Institute for Reference Materials and Measurements

ERM European Reference Materials

CERTIFICATION REPORT

Certification of a Master Batch of Charpy V-Notch Reference Test Pieces of Nominal Energy Level 30 J for Tests at 0 °C

Certified Reference Material ERM®-FA013ay

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Certification of a Master Batch of Charpy V-Notch Reference Test Pieces of Nominal Energy Level 30 J for Tests at 0 °C

Certified Reference Material ERM®-FA013ay

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Disclaimer

Certain commercial equipment, instruments, and materials are identified in this report to specify adequately the experimental procedure. In no case does such identification imply recommendation or endorsement by the European Commission, nor does it imply that the material or equipment is necessarily the best available for the purpose.

Summary

This certification report describes the processing and characterisation of ERM®-FA013ay, a batch of steel Charpy V-notch certified reference test pieces for tests at 0 °C. This batch will serve as a Master Batch, to be used by IRMM for the certification of secondary batches. Sets of five pieces taken from a secondary batch are distributed by IRMM and its authorised distributors for the verification of pendulum impact test machines according to EN 10045-2 [1] and ISO 148-2 [2].

The certified values for *KV* (= absorbed energy = energy required to break a V-notched test piece using a pendulum impact test machine) are estimates of the mean value of the whole batch. The obtained values, deduced from tests at 0 °C, are shown in the table below. The associated uncertainties are standard uncertainties corresponding to a confidence level of about 68 %. The certified values are defined by the Charpy impact test method as described in EN 10045-1 [3] and ISO 148-1 [4] and are traceable to the International System of Units (SI).

	Certified value KV_{MB} **[J]**	**Standard uncertainty** u_{MB} **[J]**
ERM®-FA013ay	26.06	0.35

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Glossary

AISI	American Iron and Steel Institute
ASTM	American Society for Testing and Materials
BCR	Community Bureau of Reference
CRM	Certified Reference Material
EC	European Commission
EN	European Norm
Eq.	Equation
ERM®	European Reference Material trademark
g	Gravitational acceleration
HRC	Hardness value according to the Rockwell C method of indentation
IMB	International Master Batch
IRMM	Institute for Reference Materials and Measurements
ISO	International Organization for Standardization
JRC	Joint Research Centre
k	Coverage factor
KV	Absorbed energy = energy required to break a V-notched test piece of defined shape and dimensions when tested with a pendulum impact testing machine
KV_{char}	Mean of *p* accepted mean *KV* values
KV_{MB}	Certified *KV* value of the master batch test pieces
LNE	Laboratoire National de Métrologie et d'Essais
MB	Master Batch
m	Mass of pendulum
p	Number of labs contributing to the set of accepted data
RM	Reference Material

RSD	Relative Standard Deviation
s	Displacement of the hammer
SB	Secondary Batch
s_{char}	Standard deviation of the *p* accepted mean values of the laboratories participating in the characterisation study
SD	Standard deviation
s_{hom}	Homogeneity contribution to the uncertainty of the laboratory mean values
SI	International System of Units
s_l	Lowest within-laboratory standard deviation
s_m	Highest within-laboratory standard deviation
u_{char}	Standard uncertainty of KV_{char}
u_{hom}	Standard uncertainty component from homogeneity
u_{lts}	Standard uncertainty component corresponding to long-term stability
u_{MB}	Standard uncertainty of KV_{MB}
u_{sts}	Standard uncertainty component corresponding to short-term stability
W_t	Total impact energy (absorbed energy as measured in an instrumented impact test)
Δh	Difference between the height of the centre of gravity of the pendulum prior to release and at end of first half-swing, after breaking the test sample
δKV_{hom}	Error term due to variation between samples
δKV_{lts}	Error term due to long-term instability of the reference material
δKV_{sts}	Error term due to short-term instability of the reference material
ν_{eff}	Effective number of degrees of freedom associated with the uncertainty of the certified value

1 Introduction: the Charpy pendulum impact test

The Charpy pendulum impact test is designed to assess the resistance of a material to impact loading. The test, which consists of breaking a notched bar of the test material using a hammer rotating around a fixed horizontal axis, is schematically presented in Figure 1.

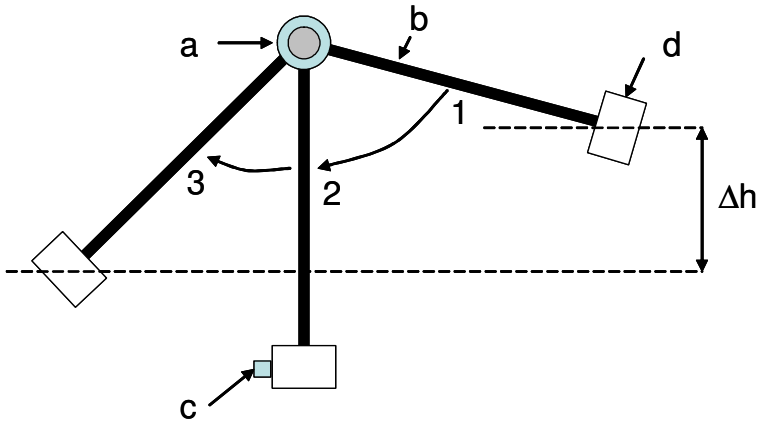

Figure 1: Schematic presentation of the Charpy pendulum impact test, showing a: the horizontal rotation axis of the pendulum, b: the hammer, of mass m, consisting of a stiff shaft onto which is fixed d: the striker. The hammer is released from a defined height (position 1). The hammer strikes c: the test sample, when the hammer has reached maximum kinetic energy (shaft in vertical position 2). The height reached by the hammer after having broken the sample (position 3) is recorded. The difference in height between position 1 and 3 (Δh) corresponds to a difference in potential energy (= m × g × Δh, with g = gravitational acceleration), and is a measure of the energy required to break the test sample.

The energy absorbed by the test sample depends on the impact pendulum construction and its dynamic behaviour. Methods to verify the performance of an impact pendulum require the use of reference test pieces as described in European, ISO and American standards [1, 2, 5]. The reference test pieces dealt with in this report comply with a V-notched test piece of well-defined geometry [1, 6], schematically shown in Figure 2.

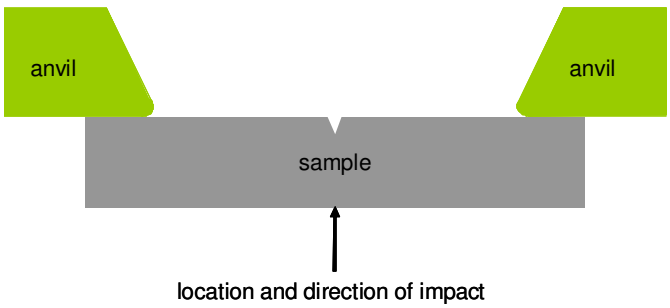

Figure 2: Schematic drawing of a V-notched Charpy sample (top-view when sample is in place for test), indicating the location and direction of the impact.

2 The concept of master batch and secondary batch

This report describes the production of a “Master Batch” (MB) of Charpy V-notch certified reference test pieces. This work was performed in accordance with procedures described in the BCR reports [7] and [8], and in compliance with the ISO Guide 34 [9] requirements for the producers of certified reference materials (CRMs). IRMM is accredited by BELAC for the production of Charpy reference materials according to ISO Guide 34. The certified value of a master batch is obtained using an international interlaboratory comparison, in accordance with ISO Guide 34.

The certification of a secondary batch (SB) is based on the comparison of a set of SB test pieces with a set of MB test pieces having a similar absorbed energy, using a single pendulum under repeatability conditions. The BCR reports [7] and [8] describing the SB certification approach, were published in 1991 and 1999, respectively. Since 2000, the calculation of the certified value and the estimation of its uncertainty have been updated to an approach compliant with the ISO Guide 98 (Guide to Expression of Uncertainty in Measurement [10]). This revised approach was developed and presented by Ingelbrecht *et al.* [11, 12].

3 Rationale for a Charpy reference material for tests at 0 °C

All Charpy V-notch certified reference test pieces previously released by IRMM were intended for tests at 20 °C ± 2 °C. This section explains why the FA013ay batch is intended for use at 0 °C ± 2 °C.

3.1 Jamming

It has been reported (see previous Master Batch certification report [13]) that for certain combinations of test piece and impact pendulum, broken sample halves are caught between the anvil and the pendulum hammer immediately after impact. This is called *jamming* and it results in the deformation of sample edges, the absorption of additional energy and anomalously high measurement results.

When testing samples with an elevated *KV* value, jamming does not occur because the sample is bent before it breaks. When it breaks, the two sample halves leave the pendulum in the direction of the hammer swing. When testing harder samples with a low *KV* value, the sample hardly bends before fracture, and the broken sample halves are not dragged through the anvils. Instead, they bounce backwards, away from the face of the anvils against which they were placed prior to testing. Jamming occurs exactly when the hardness and impact toughness of the tested samples are in the intermediate range, where some samples are dragged through the anvils, and others bounce back. This intermediate range is specific for the combination of test material (*KV*, hardness) and pendulum (hardness of anvils).

Jamming has been observed for some ERM-FA013 batches, for a few impact pendulums [13]. The corresponding outlier data points can be easily identified

by investigating the broken samples. Users of ERM-FA013 batches can and have to remove these data from their analysis.

3.2 Lower temperature tests to avoid jamming

A series of preliminary tests with ERM-FA013 samples indicated that lowering the test temperature from room temperature to 0 ºC results in a higher hardness and lower *KV* value, promoting the bounce-back fracture mode. To offer an alternative solution to the users of pendulums which show jamming on ERM-FA013 samples, IRMM decided to produce new batches of Charpy reference test pieces with the same steel as the previous ERM-FA013 batches, but for tests at lower temperatures.

Therefore, a new batch of test pieces was submitted to an interlaboratory characterisation exercise for tests at 0 ºC. The production and certification of this batch, ERM-FA013ay, are described in this report. ERM-FA013ay will be used as the Master Batch for certifying later batches of the ERM-FA013 series for tests at 0 ºC.

There is a risk that impact pendulums which do not show jamming effects with ERM-FA013 samples tested at room temperature, do show jamming effects with ERM-FA013 samples tested at 0 ºC. This risk was accepted, as the CRM needs for these impact pendulums will be satisfied by the continued production of ERM-FA013 batches for tests at room temperature.

4 Participants

4.1 Processing

- Cogne Acciai Speciali, Aosta (IT): production of steel bars
- Aubert & Duval, Gennevilliers (FR): heat treatment of steel bars
- Laboratoire National de Métrologie et d'Essais (LNE), Trappes (FR): processing of the V-notch test pieces

4.2 Characterisation

The following laboratories participated in the interlaboratory characterisation[1]:

Non-instrumented tests:

- Bodycote Materials Testing (now Exova), Emmen, Netherlands* (RvA testen L085)
- Bodycote Materials Testing (now Exova), Spijkenisse, Netherlands* (RvA testen L085)
- Bundesanstalt für Materialforschung und -prüfung (BAM), Abteilung V Werkstofftechnik, Berlin, Germany* (DAP-PL-2614.16)
- Centro de Apoio Tecnologico a Industria Metalomechanica (CATIM), Laboratório de Ensaios, Porto, Portugal* (IPAC L009)
- Fraunhofer-Institut für Werkstoffmechanik IWM, Freiburg, Germany

[1] Laboratories indicated with an asterisk performed the measurements within the scope of accreditation to ISO/IEC 17025 [14].

- European Commission Joint Research Centre (JRC), Institute for Reference Materials and Measurements, Geel, Belgium* (BELAC 268-Test)
- Laboratoire National de Métrologie et d'Essais, Charpy Laboratory, Trappes, France* (COFRAC SMH 2-1287)
- SCK-CEN, Labo Reactormaterialenonderzoek, Mol, Belgium* (BELAC 015-Test)
- SIRRIS, Beproevingslaboratorium Gent, Zwijnaarde, Belgium* (BELAC 232-Test)
- U.S. Steel Košice, Labortest, Košice, Slovakia* (SNAS 026/S012)
- Universität Stuttgart, Materialprüfungsanstalt, Stuttgart, Germany* (DAP-PL-2907.02)

Instrumented tests:
- Bundesanstalt für Materialforschung und -prüfung (BAM), Abteilung V Werkstofftechnik, Berlin, Germany* (DAP-PL-2614.16)
- SCK-CEN, Labo Reactormaterialenonderzoek, Mol, Belgium* (BELAC 015-Test)
- Fraunhofer-Institut für Werkstoffmechanik IWM, Freiburg, Germany

4.3 Evaluation and reporting

Evaluation of the raw data and reporting in a pre-defined format was performed by the laboratories participating in the characterisation tests. Further data evaluation and reporting was performed by IRMM.

5 Processing

The processing of the steel test pieces consisted of the following main steps:

1. Melting and casting of a steel ingot with appropriate composition, and subdivision of the ingot into a number of smaller billets.
2. Hot-rolling of the billets into long (4 to 7 m) bars of square cross-section (about 12 mm x 12 mm).
3. Heat treatment of the bars to obtain the appropriate steel microstructure.
4. Cutting of the bars into pieces, and machining of rectangular test pieces (55 mm x 10 mm x 10 mm).
5. Machining a V-notch in each sample.

5.1 From steel to hot-rolled bars

The base material for all ERM-FA013 batches is AISI 4340 steel. To limit the amount of impurities potentially affecting the homogeneity of the fracture resistance, the following compositional tolerances were imposed on the selected steel batch: Mn 0.7 – 0.8, Mo 0.23 - 0.28, Ni 1.7 – 1.85, P < 0.01, Si 0.2 – 0.35, S < 0.008 (all values in mass %), which is stricter than generally allowed for AISI 4340. After melting and casting, the steel ingots are subdivided, marked, and checked for microstructural homogeneity (inclusion content, grain size) and mechanical properties (Jominy hardenability test, absorbed energy *KV* as a function of annealing temperature).

The ingot was prepared and hot rolled at Cogne Acciai Speciali (Aosta, IT), resulting in bars that were 4 m long and with a squared cross-section of 11.5 mm. Steel was used from ingot number 960133, billet F. A full description of the processing and quality check of the steel bars is available in [15].

5.2 Heat treatment of hot-rolled bars

The heat treatment of the hot-rolled bars was performed at Aubert & Duval, Gennevilliers (FR), under the conditions indicated in Table 1.

Table 1: Heat treatment conditions

Batch	Number of bars	Austenisation		Annealing	
		T (°C)	Time (min)	T (°C)	Time (min)
ERM®-FA013ay	22	850	30	350	120

During the heat treatment, bars were placed onto rollers which slowly move the bars back and forth inside the furnace during the heat treatment to increase the homogeneity of the resulting microstructure. The first heat treatment was an austenisation treatment performed in a furnace of 'class 10 °C'[1]. From this furnace, the bars were quenched in oil at 40 °C. After the oil-quench, the samples were annealed in a second furnace ('class 5 °C'). After this annealing treatment, the samples were cooled down in air.

After heat treatment, a limited number of samples were machined for a preliminary check of the obtained energy level. Results obtained at Aubert & Duval indicated an average absorbed energy value of 21 J, close to the desired energy level (25 J).

5.3 Machining of Charpy test pieces

After the heat treatment, the samples were machined to dimensional tolerances imposed in ISO 148-3 [6]. In this production step, the major part of the microstructural gradient from sample surface to sample core is removed. The batch code ('AY 30', with '30' indicating the nominal absorbed energy level at room temperature (30 J) and 'AY' the letter code assigned consecutively to batches of the same nominal absorbed energy) and an individual sample code (e.g. C047, with 'C' indicating the bar from which the sample was cut and '047' the position of the sample in the bar) were engraved on the long face of the sample that is facing up when the sample is positioned for testing. Batch and sample code are engraved twice on each sample, once on both sides of the notch, which provides easier identification of the broken samples after the test.

[1] In a furnace of 'class x °C', the variation of the temperature is smaller than x °C. The furnaces used have 10 heating zones. Each zone has 3 controlling thermocouples and 3 measurement thermocouples. These are regularly calibrated. When one faulty thermocouple is detected, it is replaced by a thermocouple produced with wire from the same roll. When a roll is exhausted, all thermocouples are replaced with new ones.

The V-notch was introduced using electric discharge machining. Since the notch is 2 mm deep, its tip is well below the surface layer, the properties of which might be affected to some extent by the near-surface gradient in microstructure resulting from the successive heat treatments.

Both machining and notching operations are performed in accordance with strict and controlled procedures.

5.4 Quality control

When all samples from the batch were fully machined, a randomised selection of 25 samples was made. The dimensions of the 25 samples were checked on July 18, 2007 against the criteria specified in EN 10045-2 [1]: length $55.0\ ^{+0.0}_{-0.25}$ mm, height (10.00 ± 0.06) mm, width (10.00 ± 0.075) mm, notch angle (45 ± 1) °, height remaining at notch root (8.00 ± 0.06) mm, radius at notch root (0.25 ± 0.025) mm, distance between the plane of symmetry of the notch and the longitudinal axis of the test piece (27.50 ± 0.10) mm. All samples met all requirements, with the exception of one measure of the notch angle on 1 sample (D008). None of the other samples was near the limit of the allowed range for this dimension. Also, when broken, this sample had a *KV* value (28.8 J) close to the average *KV* (28.3 J, *SD* = 0.7 J), indicating that the deviation did not affect the measured *KV* value.

The samples checked for geometrical compliance were impact tested on August 2, 2007 on the Tinius Olsen 350 Joules pendulum - which is one of the French reference pendulums - at LNE. The results are reported in certificate LNE No. F031180/CQPE/1 [16]. The average *KV* of the 25 samples, at room temperature, was 28.3 J, sufficiently close to the target value (30 J). The standard deviation of the test results (*SD* = 0.7 J, *RSD* = 2.5 %) was smaller than the maximum level of 5 % allowed by ISO 148-3 [6]. The sample-to-sample homogeneity was checked again during the characterisation tests (see Section 6).

5.5 Packaging and storage

The samples were packed in oil-filled and closed plastic bags in sets of 5. The samples were closely packed in the bag to eliminate the possibility that corners or edges of one bar scratch the other bars. The oil-filled bags, together with a label, were packed in a sealed plastic bag, and shipped to IRMM. The 253 sets of ERM-FA013ay samples (delivery August, 2007) were registered and stored at room temperature.

6 Characterisation

6.1 Laboratory selection

Characterisation of this candidate master batch was carried out in an interlaboratory comparison between a statistically representative set of 12 pendulum impact testing machines (from 11 different laboratories). 6 of the pendulum hammers were of the U-type, and 6 of the C-type. 3 laboratories performed instrumented Charpy tests, the results of which can provide

additional information to better understand the results of the non-instrumented tests.

The laboratory selection was a multi-step process. First a list of laboratories with sound and demonstrated expertise in the field of 'mechanical testing' was put together based on an open call for interest. Laboratories were selected on the basis of a combination of quality management and technical criteria. All laboratories have a quality system, and most of them are accredited. Results of proficiency testing schemes or other published interlaboratory comparison data were used to assess the technical quality of the laboratories. In summer 2008, all laboratories qualified for the 'mechanical testing' field at that time, were invited to submit a tender for the execution of Charpy impact tests in accordance with EN 10045-2 [1] and ISO 148-2 [2], and in compliance with the quality criteria imposed by ISO/IEC 17025 [14]. A further selection was then made based on an evaluation of these tenders in terms of cost, and in terms of technical criteria specific for Charpy impact tests, such as the control over anvil spacing and temperature, the results from direct verification tests control (hammer tip and anvil radii) and results from tests on reference materials.

Details of the 12 pendulums used in this study are given in Annex 1. All selected pendulums are regularly verified with instruments and tools that are traceable to the respective national standards. This is essential, because it implies that the measured values, as well as the resulting certified value of the master batches, will be traceable to the SI.

The interlaboratory comparison exercise was performed between November 2008 and March 2009.

6.2 Test protocol

Each laboratory tested 20 samples of the ERM-FA013ay batch, corresponding to 4 sets of 5 samples randomly selected from the whole batch. A strict test protocol was imposed, referring to the ISO 148 and EN 10045 series of standards [1, 2, 3, 4, 6], and additionally imposing a randomised order of the tests, distributed over two test days (10 samples on day 1, 10 samples on day 2). All tests were performed at nominally 0 °C. All laboratories respected the tolerance of ± 2 °C, but most laboratories provided a sample temperature control better than ± 1 °C, as this was one of the tender award criteria. The measured absorbed energy values were corrected for friction and windage losses.

For quality control purposes, the test protocol also included the testing at 0 °C of 10 samples (5 samples on day 1 and 5 samples on day 2) of a previous CRM (FA013ba). Therefore, on both testing days, 15 samples had to be tested. The order of testing the samples was fully randomised, mixing the different batches. The average values of the FA013ba and FA013ay batches could be expected to be different, due to slight differences in the material's microstructure, even if both batches are nominally at the same energy level.

The laboratories performing instrumented impact tests were requested to follow the testing and reporting procedures described in ISO 14556:2000 [17]. The test schedule was the same as for the non-instrumented tests. Actually, all three laboratories obtained their instrumented and non-instrumented data simultaneously, on the same samples.

6.3 Data analysis

6.3.1 Screening and elimination of individual data points

The reporting laboratories eliminated a limited number of data points for technical reasons observed during or immediately after testing, e.g, because a sample was not well centred, as revealed by the position of the anvil marks.

IRMM collected all broken samples and inspected all sample halves for marks that can indicate technical problems. After testing, all Charpy samples show 'first-strike' marks: these are the marks caused by the interaction between sample, hammer tup and anvils during the first and intended hammer impact. Upon fracture, the broken samples halves lose contact with hammer and anvils and follow one of a variety of possible trajectories, depending on the properties of both pendulum and test material. Some samples show 'second-strike' marks. These are marks caused by a second impact of the already broken samples halves back onto the anvils. This phenomenon has been described by Schmieder et al. [18]. All broken samples of the ERM-FA013ay and –ba batches show second-strike marks, for all of the pendulums used. A second impact of the broken half samples onto the anvils does not affect the measured *KV* value, since it does not slow down the swinging pendulum.

Of the 120 tested FA013ba samples, 9 samples show possible indications of the previously discussed 'jamming' effect (see Section 3.1). These samples were tested on 4 out of the 12 participating pendulums (lab 2 (1 sample), lab 3 (3 samples), lab 9 (2 samples) and lab 11 (3 samples)). The corresponding data points are eliminated from the analysis, also if they did not correspond with a statistical outlier value.

Also 6 of the 240 tested FA013ay samples show possible indications of the 'jamming' effect, and the corresponding data points were eliminated from the analysis. Interestingly, all 6 samples were tested on the same pendulum (lab 3). For this lab 3, no jamming effects were observed when testing batches FA013ba and FA013at at room temperature. This is a good indication that most laboratories will in future be able to avoid the jamming of ERM-FA013 samples by choosing either an FA013 batch for tests at room temperature or an FA013 batch for tests at 0 °C.

After the inspection of the broken samples, and the elimination of results corresponding to samples that show traces of the jamming effect, a statistical outlier test was performed on the remaining data for each lab. 1 outlier value (99 % confidence level), and no strangler values (95 % confidence level) were detected. The outlier value (sample FA013ay, Q037, lab 11) could easily be related to an incorrect (non-symmetric) positioning of the sample on the

anvils, via inspection of the first-strike marks. The data-point was eliminated from the analysis.

6.3.2 Qualification of laboratories using quality control samples

Results obtained on samples of the previously certified batch ERM-FA013ba were used for an additional qualification of the participating laboratories (additional to the laboratory selection described in section 6.1). Average values obtained at the different laboratories on the CRM (10 samples/lab) are shown in Figure 4. Individual data are shown in Annex 2.

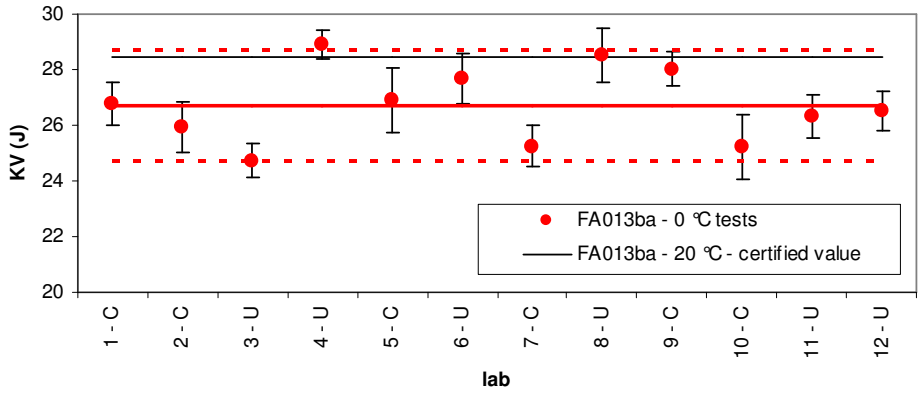

Figure 3: Mean KV values obtained at the participating laboratories on the quality control samples (batch ERM®-FA013ba; 10 samples/lab). Thin straight line: certified value of FA013ba at 20 °C; bold straight line: average value of FA013ba tests at 0 °C; dotted lines: corresponding limits for reference pendulums (± 2 J; see ISO 148-3 [6]); error bars indicate average value ± 1 SD; the symbols 'U' and 'C' indicate the type of hammer.

The laboratory qualification is based on a bias criterium taken from ISO 148-3 [6]. For tests at energy levels < 40 J, the difference between the average value obtained on a CRM shall not deviate from the certified value by more than 2 J. The certified *KV* value of the ERM-FA013ba batch is only valid for tests at 20 °C ± 2 °C. Therefore, the values measured by the individual laboratories were compared against the interlaboratory mean value. The resulting upper and lower limits (mean *KV* ± 2 J) are indicated in Fig. 3.

The mean values from labs 3 and 4 are just outside the 2 J range, but the data are not eliminated, as the deviation from the certified value is within the allowed range after rounding. Additional arguments for keeping the data is the fact that the interlaboratory mean value does not have the same status as a certified value, thereby increasing the uncertainty associated with the used bias criterium, and the symmetric position of both outliers, meaning that, combined, they have little effect on the position of the later certified *KV* value.

One notes that the interlaboratory mean value (26.7 J) is about 2 J lower than the certified value for room temperature tests (28.46 J, also indicated in

Figure 3). This shows that the reduction in test temperature has reduced the material's impact toughness in a significant manner, as intended.

6.3.3 Evaluation of the accepted results

Average values obtained by the different laboratories on batch ERM-FA013ay (20 samples/lab) are shown in Figure 4. Individual data are shown in Annex 2.

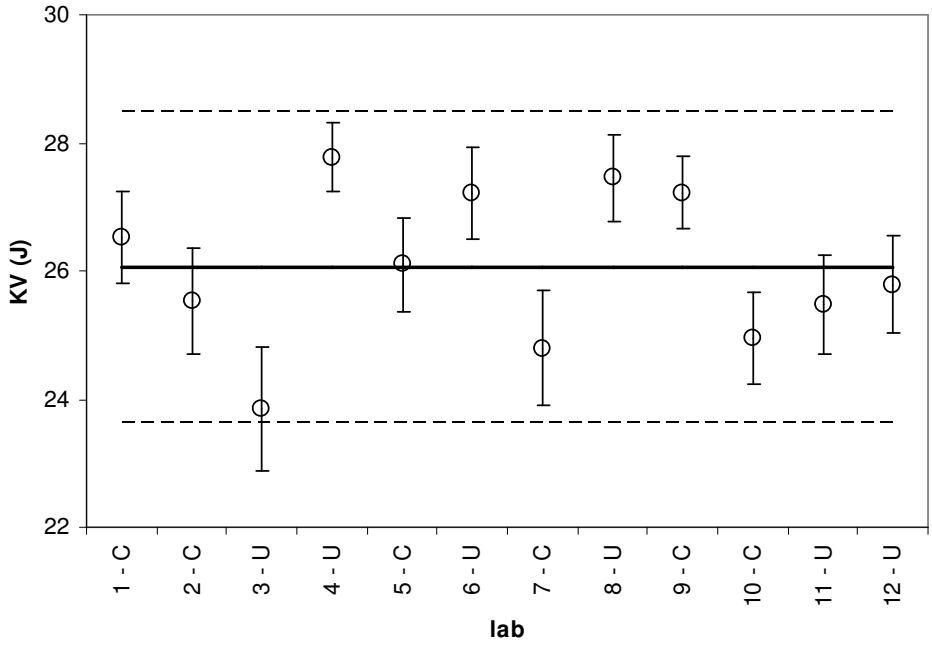

Figure 4: Laboratory mean KV values (20 test pieces/lab) for tests on ERM®-FA013ay at 0 °C; full line: mean of laboratory mean values; dashed lines: 95 % confidence interval of the distribution of the laboratory mean values; error bars indicate the laboratory mean value ± 1 SD.

The distribution of the FA013ay data was judged from a histogram and a normality plot, which show a normal distribution. Earlier observations of a systematic difference between the results obtained with U-type and the C-type hammers are not confirmed [13]. The analysis of the data therefore is done by pooling the results obtained from all 12 pendulums, and the numerical results are summarised in Table 2, which gives the mean of the accepted mean values (KV_{char}), the number of accepted data sets (p), the standard deviation between the accepted mean values (s_{char}), and u_{char}, the resulting uncertainty of KV_{char}. The latter is calculated as $u_{char} = \frac{s_{char}}{\sqrt{p}}$. These are the values that are required when later using the master batches in the certification of secondary batches.

Table 2: Summary of the analysis of the results of the ERM®-FA013ay characterisation measurements: average value (mean of laboratory means) KV_{char}; number of labs contributing accepted data; standard deviation of laboratory mean values; and the resulting uncertainty u_{char}

	Average value KV_{char} [J]	**Number of labs** p [J]	**Standard deviation** s_{char} [J]	**Uncertainty of** KV_{char} u_{char} [J]
ERM®-FA013ay	26.06	12	1.21	0.35

6.3.4 Analysis of data from instrumented impact testing

The three laboratories which performed instrumented impact tests all reported force-displacement curves with a characteristic oscillation pattern during the loading stage. Figure 5 shows a curve for one sample, with a shape that is representative for samples of both of the 2 batches tested.

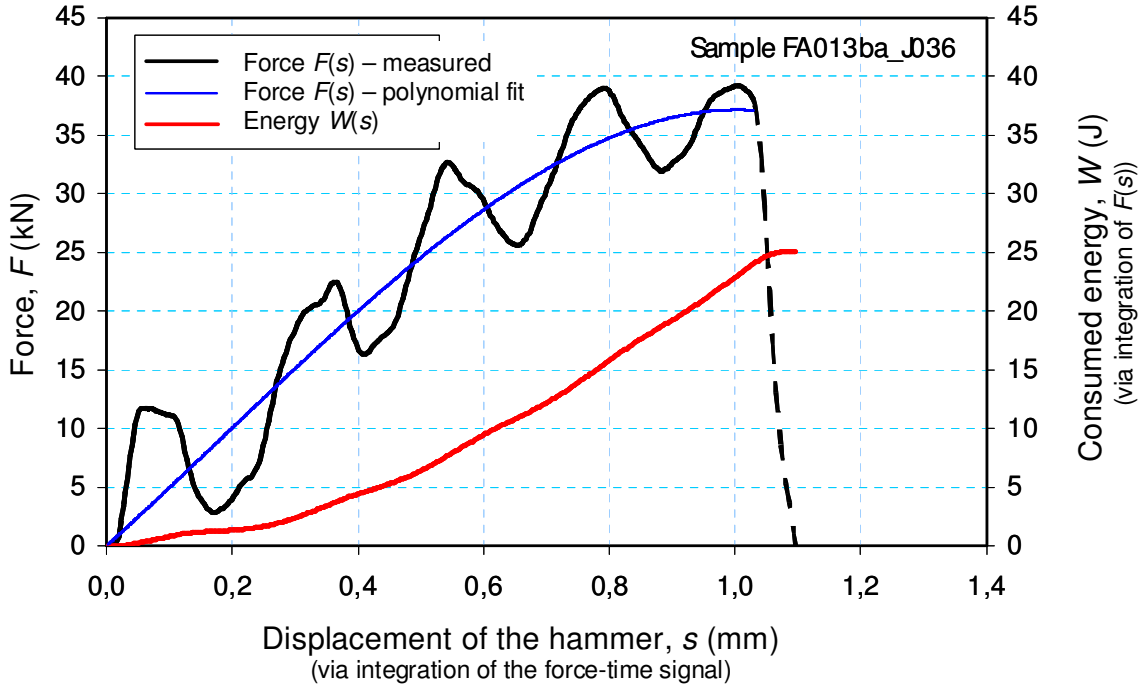

Figure 5: Results of an instrumented Charpy-V test according to ISO 14556: the measured force F with a polynomial fit, and the consumed energy W (calculated via the integration of the force-displacement signal) versus the displacement s of the impacting hammer (calculated via integration of the force-time signal) [image: courtesy W. Böhme, M. Hug, Fraunhofer IWM, Freiburg].

ISO 14556:2000 [17] provides a procedure for determining characteristic values of force, displacement and energy. In particular, the total impact energy (W_t) values are fairly robust. This is confirmed by the reported data:

the three laboratories report compatible instrumented and non-instrumented results (Table 3), as these results overlap within their standard deviation. Therefore, the W_t values were further analysed.

The average results of the instrumented and the non-instrumented test results submitted by the three laboratories were compared. The average of the instrumented values matches with the average of the non-instrumented values (overlapping standard deviations). Moreover, the standard deviation of the laboratory means is smaller for the instrumented results. This indicates that:

- results obtained with instrumented impact machines can be combined with the results of non-instrumented impact machines.

- including more instrumented impact pendulums in an interlaboratory comparison for the certification of CRMs, is actually favourable given the apparent smaller between-instrument scatter.

Both conclusions will be considered for the design of future certification exercises.

Table 3: Summary of instrumented impact test results.

Lab Code	**FA013ay**		**FA013ba**	
	non-instrumented ($KV \pm SD$)	instrumented ($W_t \pm SD$)	non-instrumented ($KV \pm SD$)	instrumented ($W_t \pm SD$)
	J	J	J	J
2	25.53 ± 0.82	26.65 ± 0.70	25.94 ± 0.92	26.17 ± 0.97
5	26.10 ± 0.74	26.57 ± 0.84	26.89 ± 1.18	27.51 ± 1.03
9	27.22 ± 0.57	26.30 ± 0.49	28.03 ± 0.64	27.01 ± 0.47
Average over labs 2, 5, 9	**26.28 ± 0.86**	**26.51 ± 0.18**	**26.95 ± 1.05**	**26.90 ± 0.68**

7 Homogeneity

7.1 Check of the maximum homogeneity allowed by EN 10045/ISO 148

The homogeneity of each batch of certified reference test pieces for Charpy impact tests needs to meet a criterion imposed by ISO 148-3 [6]. The criterion is expressed as the relative between-sample standard deviation of *KV* data as obtained on a reference pendulum, and the highest accepted value is 5 %. The homogeneity of the ERM-FA013ay batch at room temperature was tested both at IRMM (November 6, 2007; *RSD* = 2.4 %) and at LNE (August 2, 2007; *RSD* = 2.5 %). Both results confirm the suitability of the ERM-FA013ay batch.

However, the tests at room temperature will not necessarily reveal the same heterogeneity as tests at 0 °C, the test temperature for which the *KV* value of ERM-FA013ay is to be certified. That is why in the following sections the contribution of heterogeneity to the uncertainty of the certified value of ERM-FA013ay will be assessed using results obtained during the interlaboratory characterisation study, where tests were performed at 0 °C.

7.2 How does homogeneity contribute to the uncertainty of KV_{MB}?

For secondary batches of Charpy test pieces, the certified value and uncertainty pertain to the average *KV* of 5 samples. Therefore, the contribution of the measured heterogeneity to the uncertainty of the certified value is calculated as $u_{hom} = \frac{SD}{\sqrt{5}}$. ERM-FA013ay is not a secondary batch but a Master Batch, and the certified value of ERM-FA013ay pertains to the average of the whole batch. This certified value is determined as the mean of the laboratory mean values collected in an interlaboratory study. Since 20 samples were tested per lab, the homogeneity contribution to the uncertainty of each of the laboratory mean values can be deduced from *s*, the within-laboratory standard deviation, as $s_{hom} = \frac{SD}{\sqrt{20}}$. The effect of s_{hom} on the uncertainty of KV_{MB} depends on the number of participating laboratories, *p*, ($u_{hom} = \frac{s_{hom}}{\sqrt{p-1}}$).

7.3 Results

Each laboratory tested 20 samples selected from the batch of about 1250 samples. Samples were randomly selected from all bars constituting the batch, and from all positions along the bars. Therefore, the standard deviations per laboratory can be considered as statistically valid estimates of the standard deviation over the whole batch.

The observed standard deviation values, *s*, vary between laboratories, mainly because the repeatability of measurements varies between laboratories. For all accepted data sets (evaluated per laboratory) the observed relative standard deviation is better than the required 5 %. (The largest value is 4.01 %.) The average within-laboratory standard deviation, as deduced from a single-factor ANOVA analysis is 0.74 J or 2.8 %, the resulting s_{hom} is 0.17 J, and the value of u_{hom} is 0.05 J.

Table 4: Summary of homogeneity assessment results

Batch Code	**Observed within-laboratory relative standard deviations**			s_{hom}	u_{hom}
	lowest	**highest**	**average (from ANOVA)**		
	%	**%**	**%**	**J**	**J**
ERM®-FA013ay	1.93	4.01	2.84	0.17	0.05

7.4 Conclusion

The s_{hom} value shown in Table 4 is 7 times smaller than s_{char}, the standard deviation of the laboratory mean values (see Table 2). The same is true for the corresponding uncertainty values, u_{hom} and u_{char}. This proves that the differences observed between laboratory mean values are not due to inhomogeneity of the samples, but due to genuine differences in the performance of the different pendulums and in the way they are operated. It can be concluded that the homogeneity contribution to the certified value of a Master Batch is negligibly small, in contrast with the homogeneity contribution to the uncertainty of a Secondary Batch. It must be noted here that the sample-to-sample heterogeneity of the Master Batch samples will be added in the uncertainty budget of the certified value of the secondary batches.

8 Stability

Microstructural stability of the certified reference test pieces is obtained by the annealing treatment to which the samples were subjected after the austenisation treatment. Annealing is performed at temperatures where the equilibrium phases are the same as the (meta-)stable phases at ambient temperature (α-Fe and Fe_3C). The only driving force for instability stems from the difference in solubility of interstitial elements in the α-Fe matrix, between annealing and ambient temperature. Relaxation of residual (micro-)stresses by short-range diffusion or the additional formation or growth of precipitates during the shelf-life of the certified reference test pieces is expected to proceed but slowly.

Given the sample-to-sample heterogeneity of about 3 % (see Table 4), the ageing effects are difficult to detect when testing limited numbers of samples. Dedicated efforts have been spent to quantify the stability of the certified values of batches of Charpy CRMs. The first systematic investigation was performed for samples of nominally 120 J by Pauwels *et al.*, who did not observe measurable changes of absorbed energy over a period of 1.5 years, even with exposure to 90 ^{o}C [20]. New evidence for the stability of the reference test pieces produced from AISI 4340 steel of other energy levels (nominally 15 J, 30 J and 100 J) has been obtained during the International master batch (IMB) project [21]. In the IMB-project, the stability of the certified test pieces is confirmed by the unchanged value of the mean of means of the absorbed energy obtained on 7 reference pendulums over a three year period.

Taking into account the above, the uncertainty contribution from instability is considered to be negligible compared to the contribution of between-laboratory variation to the uncertainty of the certified value. Nevertheless, until further notice, it is decided to specify a limited shelf-life for the new master batches. A period of 10 years is chosen, counting from the date of the characterisation tests. Since the materials were characterised between November 2008 and March 2009, the validity of the certificate stretches until November, 2018.

The proposed shelf-life may be extended as further evidence of stability becomes available. In this respect, a dedicated, isochronous post-certification monitoring test has been initiated on the FA013ba batch in March 2008 (reference temperature -20 °C, time-points 12, 24, 36 and 48 months). Results will be available early 2012.

9 Uncertainty from sample temperature

9.1 Introduction

The laboratory data and the resulting certified values are reported for a sample temperature of 0 °C. The test procedure includes a transfer of the sample from a cooling bath to the pendulum which is operated at the higher laboratory temperature. At first sight, one expects that the sample temperature will be higher than 0 °C at the actual time of measurement. The resulting temperature uncertainty contributes to the overall uncertainty of the *KV* values, because there is a strong relation between temperature and impact toughness. To limit this effect, ISO 148-1 [4] prescribes a maximum time of 5 s between taking the sample out of the cooling bath and the impact. In this section, the remaining uncertainty is assessed, investigating both parts of the issue:
1) How big is the change of the sample temperature between the moment the sample leaves the cooling medium and the moment the sample is broken?
2) How big is the effect of this temperature change on the measured *KV* values?

9.2 Assessment of the possible temperature change

The change of the sample temperature depends on a number of physical phenomena: radiation, conduction (from the supported specimen ends to the supports and anvils; from the notch tip to the specimen ends), evaporation (in case of a liquid cooling medium), convection, … Even if some of these contributions may be negligible (e.g., loss by heat radiaton is small at 0 °C), it is hard to make a reliable calculation of the change of temperature along the notch tip with time.

Instead, Nanstad et al. [22] have performed a large number of experiments, using thermocouples buried in Charpy test samples to measure the change of temperature in a sample taken out of a thermal conditioning medium. Tests were performed between -100 °C and 100 °C, for different media (nitrogen gas, air, acetone, methanol, oil and water), on a low-alloy steel (which implies a thermal conductivity that is similar to that of the low-alloy steel used to make ERM-FA013 samples). Nanstad et al. [22] showed that for none of the media used in our study, sample temperature changes more than 0.5 °C within the allowed 5 s, if the temperature difference between cooling medium and laboratory temperature is less than 25 °C. Larger temperature changes were measured due to evaporative cooling, when taking samples out of a liquid that is near its boiling point. When using such liquid as a cooling medium, the evaporative cooling effect would actually counter the natural heating from the cooling medium temperature to the laboratory temperature.

In the tests on FA013ay, none of the participating laboratories used a liquid cooling medium that was near its boiling point. The results shown in Figure 6 confirm the limited effect of evaporative cooling the particular cooling medium used (water, petrol, ethanol, acetone): the *KV* values of laboratory 12, using the cooling medium with the lowest boiling point (acetone, boiling at 56 °C), are in perfect agreement with the mean of laboratory averages. Figure 6 also shows that there is no significant difference between the tests performed on samples cooled in a gaseous or a liquid cooling medium.

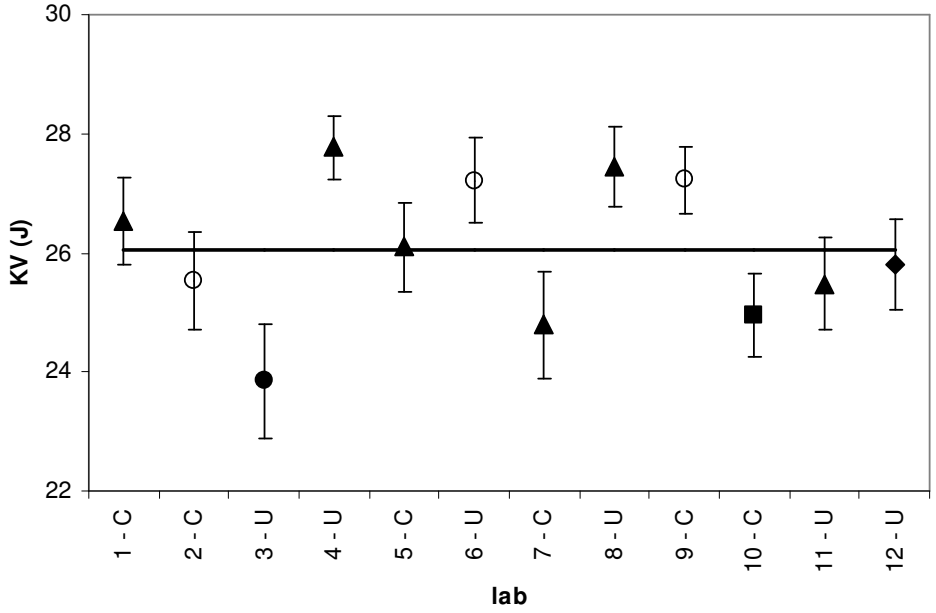

Figure 6 Distribution of laboratory average values (error bars: within-laboratory standard deviation) with indication of the applied cooling medium: open symbols: gas (air or air/nitrogen), full symbols: liquid (▲: ethanol or alcohol; ●: petrol; ■: water; ♦: acetone).

9.3 Assessment of the effect of temperature change on measured KV

The effect of temperature on measured *KV* is exactly the reason for having chosen 0 °C as the test temperature for ERM-FA013ay (see section 3.2). It was already reported (see section 6.3.2) that the decrease of the test temperature from 20 °C to 0 °C results in a decrease of the ERM-FA013ba *KV* value of 1.8 J. Using a linear approximation, the conclusion is that *KV* changes at about 0.09 J/°C.

The change of *KV* with temperature was also investigated between 0 °C and -38 °C for the ERM-FA013ay batch itself [23]. The results (Figure 7) indicate an average decrease of about 0.09 J/°C, which is in perfect agreement with the results obtained on the ERM-FA013ba batch.

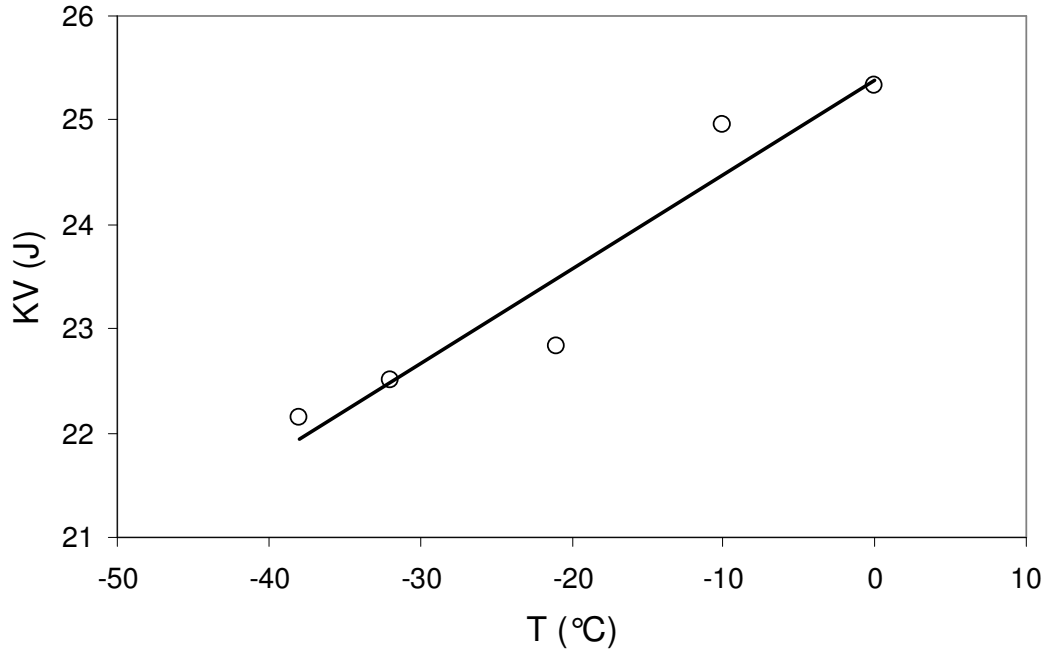

Figure 7 Change of KV value with temperature for ERM-FA013ay.

9.4 Calculation of the uncertainty contribution due to possible changes in sample temperature

Combining the findings of the above sections, one concludes that the possible effect of the change in sample temperature between the moment of leaving the cooling bath and the moment of test, is about 0.5 °C × 0.09 J/°C = 0.045 J. Because of the many uncertainties associated with this estimation, the certified value will not be corrected with this value. Instead, the value will be used as a type-B standard uncertainty contribution, with an infinite number of degrees of freedom, u_T, in the calculation of the combined uncertainty of the certified value.

10 Evaluation of results

10.1 Calculation of certified value, combined and expanded uncertainty

ISO Guide 35 [24] provides a generic, ISO Guide 98 [10] compliant uncertainty model for use in the certification of batches of CRMs. In Charpy terms, the model can be expressed as follows:

$$KV_{MB} = KV_{char} + \delta KV_{hom} + \delta KV_{lts} + \delta KV_{sts} \qquad \text{Eq. 1}$$

with KV_{char} the KV value obtained from the characterisation of the batch, δKV_{hom} an error term due to variation between samples, δKV_{lts} and δKV_{sts} error terms due to the long-term and short-term instability of the RM. Homogeneity and stability studies are designed in such a way that the values of the corresponding error terms are zero. However, the uncertainties of the error terms are not (always) zero. Assuming independence of the variables,

and adding the uncertainty contribution due to sample temperature changes, the uncertainty of the certified value of the Charpy CRM can therefore be expressed as:

$$u_{MB} = \sqrt{u_{char}^2 + u_{hom}^2 + u_{lts}^2 + u_{sts}^2 + u_T^2} \qquad \text{Eq. 2}$$

Table 2 summarised the results of the characterisation tests. The preceding paragraphs have explained why the homogeneity and stability contributions are insignificant. The certified uncertainty therefore is a combination of u_{char} and u_T. The effective number of degrees of freedom of the uncertainty value u_{char} is directly calculated as (p-1), with p the number of accepted data sets. Due to the small value of u_T, and the infinite number of degrees of freedom associated with u_T, the number of degrees of freedom of u_{MB} (ν_{eff}) is the same as that of u_{char}: $\nu_{char} = \nu_{eff} = 11$.

The uncertainty reported on the certificate is the standard uncertainty, with a confidence level of about 68 %, since this is the value that will need to be combined later, during the certification of secondary batches, with other standard uncertainty contributions. (It is noted that the relevant number of degrees of freedom is sufficiently large ($\nu_{eff} = 11$) to justify the use of a coverage factor $k = 2$ to expand the confidence level to about 95 %.)

10.2 Metrological traceability

The absorbed energy *KV* is a method-specific quantity, and can only be obtained by following the procedures specified in EN 10045-1 [3] and ISO 148-1 [4]. The certified values of the new master batches certified in this study are defined by these standard procedures as they were obtained using an interlaboratory comparison, involving a representative selection of qualified laboratories performing the tests in accordance with the standard procedures.

The certified values of the new master batches certified in this study are traceable to the SI, since the results were obtained on pendulums operated in an ISO/IEC 17025 [14] compliant system, involving regular direct verification of the pendulums using tools that are calibrated in an SI-traceable manner.

10.3 Commutability

The commutability issue concerns both the choice of material as well as the method chosen to characterise the reference material.

During this certification study, 12 different pendulums were used, each equipped with an ISO-type striker of 2 mm striker edge radius [4]. The reference materials are commutable if tested with 2 mm strikers, and when following the EN and ISO standard test procedures [1, 4]. The certified values are not to be used when the test pieces are broken with an ASTM-type striker of 8 mm striker edge radius.

The steel chosen is of an industrial type, combining hardness and absorbed energy properties that impose forces on the pendulum that cover the same range of forces as met in routine use. The reference material is therefore

bound to trigger the same potential instrumental problems as those that are experienced in practice. This guarantees the commutability of the reference material.

10.4 Summary of results

The certified value, associated uncertainty and the effective degrees of freedom are summarised in Table 5.

Table 5: Certified values and associated uncertainties

	Certified mean value KV_{MB} **[J]**	**Standard uncertainty** u_{MB} **[J]**	**Degrees of freedom** ν_{eff}
ERM®-FA013ay	26.06	0.35	11

10.5 A posteriori comparison of laboratory mean values and KV_{MB}

Figure 8 shows the results of the contributing laboratories, with their expanded measurement uncertainty. The uncertainties of the laboratory mean values were calculated at IRMM, based on measurement results reported by the labs, using an uncertainty budget proposed earlier [19]. This uncertainty budget is composed of 4 main contributions[1]: bias of the pendulum as deduced from an indirect verification (here: results are taken from the recent FA013ba certification exercise), homogeneity of the test material (characterised by the within laboratory standard deviation), uncertainty of the certified value of the reference material used in the indirect verification (taken from the certificate), and instrument resolution.

[1] This uncertainty approach has recently been implemented by ISO TC 164 and is described in informative annexes to the ISO 148-1, ISO 148-2 and ISO 148-3 standards [2, 4, 6].

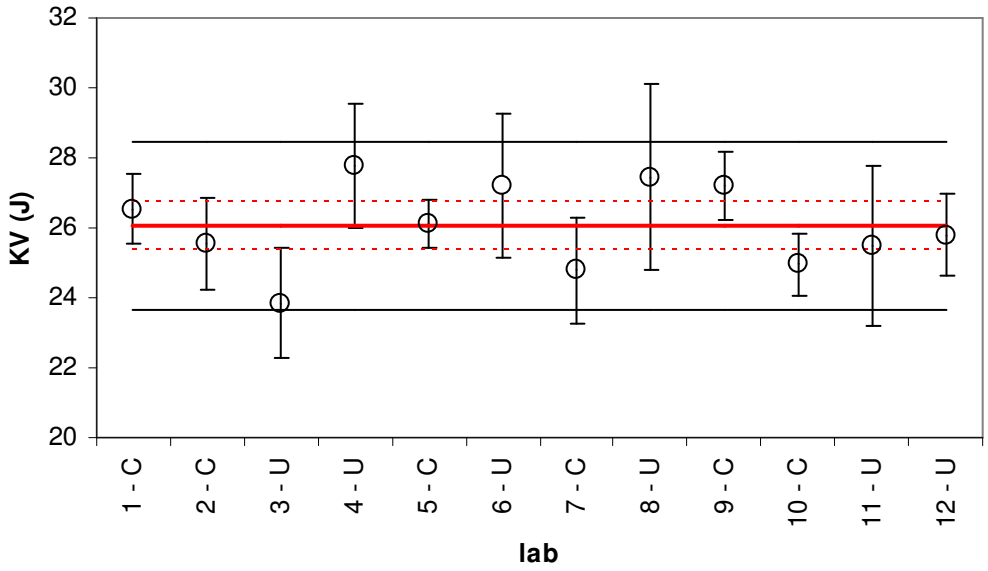

Figure 8: Graph showing the mean of laboratory means (full line, bold) and its expanded uncertainty (dotted lines) and the 95 % confidence interval of the population (full lines, thin), in comparison with the results of the contributing laboratories (open symbols) with indication of the expanded measurement uncertainty of the individual laboratories.

Figure 8 shows that, as expected for a normal distribution, all laboratory mean values fall within the 95 % confidence interval. With the exception of laboratory 3, all 12 laboratory mean values agree with KV_{char} within the respective, combined u_{char} and lab uncertainties. This indicates that the uncertainty budget proposed and used by IRMM provides realistic measurement uncertainty values.

11 Instructions for use

11.1 Intended use

Samples of ERM®-FA013ay correspond to the '(certified) BCR test pieces' as referred to in EN 10045-2 [1], as well as to the 'certified reference test pieces' as defined in ISO 148-3 [6]. In particular, the samples of this batch are intended for use by IRMM in the certification of secondary batches of certified reference test pieces for the indirect verification of impact testing machines with a striker of 2 mm striker edge radius according to procedures described in detail in EN 10045-2 [1] and ISO 148-2 [2].

11.2 Sample preparation

Special attention is drawn to the cleaning and conditioning of the specimens prior to testing. It is mandatory to remove the oil from the sample surface prior to testing, without damaging the edges of the sample. Between the moment of removing the protective oil layer and the actual test, corrosion can occur. This

must be avoided by limiting this period of time, while keeping the sample clean.

The following procedure is considered good practice.

1. First use absorbent cleaning-tissue to remove the excess oil. Pay particular attention to the notch of the sample, but do not use hard (e.g. steel) brushes to remove the oil from the notch.
2. Submerge the samples in ethanol for about 5 minutes. Use of ultrasonication is encouraged, but only if the edges of the samples are prevented from rubbing against each other. To reduce the consumption of solvent, it is allowed to make a first cleaning step with detergent, immediately prior to the solvent step.
3. Once the samples are removed from the solvent, only manipulate the samples wearing clean gloves. This is to prevent development of corrosion between the time of cleaning and the actual test.
4. Before testing, bring the specimens to the test temperature (0 ± 2 °C). To assure thermal equilibrium, the specimens shall be at least 10 minutes in a liquid cooling medium or at least 30 minutes in a gaseous cooling medium, the temperature of which is measured and monitored. The test piece shall be broken within 5 seconds of the time of removal from the cooling medium.

11.3 Pendulum impact tests

After cleaning, the samples need to be broken with a pendulum impact test machine in accordance with EN 10045-2 [1] or ISO 148-2 [2] standards. Prior to the tests, the anvils must be cleaned. It must be noted that Charpy test pieces sometimes leave debris on the Charpy pendulum anvils. Therefore, the anvils must be checked regularly and if debris is found, it must be removed.

For some pendulums and for some samples, post-fracture interaction between broken samples and pendulum hammer can affect the measured KV values. The resulting excessively high values can be due to indentations and deformations of the broken samples. Outlier values that can be related to post-fracture indentation marks on the broken samples must be eliminated from the analysis of the results.

Acknowledgements

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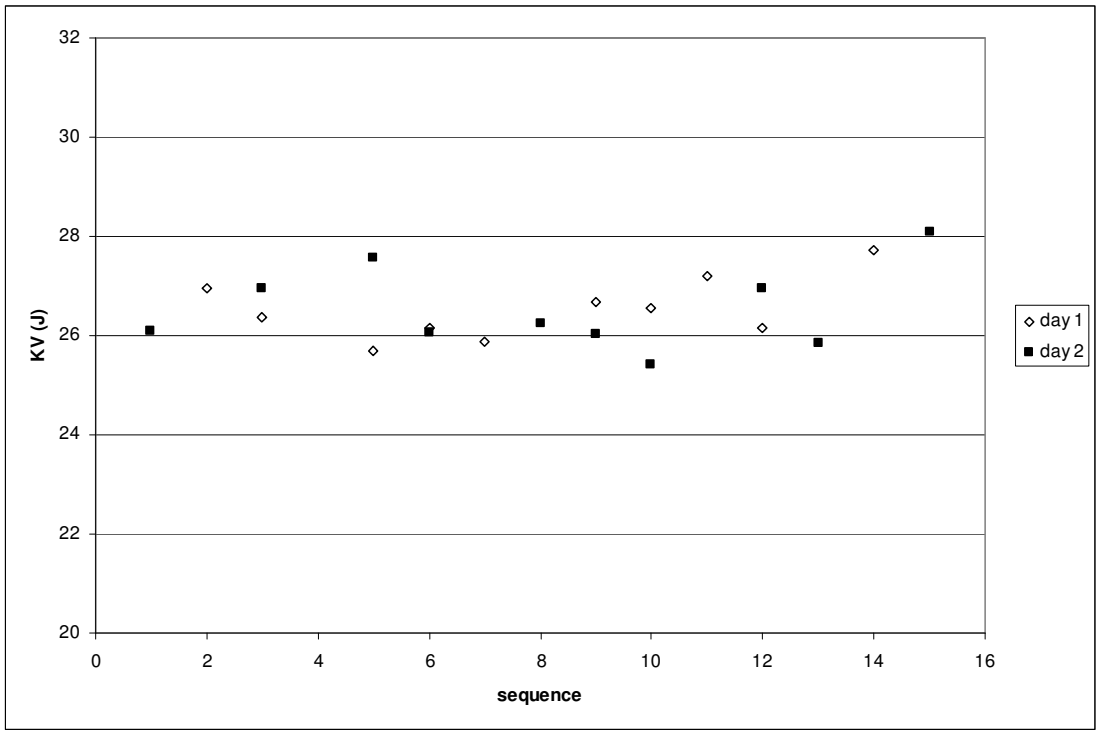
Annex 1: Details of pendulums in characterisation laboratories

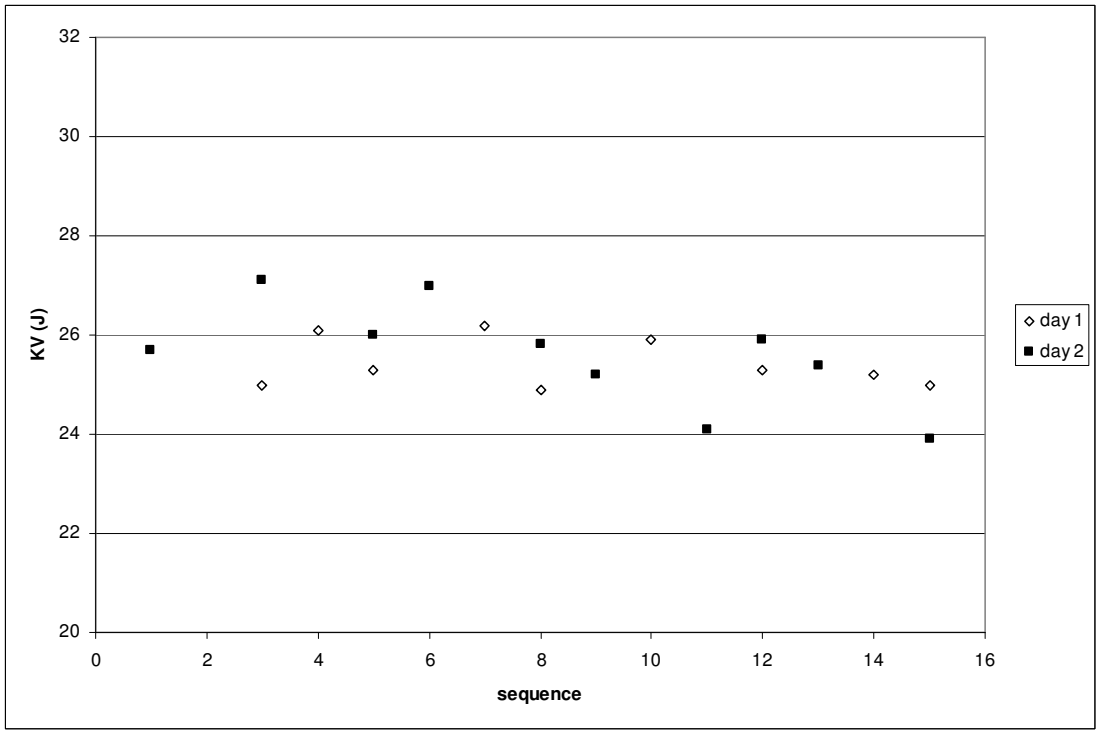
Lab code	Constructor / type	Hammer type	Nominal energy	Pendulum moment
			J	Nm
1	Wolpert PW30E	C	300	152.8
2	Wolpert PW30	C	300	154.1
3	Roell Amsler RK450	U	300	160.4
4	Tinius Olsen 74 Impact	U	359	239.5
5	Mohr & Federhaff PSW 30/15	C	300	152.3
6	Roell & Korthaus Amsler RKP300	U	302	161.8
7	MFL PSW300	C	300	154.1
8	Zwick / Roell RKP450	U	450	241.3
9	Toni-MFL PSW300	C	300	153.3
10	Instron Wolpert PW-30	C	300	156.2
11	Amsler Otto Wolpert PW 30/15	U	300	153.9
12	Zwick Roell RKP450A	U	300	160.2

Annex 2: Individual data of characterisation laboratories

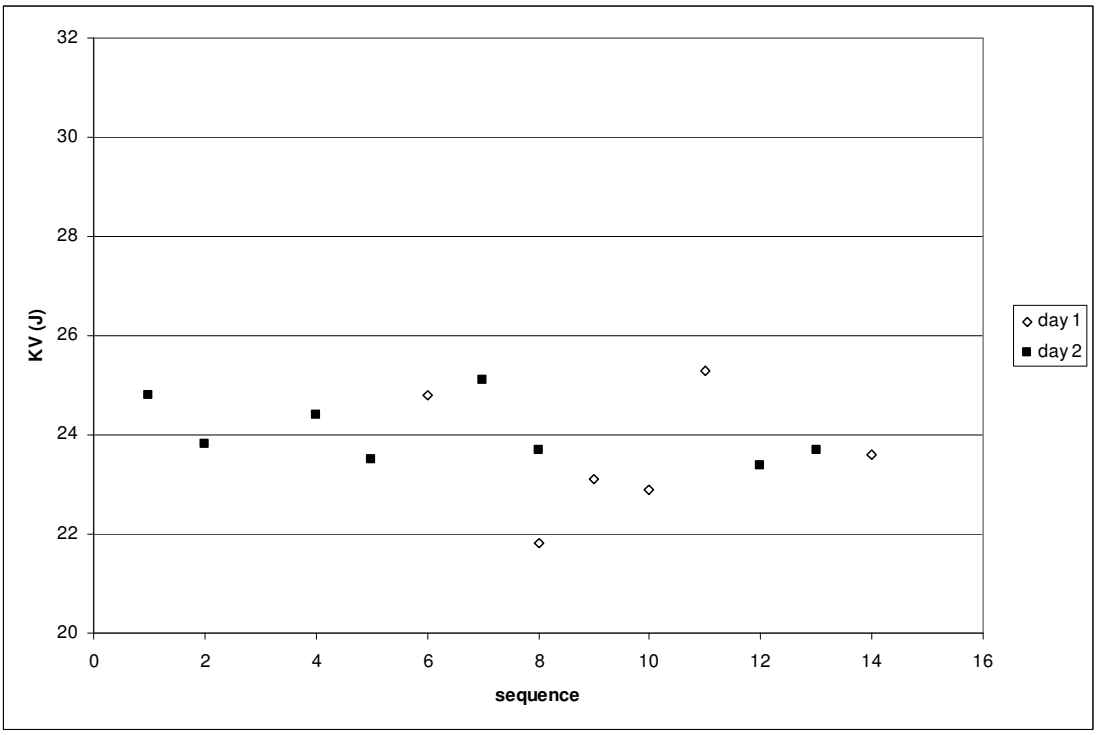
Lab 1: KV data versus test sequence



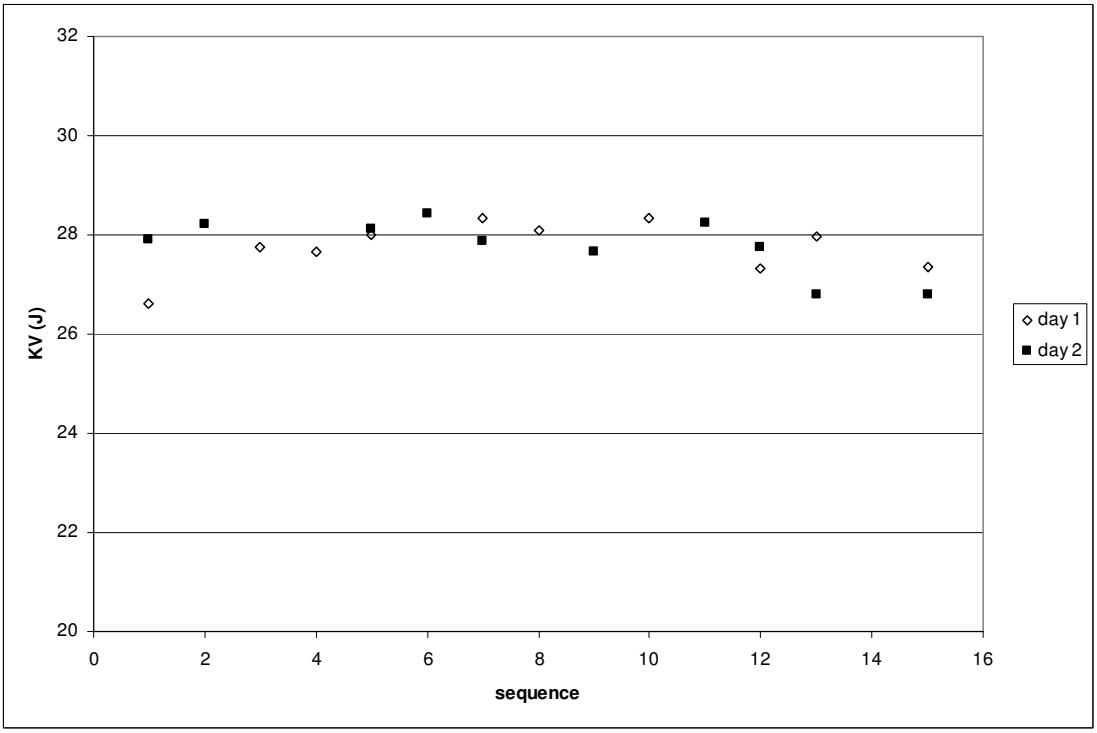
Lab 2: KV data versus test sequence



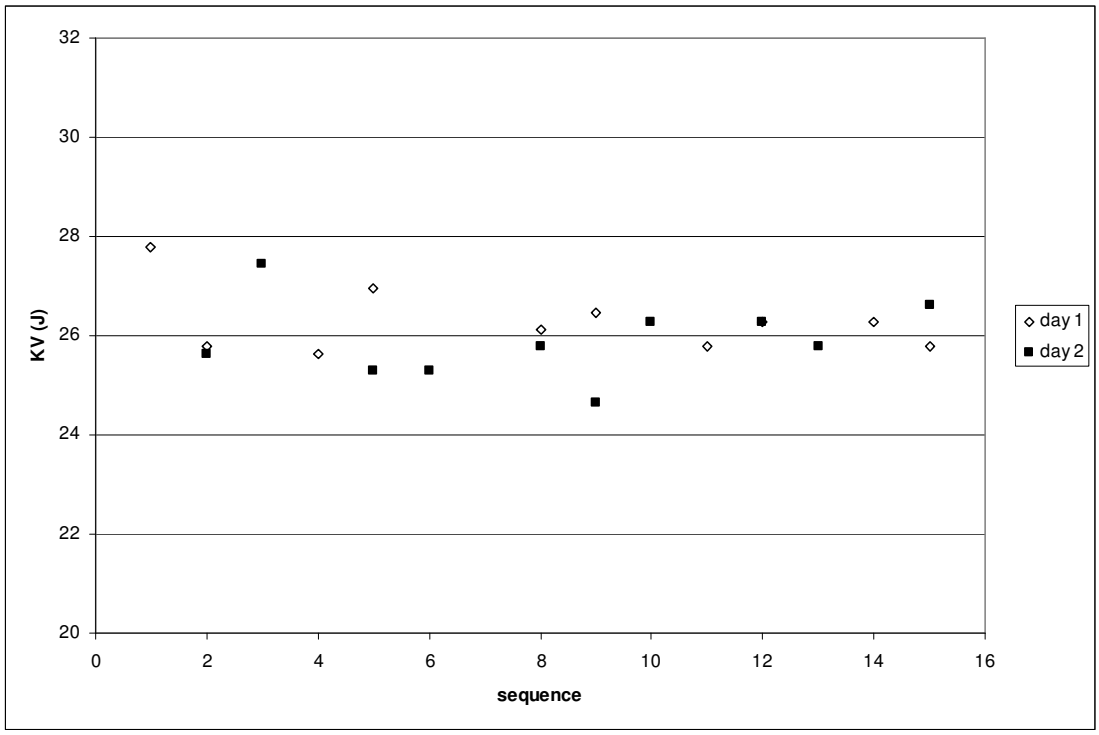
Lab 3: KV data versus test sequence



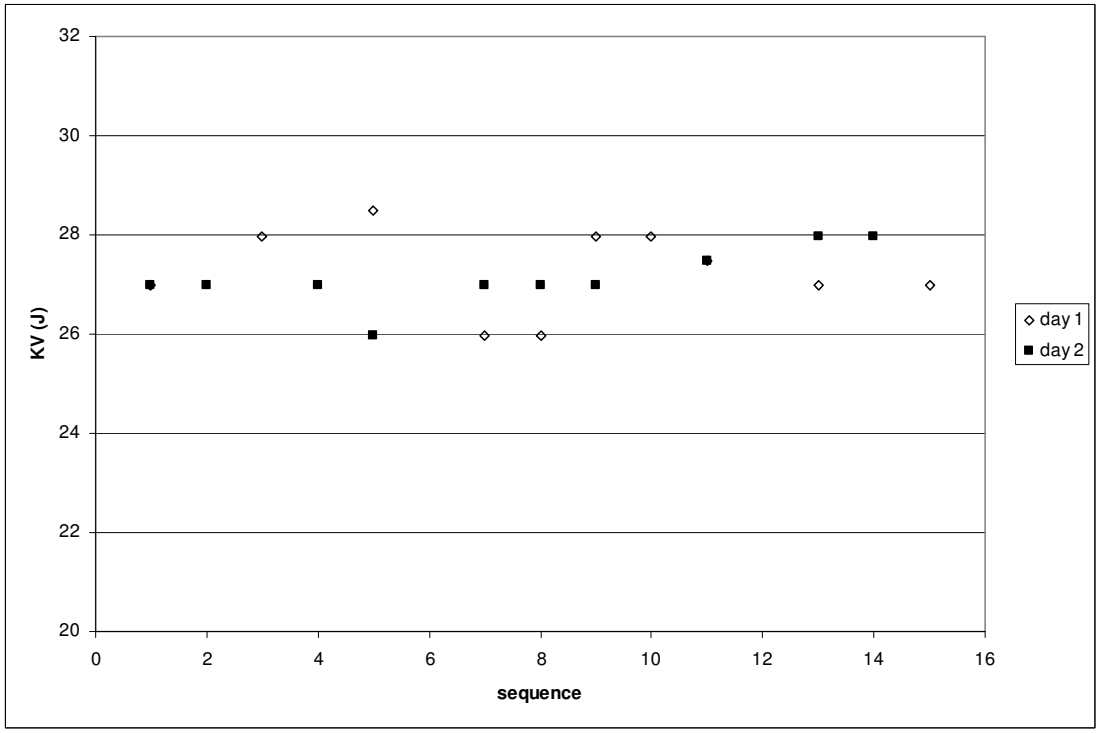
Lab 4: KV data versus test sequence



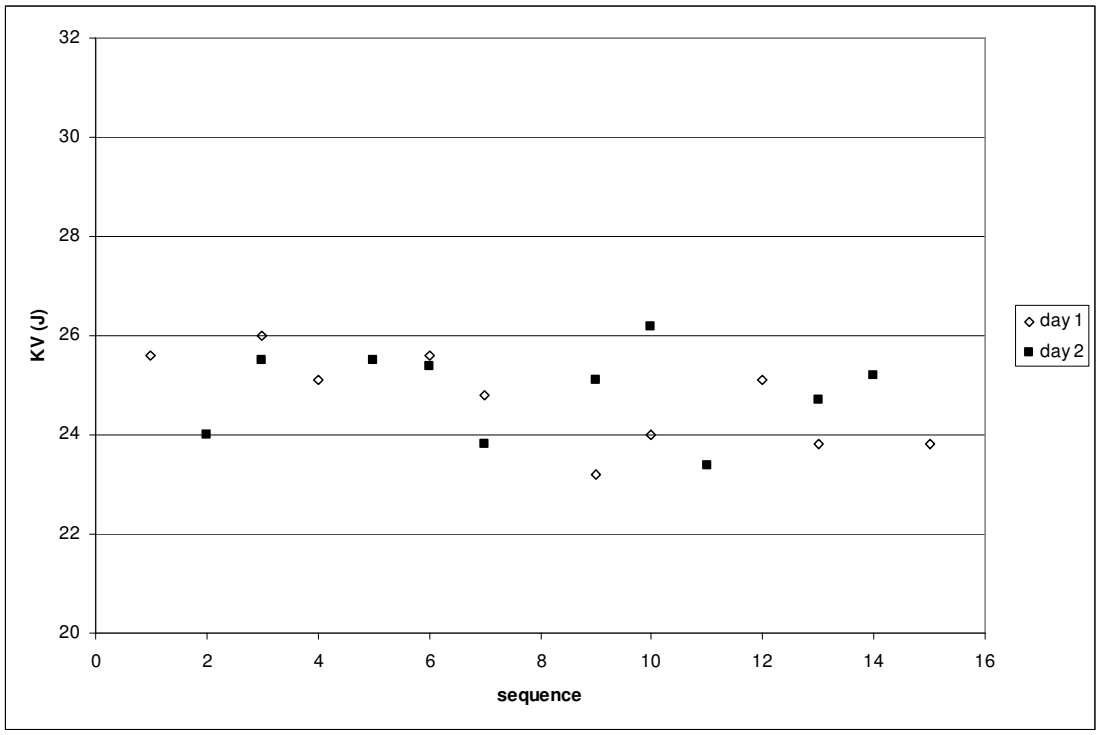
Lab 5: KV data versus test sequence



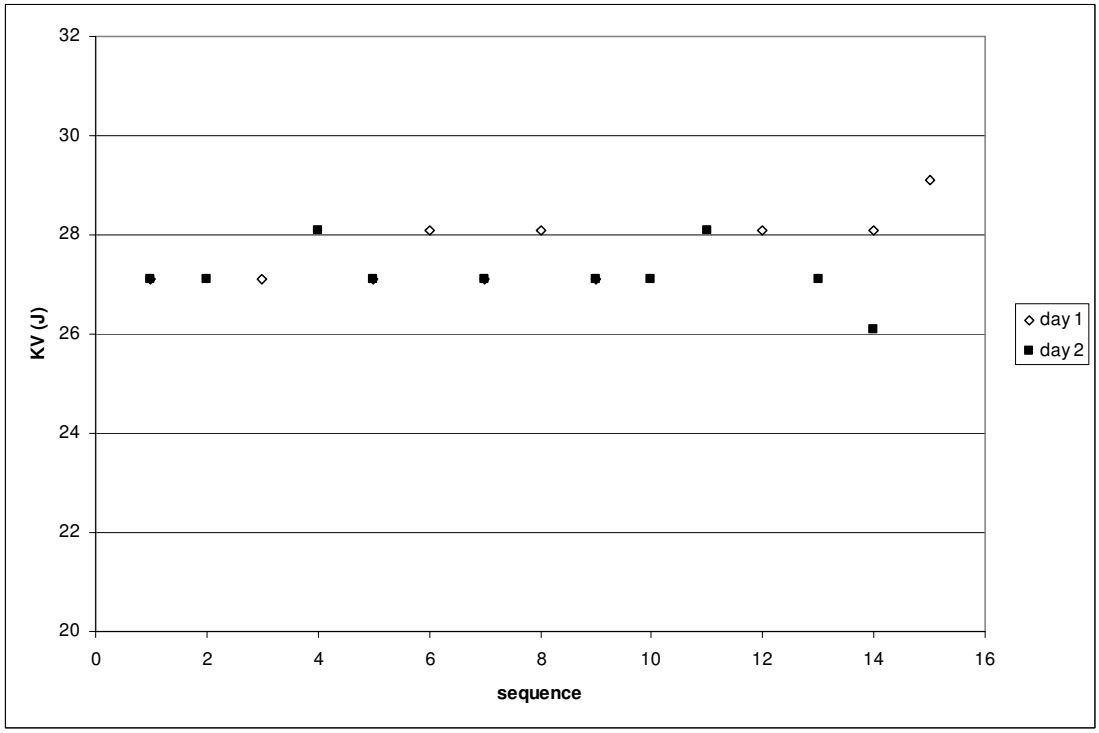
Lab 6: KV data versus test sequence



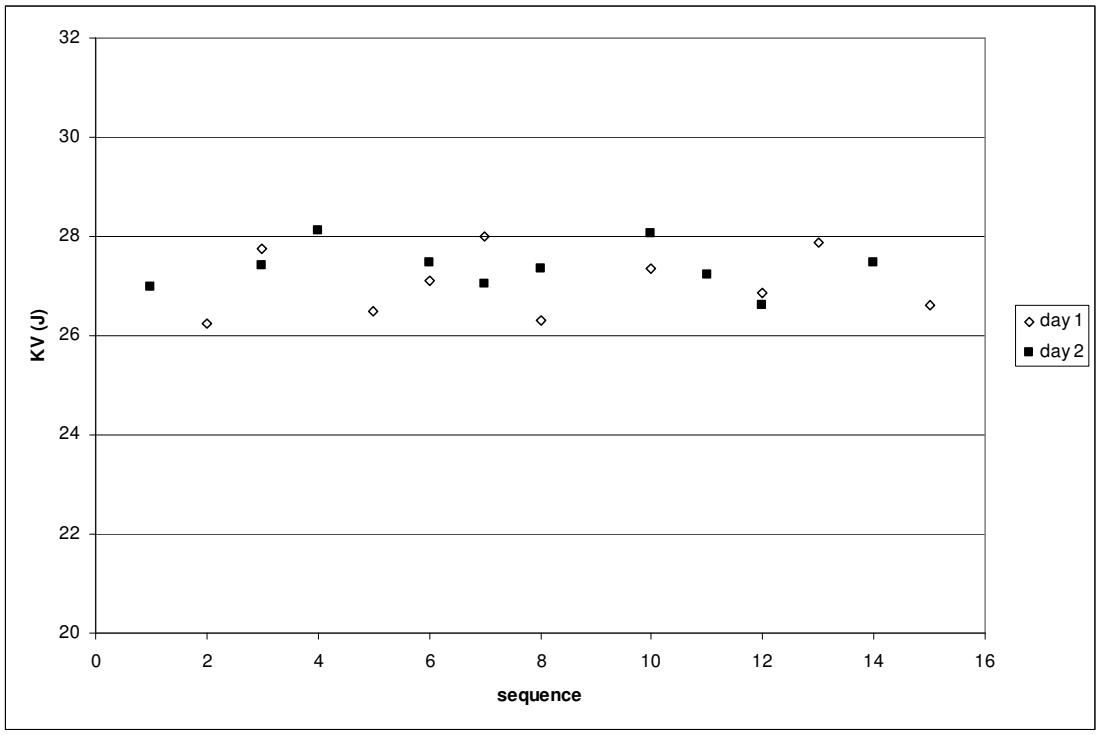
Lab 7: KV data versus test sequence



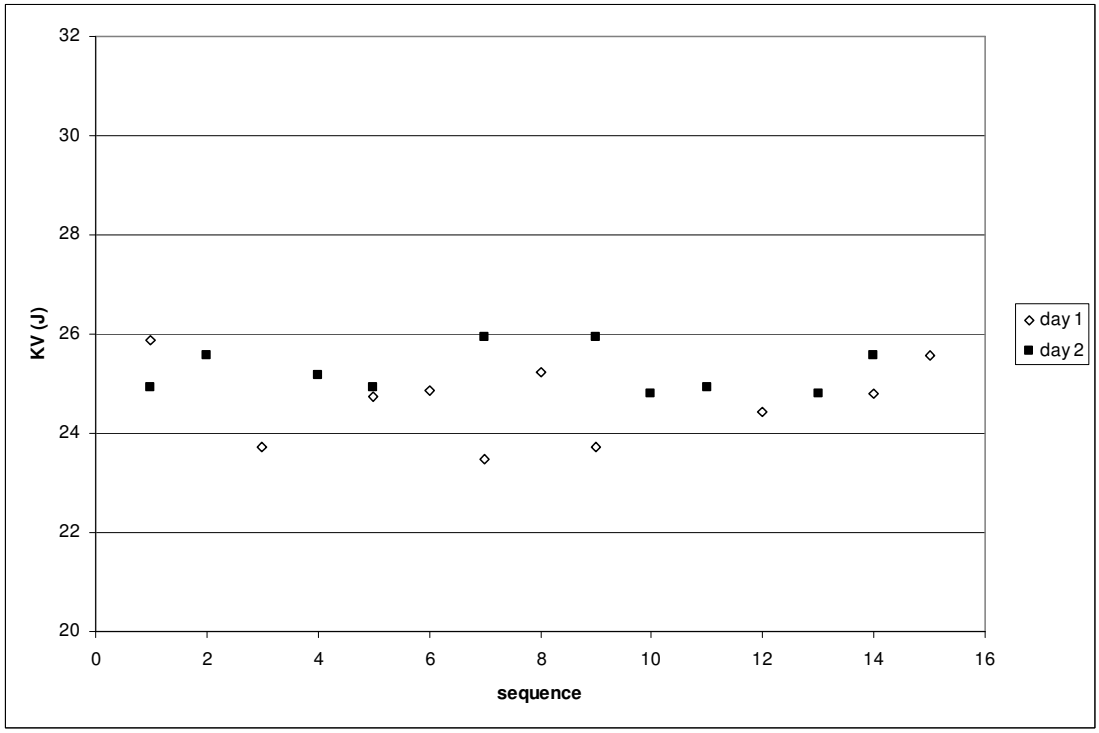
Lab 8: KV data versus test sequence



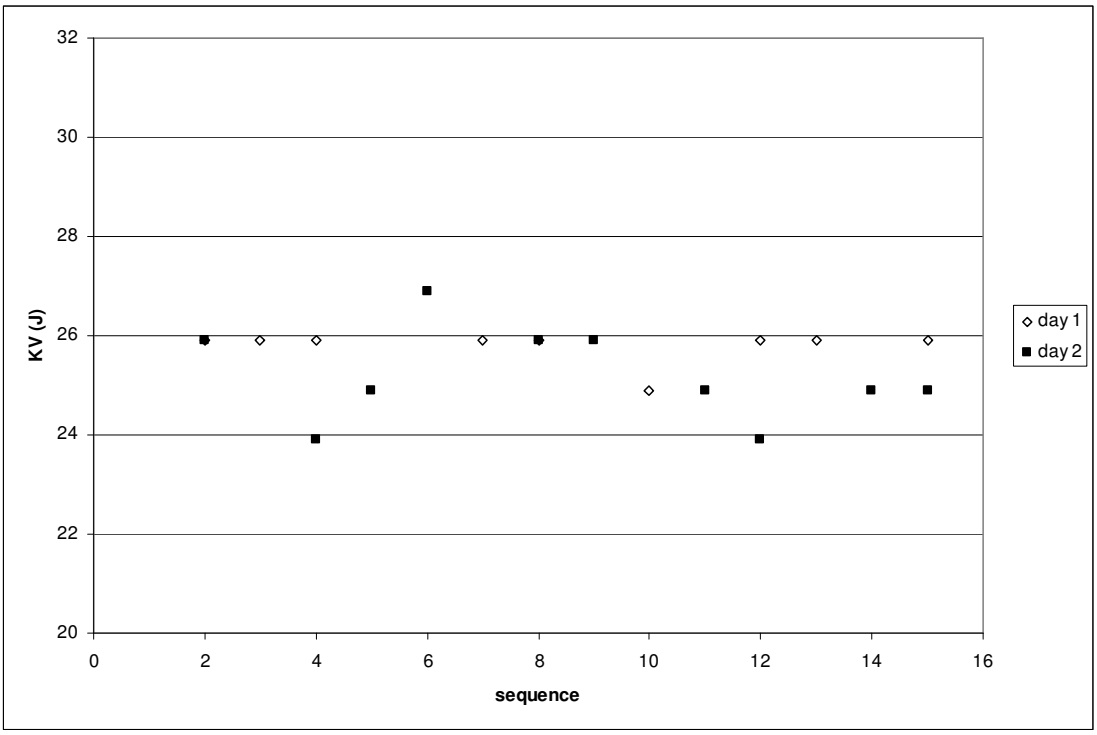
Lab 9: KV data versus test sequence



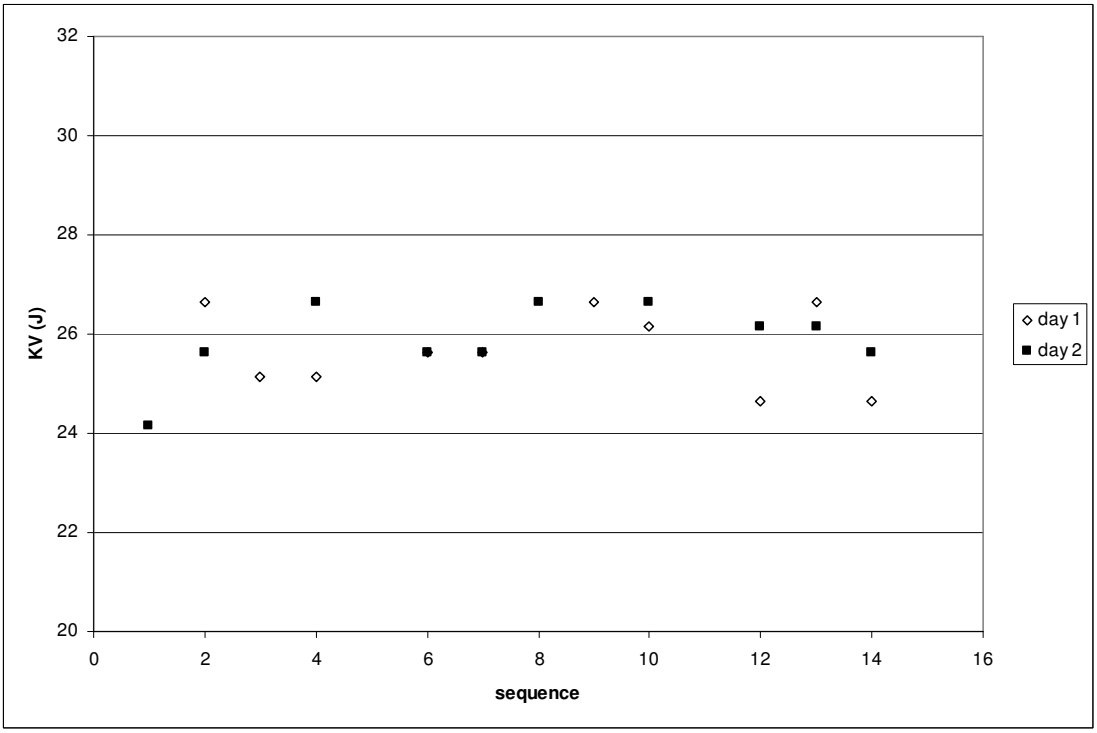
Lab 10: KV data versus test sequence



Lab 11: KV data versus test sequence



Lab 12: KV data versus test sequence



European Commission

EUR 24890 EN – Joint Research Centre – Institute for Reference Materials and Measurements
Title: Certification of a Master Batch of Charpy V-Notch Reference Test Pieces of Nominal Energy Level 30 J for Tests at 0°C, ERM®-FA013ay
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Abstract

This certification report describes the processing and characterisation of ERM®-FA013ay, a batch of steel Charpy V-notch certified reference test pieces for tests at 0 ºC. This batch will serve as a Master Batch, to be used by IRMM for the certification of secondary batches. Sets of five pieces taken from a secondary batch are distributed by IRMM and its authorised distributors for the verification of pendulum impact test machines according to EN 10045-2 [1] and ISO 148-2 [2].

The certified values for *KV* (= absorbed energy = energy required to break a V-notched test piece using a pendulum impact test machine) are estimates of the mean value of the whole batch. The obtained values, deduced from tests at 0 ºC, are shown in the table below. The associated uncertainties are standard uncertainties corresponding to a confidence level of about 68 %. The certified values are defined by the Charpy impact test method as described in EN 10045-1 [3] and ISO 148-1 [4] and are traceable to the International System of Units (SI).

	Certified value KV_{MB} **[J]**	**Standard uncertainty** u_{MB} **[J]**
ERM®-FA013ay	26.06	0.35

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