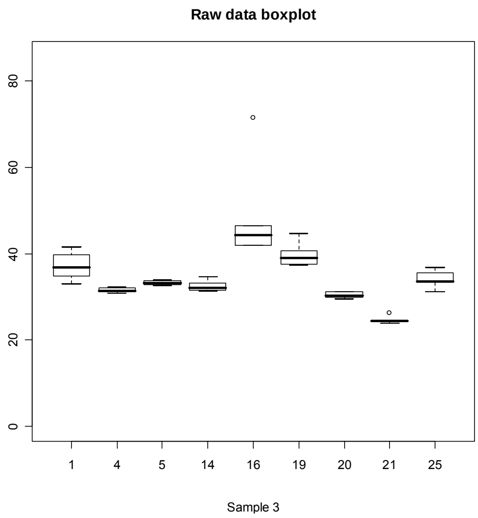
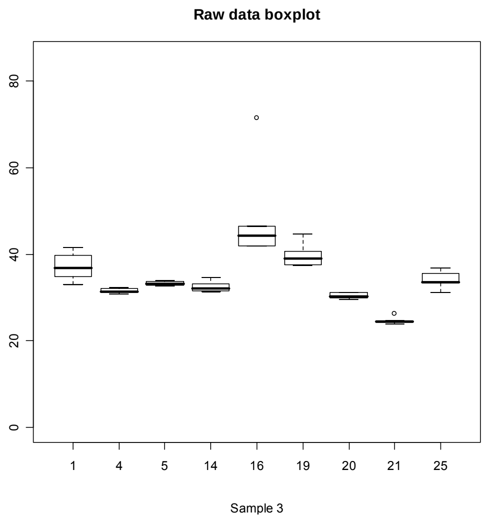
Table 128: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Manganese) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

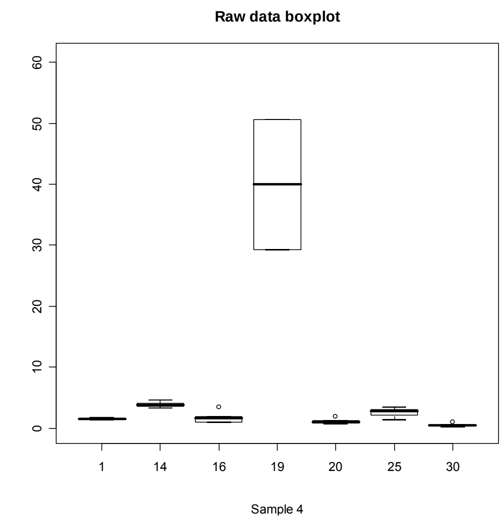
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	514356	8	256761	11	2372456	34	324	162	27
2	4777	8	140	11	969	34	324	162	27
3	116404	8	1470	11	11221	34	324	162	27
4	33261	9	3527	10	16887	40	360	180	30
5	166163	8	89343	11	313470	40	360	180	30

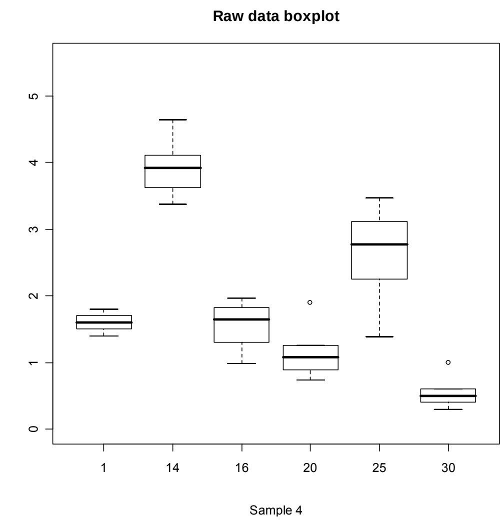
Table 129: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Manganese) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

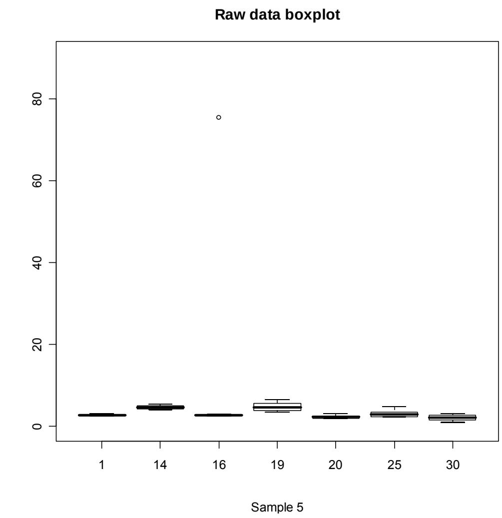
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	NA	NA	264	739.2	264	739.2
2	NA	NA	5	14	5	14
3	NA	NA	18	50.4	18	50.4
4	NA	NA	21	58.8	21	58.8
5	43	10	89	249.2	98	274.4

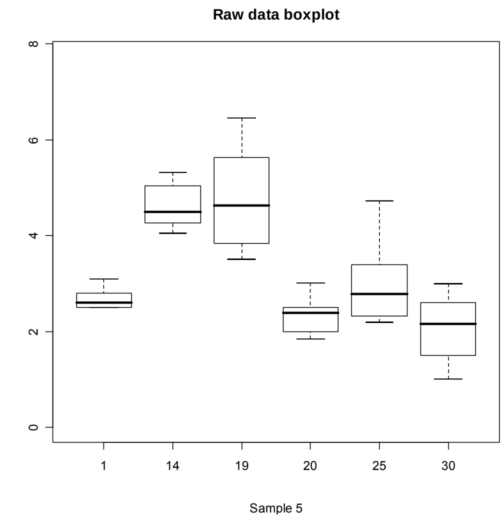
3.5.36 TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Molybdenum)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	









Results are expressed in mg/kg d.m.

Table 130: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Molybdenum) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	4.4	42
2	11.3	24
3	34.6	54
4	2.0	35
5	3.3	36

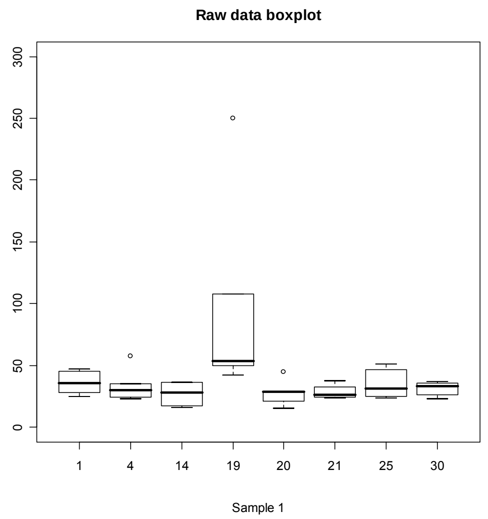
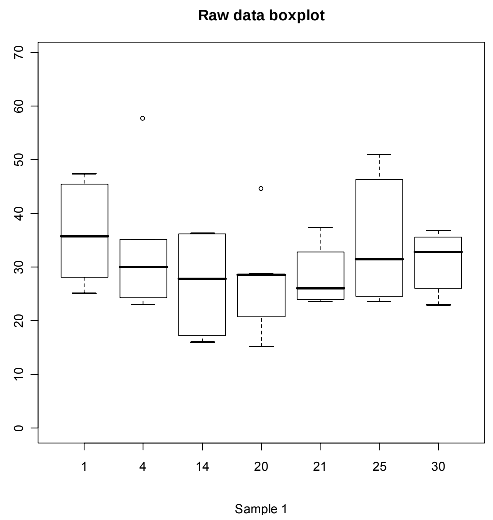
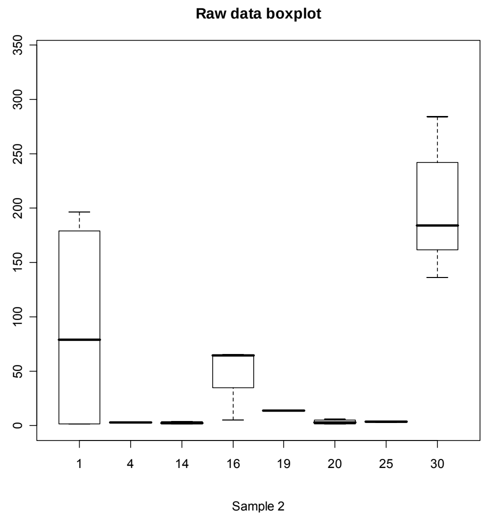
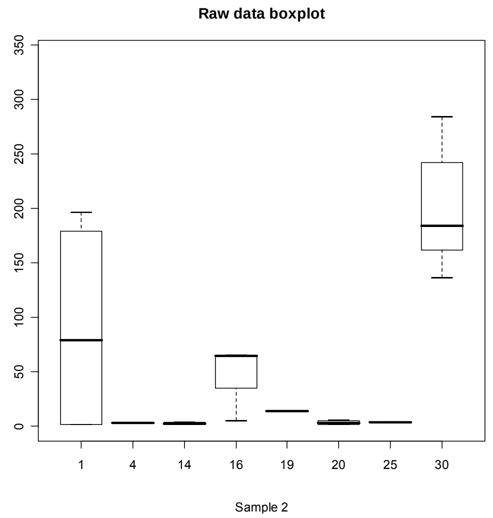
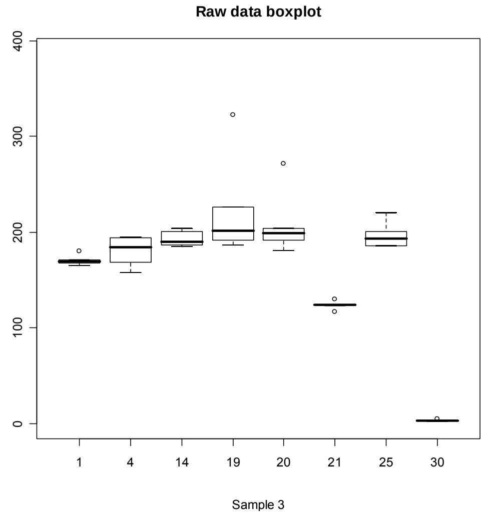
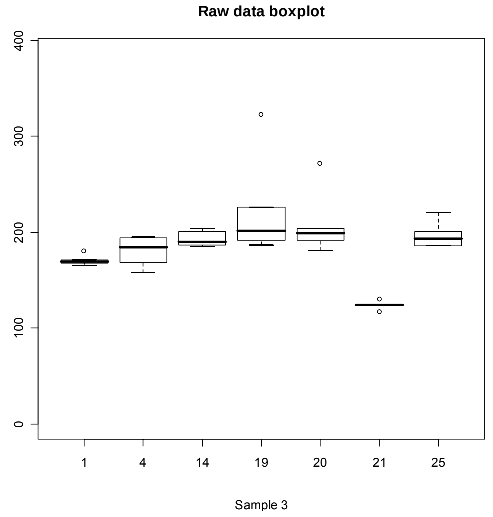
Table 131: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Molybdenum) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	175	6	32	11	103	24	252	126	21
2	2748	4	14	11	2286	20	198	108	21
3	2142	8	153	9	630	36	324	162	27
4	43	5	2	12	7	35	313	157	26.6
5	41	5	3	12	13	36	324	162	27

Table 132: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Molybdenum) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	r	s_{Rj}	R
1	NA	NA	2.07	5.796	2.07	5.796
2	NA	NA	10.69	29.932	10.69	29.932
3	NA	NA	4.18	11.704	4.18	11.704
4	NA	NA	0.44	1.232	0.44	1.232
5	NA	NA	0.6	1.68	0.6	1.68

3.5.37 TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Nickel)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	

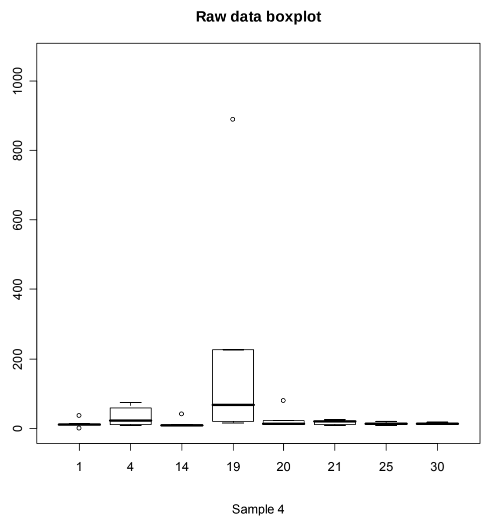
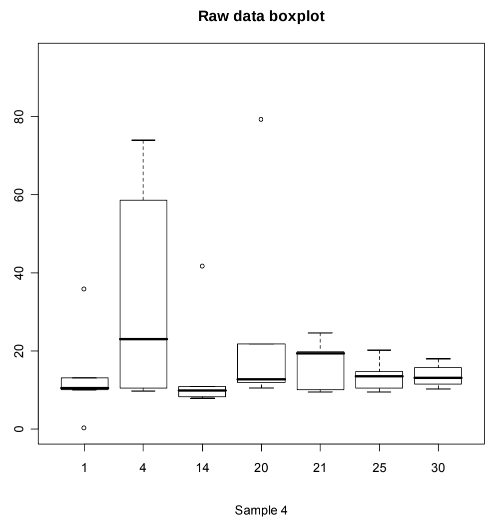
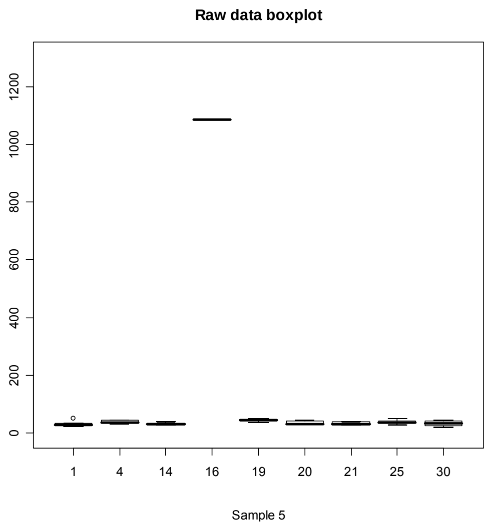
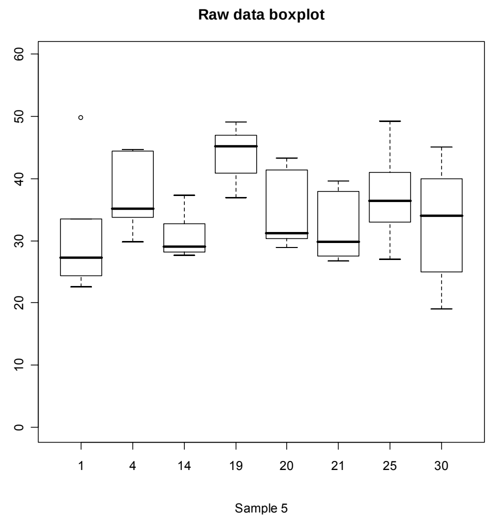
	
	
	Results are expressed in mg/kg d.m.

Table 133: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Nickel) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	31.2	42
2	55.2	35
3	184.9	42
4	18.6	42
5	34.9	48

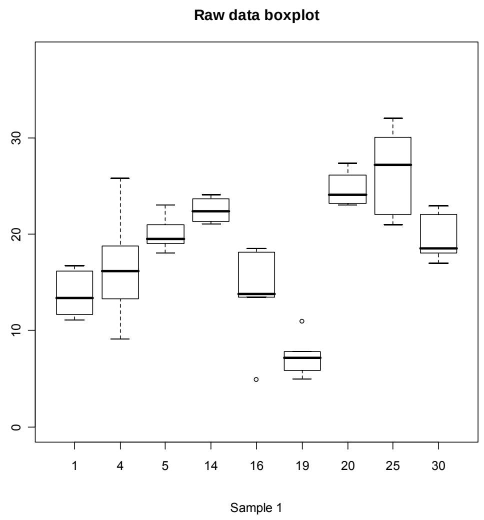
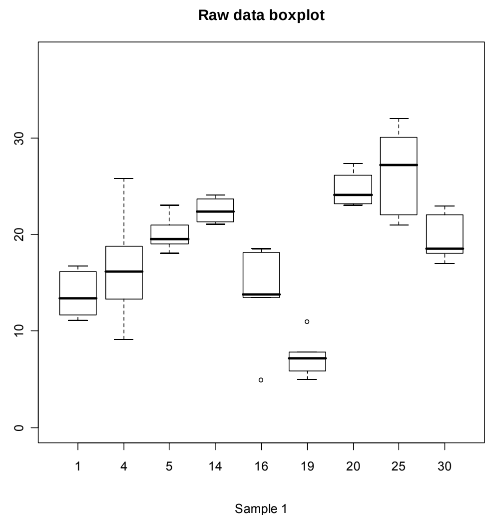
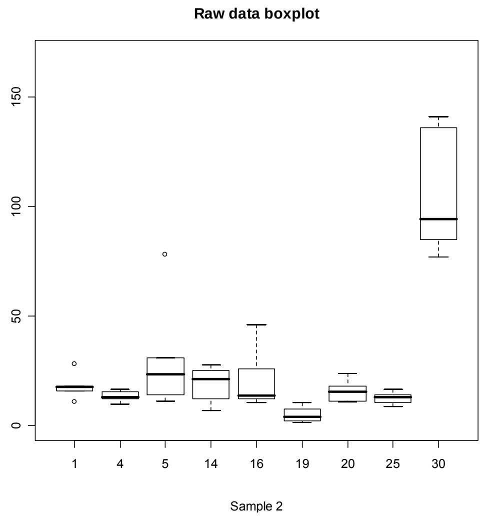
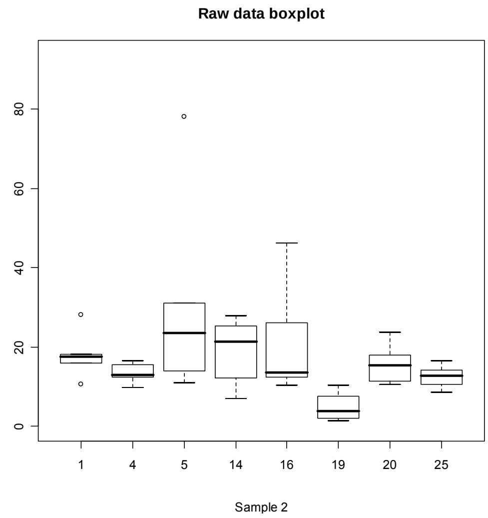
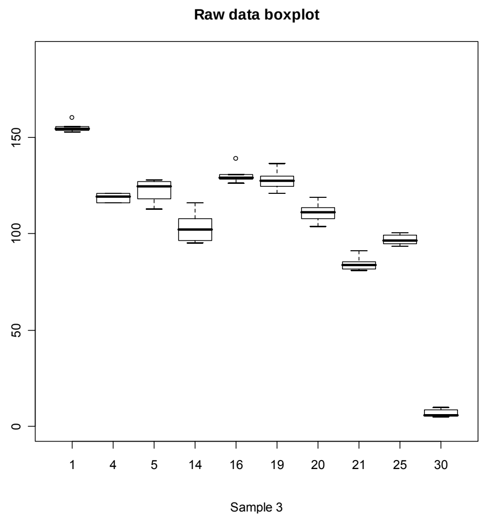
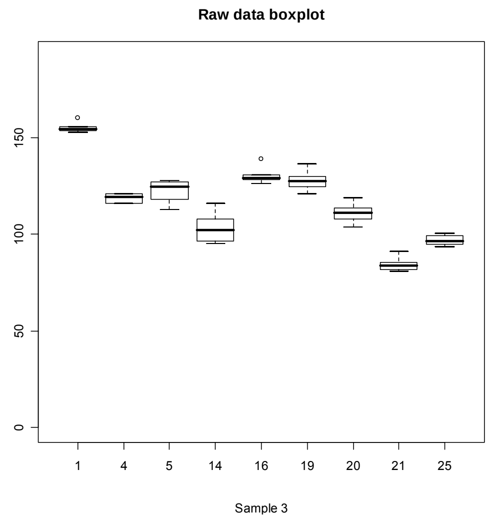
Table 134: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Nickel) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

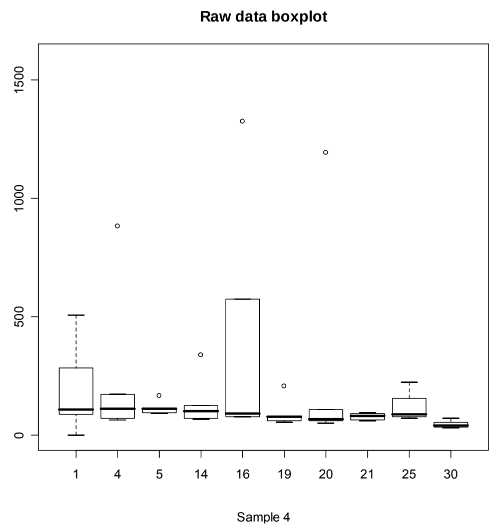
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	492	6	442	9	2730	26	252	126	21
2	184755	7	47189	6	17624	21	191	97	18.66667
3	36118	6	2325	9	18649	32	288	144	24
4	2064	6	904	9	8214	32	288	144	24
5	852	7	445	8	1517	32	288	144	24

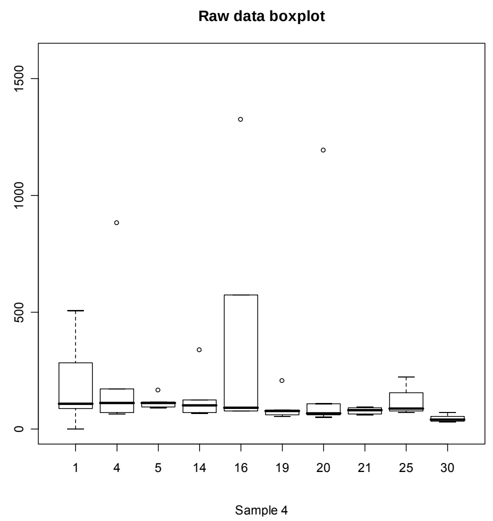
Table 135: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Nickel) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

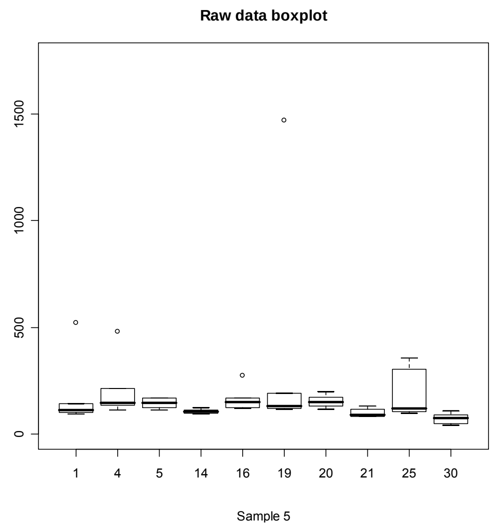
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	NA	NA	10	28	10	28
2	68	51	29	81.2	74	207.2
3	NA	NA	24	67.2	24	67.2
4	NA	NA	16	44.8	16	44.8
5	3	2	7	19.6	8	22.4

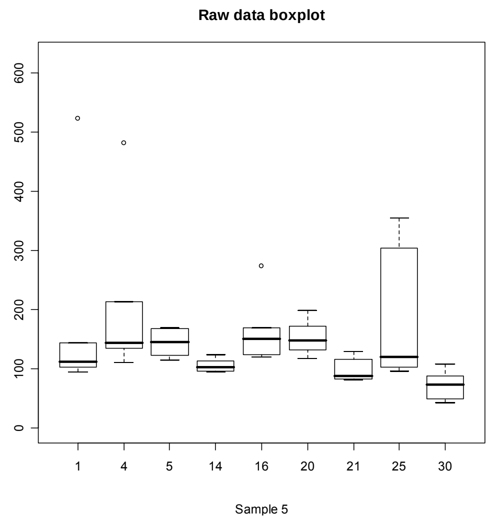
3.5.38 TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Lead)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	









Results are expressed in mg/kg d.m.

Table 136: TS 15411 A (a aqua regia, HF, boric acid and micro-wave assisted digestion: Lead) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	18	54
2	17	47
3	117	54
4	163	60
5	145	54

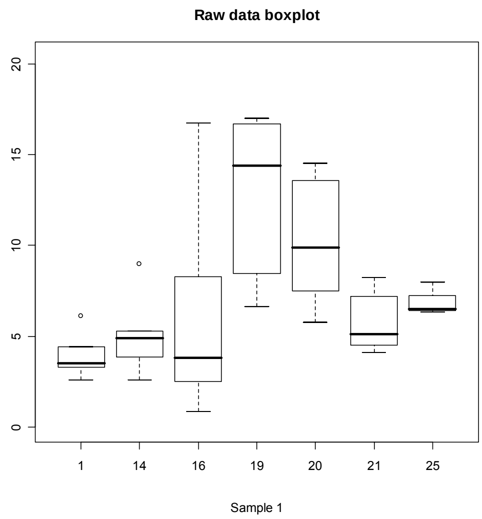
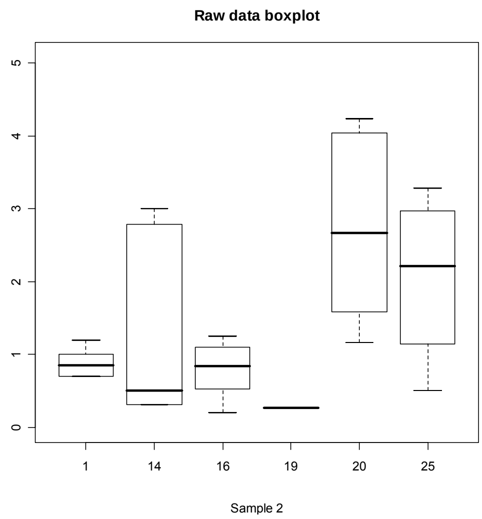
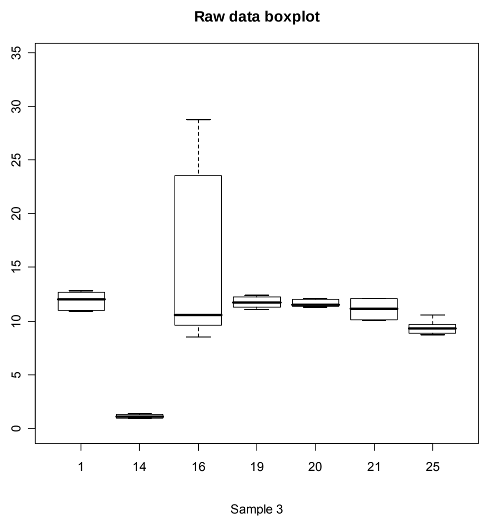
Table 137: TS 15411 A (a aqua regia, HF, boric acid and micro-wave assisted digestion: Lead) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	1772	8	56	11	449	34	324	162	27
2	2062	7	826	12	3952	33	313	157	26.6
3	20864	8	147	11	911	34	324	162	27
4	536311	9	450890	10	2650868	40	360	180	30
5	94902	8	95711	11	237987	40	360	180	30

Table 138: TS 15411 A (a aqua regia, HF, boric acid and micro-wave assisted digestion: Lead) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	NA	NA	4	11.2	4	11.2
2	NA	NA	11	30.8	11	30.8
3	NA	NA	5	14	5	14
4	NA	NA	257	719.6	257	719.6
5	19	32	77	215.6	80	224

3.5.39 TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Antimony)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	

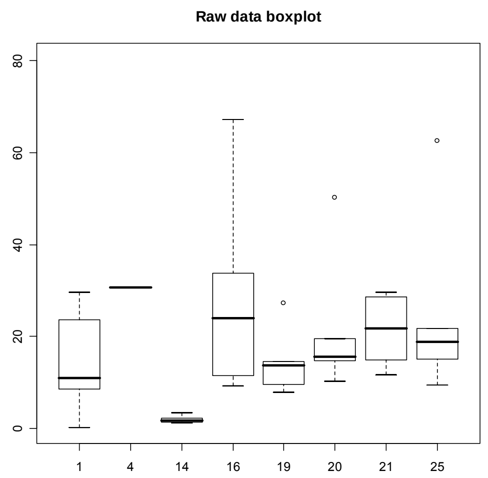
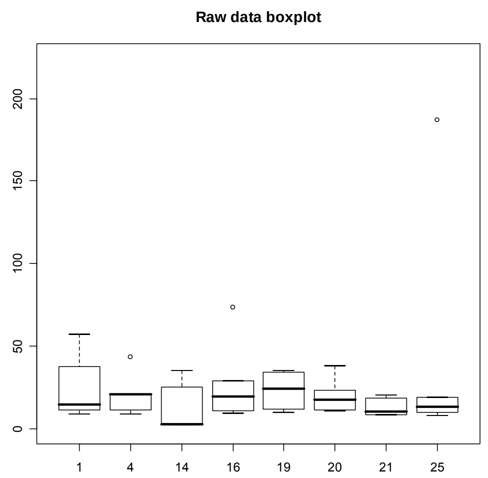
Sample 4	
Sample 5	
	Results are expressed in mg/kg d.m.

Table 139: TS 15411 A (a a aqua regia, HF, boric acid and micro-wave assisted digestion: Antimony) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	7.28	39
2	1.46	29
3	10.31	42
4	18.20	43
5	22.71	47

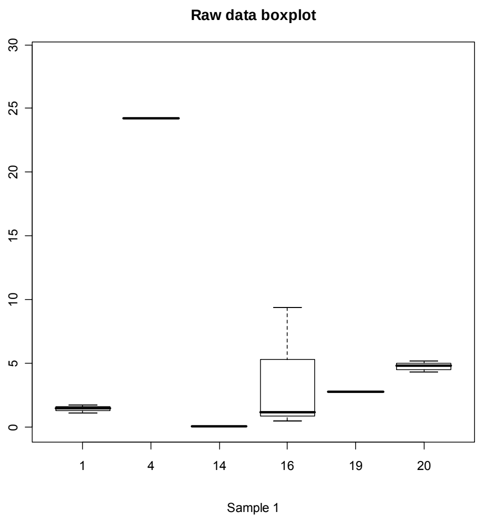
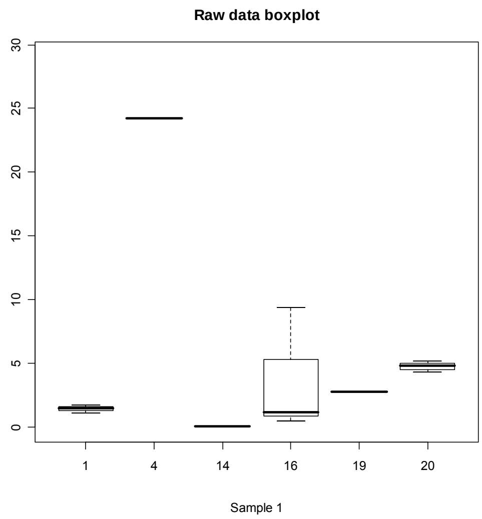
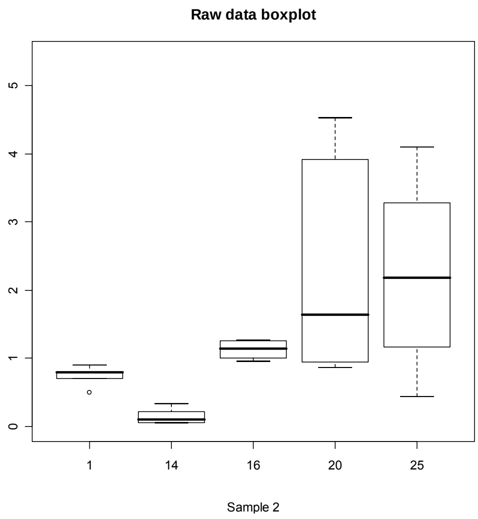
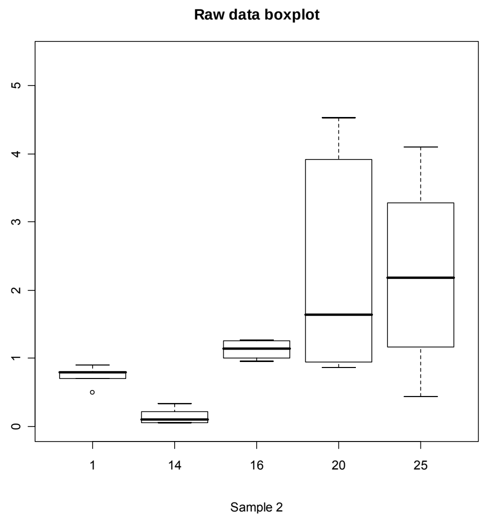
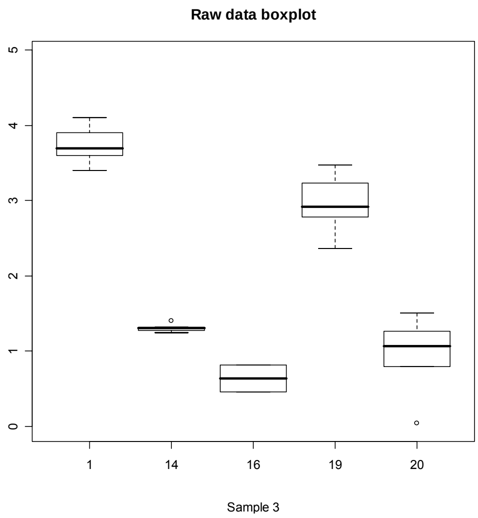
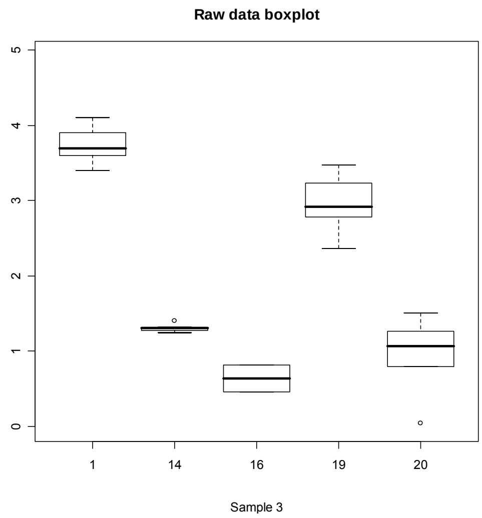
Table 140: TS 15411 A (a a aqua regia, HF, boric acid and micro-wave assisted digestion: Antimony) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	365	6	81	9	288	23	225	113	19.667
2	17	5	13	9	9	17	170	88	17.167
3	700	6	179	9	206	26	252	126	21
4	2883	7	407	7	6012	28	253	127	22
5	3657	7	6831	8	26385	31	277	139	23.6

Table 141: TS 15411 A (a a aqua regia, HF, boric acid and micro-wave assisted digestion: Antimony) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	r	s_{Rj}	R
1	NA	NA	3.54	9.912	3.54	9.912
2	0.5	0.75	0.74	2.072	0.89	2.492
3	3.95	2.26	2.82	7.896	4.85	13.58
4	NA	NA	14.65	41.02	14.65	41.02
5	NA	0.97	29.17	81.676	29.17	81.676

3.5.40 TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Selenium)

Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*
	
	
	

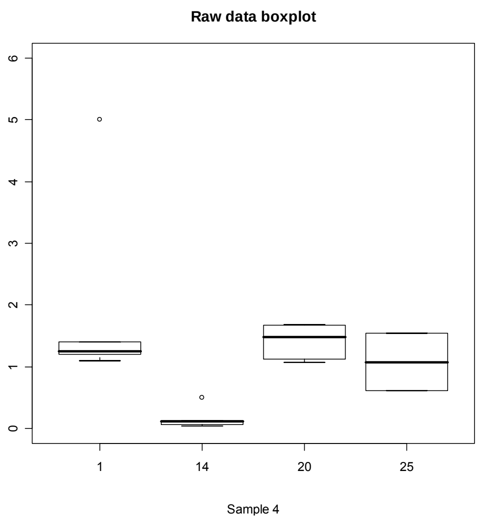
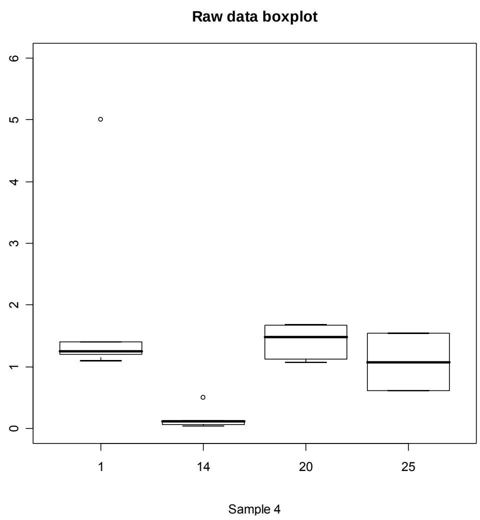
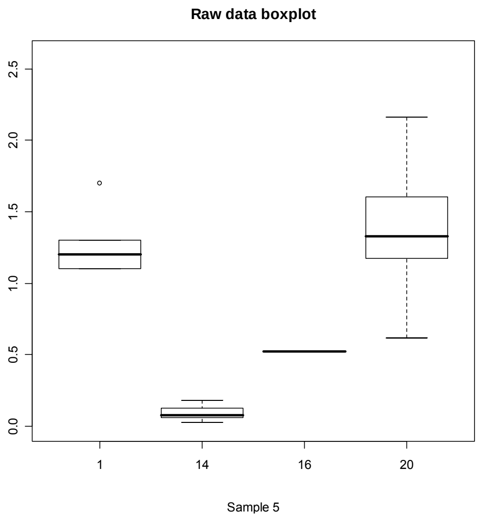
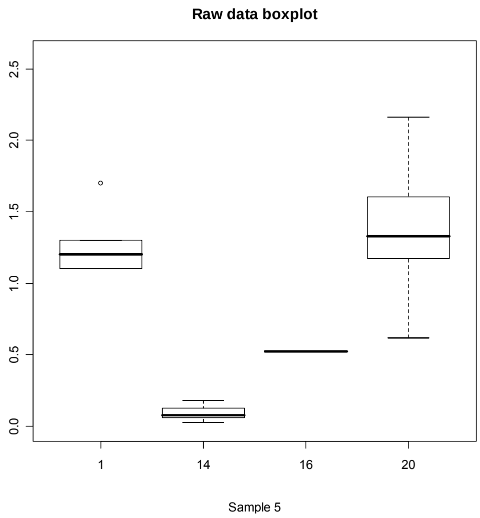
	
	
	N.B. Data set to small for meaningful outlier testing *Results are expressed in mg/kg d.m.*

Table 142: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Selenium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	3.29	23
2	1.24	26
3	2.16	25
4	1.12	19
5	0.89	19

Table 143: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Selenium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

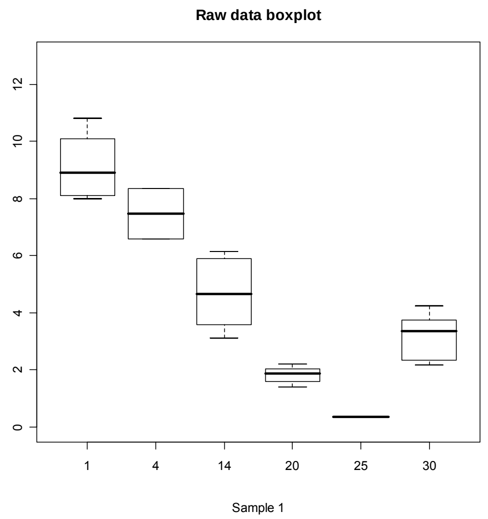
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	534.9	5	15.7	4	34.4	13	119	61	12.66667
2	18.7	4	14.7	7	5.5	20	176	92	17
3	35.1	4	1.0	6	1.4	20	173	89	16.6
4	9.3	3	3.0	8	9.8	18	162	82	15.2
5	6.2	3	0.0	7	1.6	19	170	86	15.6

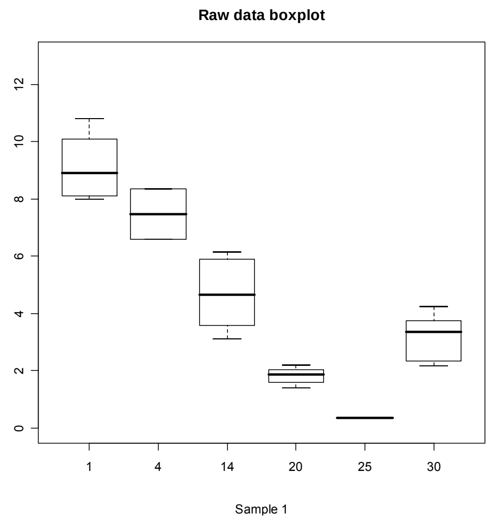
Table 144: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Selenium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

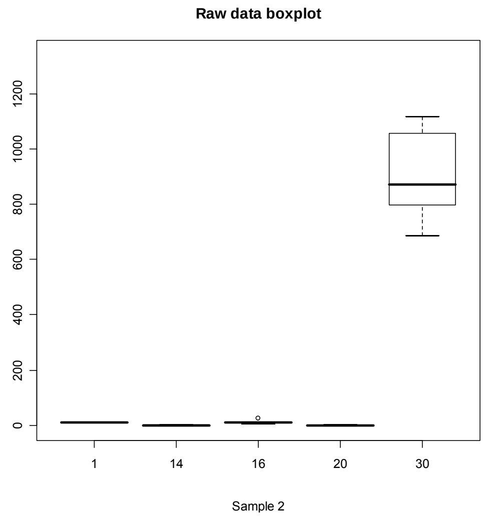
Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	5.4	0.7	1.6	4.48	5.6	15.68
2	0.5	0.9	0.5	1.4	0.7	1.96
3	1.2	0.2	0.3	0.84	1.2	3.36
4	NA	NA	0.7	1.96	0.7	1.96
5	NA	NA	0.3	0.84	0.3	0.84

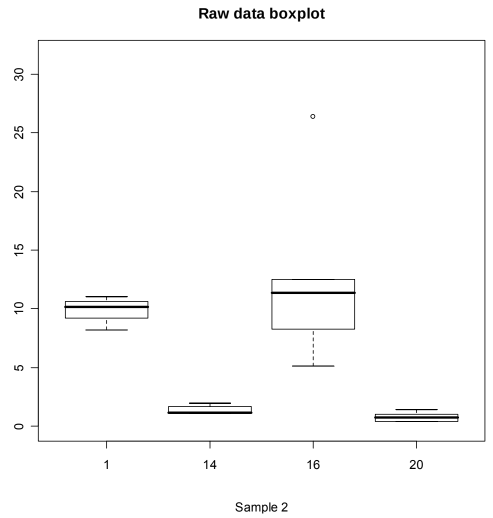
3.5.41 TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Vanadium)

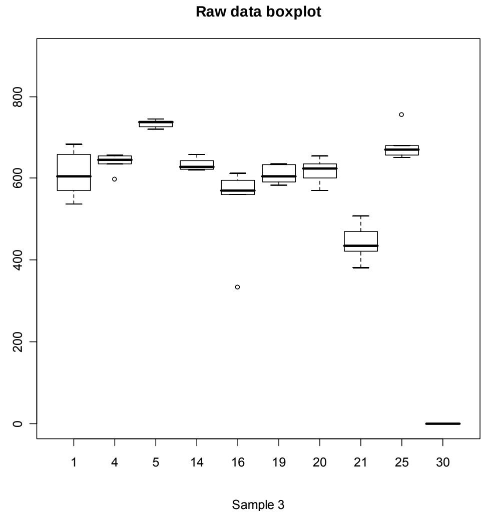
Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*

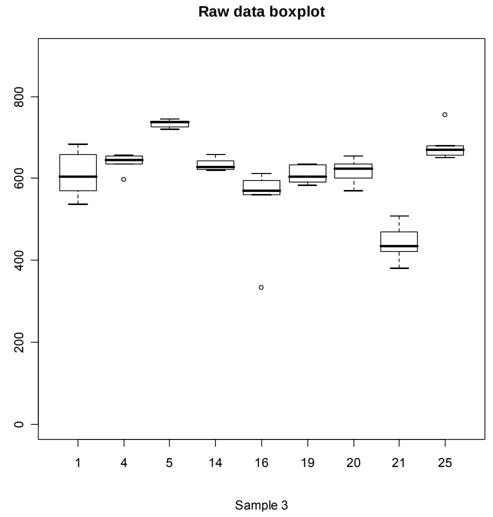












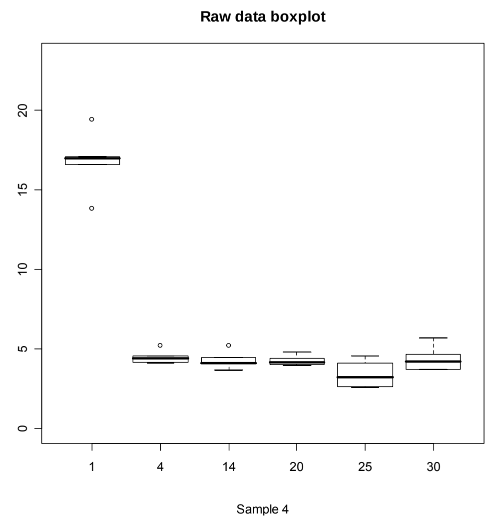
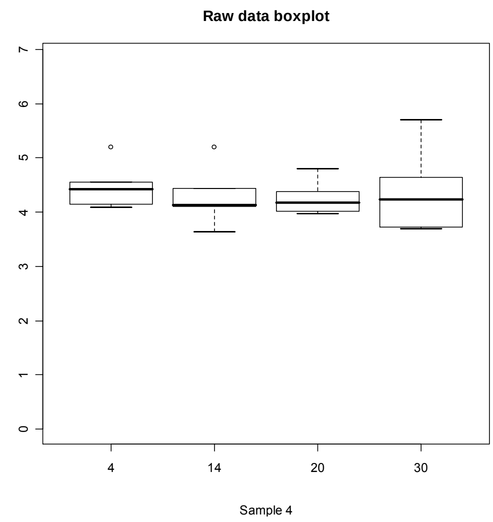
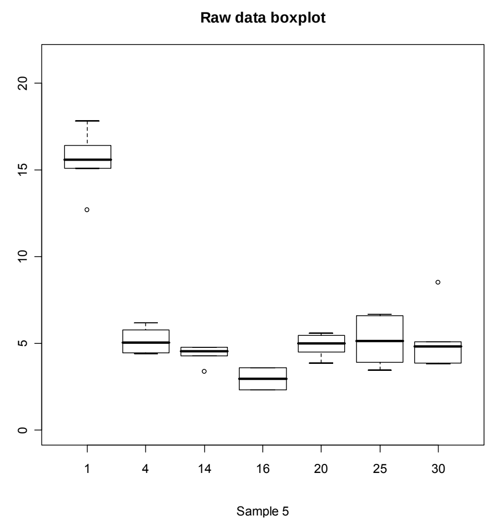
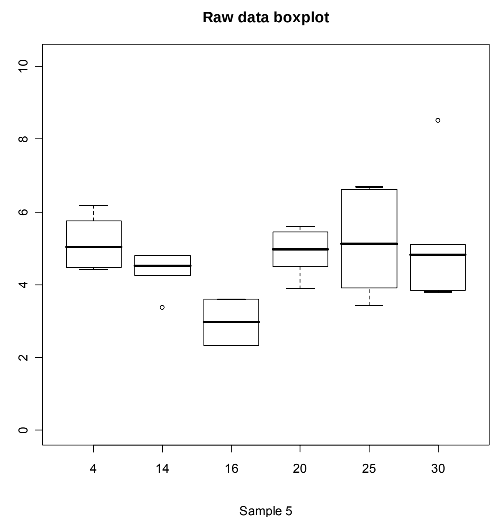
	
	
	Results are expressed in mg/kg d.m.

Table 145: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Vanadium) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	4.8	27
2	5.9	23
3	611.6	54
4	4.1	29
5	4.8	32

Table 146: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Vanadium) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS_{.Hj}$, ni_{Hj}) and for repeatability ($SS_{.rj}$, ni_{rj})

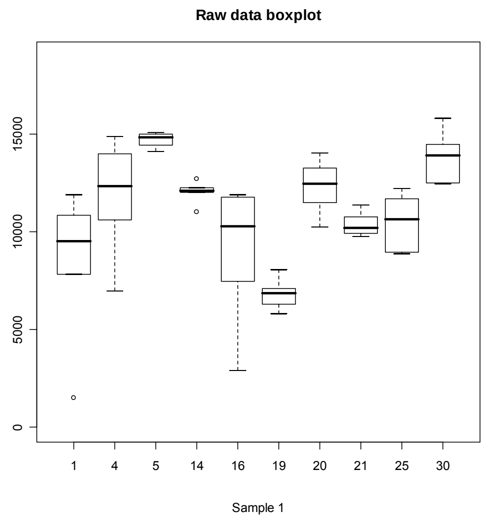
Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	215	5	2	10	18	11	149	77	15
2	611	3	122	13	150	13	170	86	15.6
3	328910	8	19477	9	76646	36	324	162	27
4	5	4	1	13	7	35	313	157	26.6
5	10	5	11	12	21	32	292	146	25

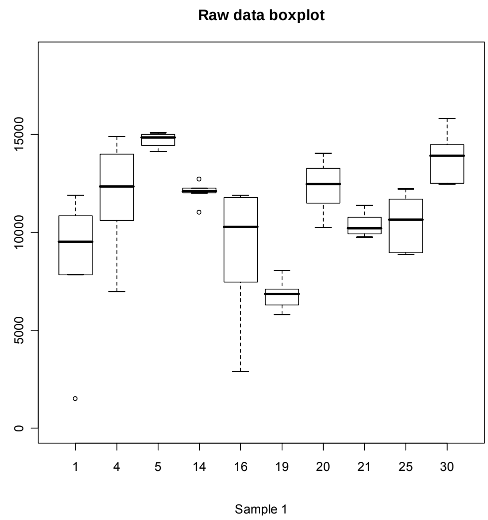
Table 147: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Vanadium) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

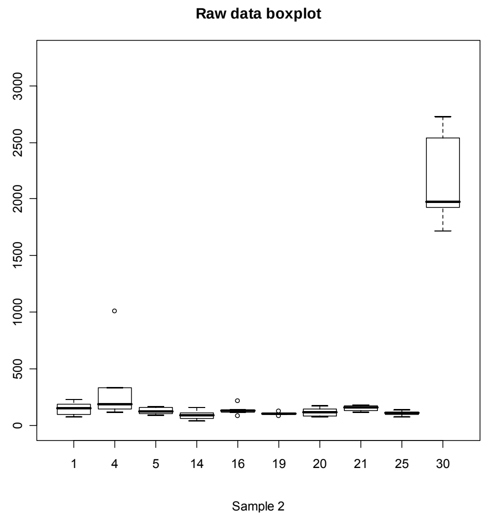
Level	s_{Lj}	s_{Hj}	s_{rj}	r	s_{Rj}	R
1	NA	NA	1.28	3.584	1.28	3.584
2	NA	NA	3.39	9.492	3.39	9.492
3	80.57	3.42	46.14	129.192	92.85	259.98
4	NA	NA	0.46	1.288	0.46	1.288
5	0.31	0.33	0.82	2.296	0.88	2.464

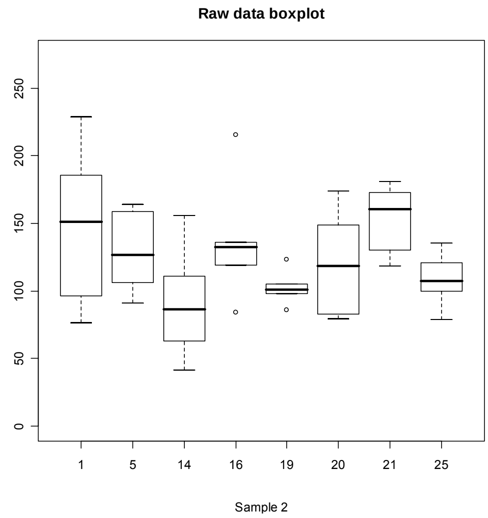
3.5.42 TS 15411 A - EN 13656 (aqua regia, HF, boric acid and micro-wave assisted digestion: Zinc)

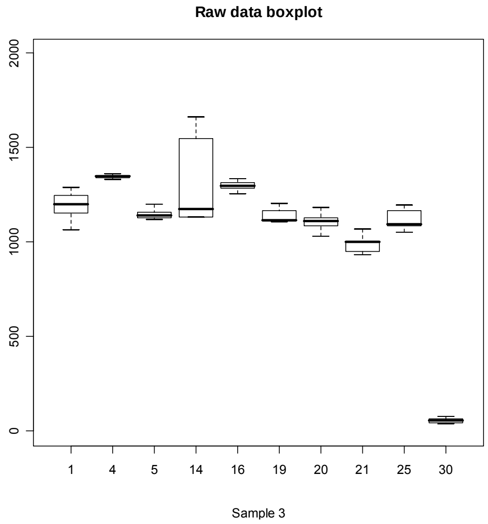
Submitted raw data prior statistical treatment	*Retained raw data after statistical treatment*

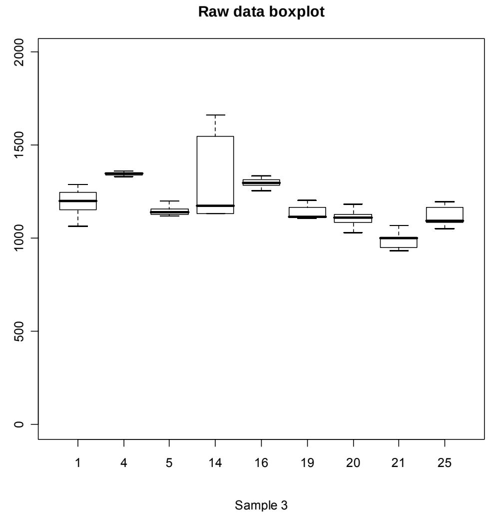


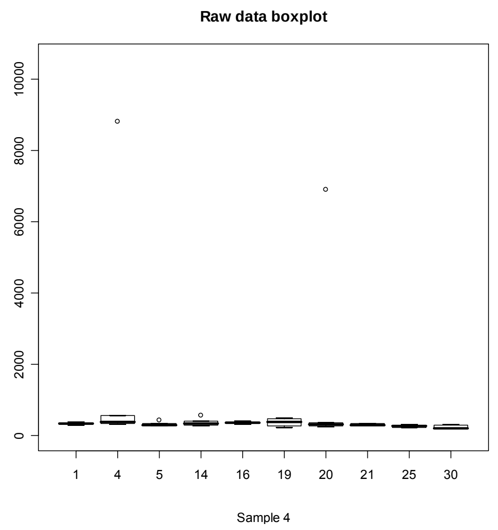


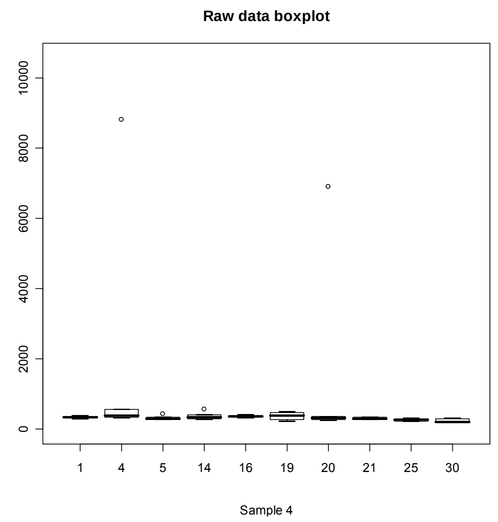


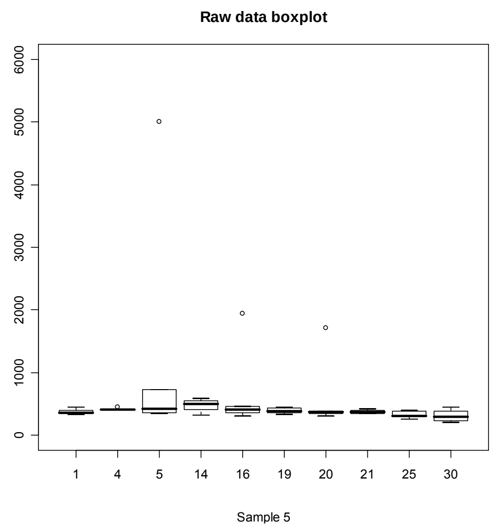


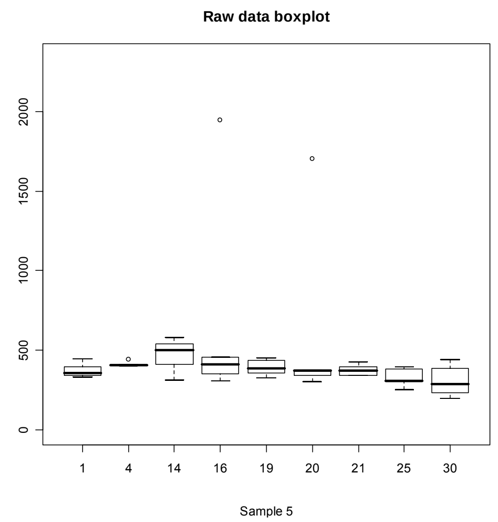












Results are expressed in mg/kg d.m.

Table 148: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Zinc) - General Averages. Results are expressed in mg/kg d.m.

Level	m_j	*Number of valid results*
1	11020	60
2	147	54
3	1183	54
4	573	60
5	431	54

Table 149: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Zinc) - Sums of squares and degrees of freedom for laboratory effects (SS_{Lj}, ni_{Lj}), for bottle effects ($SS._{Hj}$, ni_{Hj}) and for repeatability ($SS._{rj}$, ni_{rj})

Level	SS_{Lj}	ni_{Lj}	SS_{Hj}	ni_{Hj}	SS_{rj}	ni_{rj}	K_j	K'_j	K''_j
1	3.22E+08	9	58576414	10	1.5E+08	40	360	180	30
2	252931.6	8	87232.98	11	542494.1	40	360	180	30
3	621873.1	8	178401	11	193006.4	40	360	180	30
4	16374777	9	20087057	10	75161106	40	360	180	30
5	635117.8	8	830130.1	11	2854582	40	360	180	30

Table 150: TS 15411 A (aqua regia, HF, boric acid and micro-wave assisted digestion: Zinc) - Standard deviations for laboratory effects (s_{Lj}), for between-bottle effects (s_{Hj}) , for repeatability (s_{rj}) and for reproducibility (s_{Rj}). Results are expressed in mg/kg d.m.

Level	s_{Lj}	s_{Hj}	s_{rj}	*r*	s_{Rj}	*R*
1	2234	839	1935	5418	2956	8276.8
2	NA	NA	116	324.8	116	324.8
3	93	65	69	193.2	116	324.8
4	NA	208	1371	3838.8	1371	3838.8
5	21	39	267	747.6	268	750.4

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5 ANNEX 1 – Reporting forms used for data submission

European Commission

EUR 23552 EN – Joint Research Centre – Institute for Environment and Sustainability
Title: QUOVADIS Project - Organization of Validation Exercises
Author(s): VACCARO S, LOCORO G, CONTINI S, ROEBBEN G, EMTEBORG P, CICERI G, GIOVE A, IACOBELLIS S, FLAMME S, MAIER J, GAWLIK B
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