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CERTIFICATION REPORT

The certification of the absorbed energy (low energy) of Charpy V-notch reference test pieces for tests at 20 °C: ERM®-FA013bv

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European Commission
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Abstract
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The absorbed energy (KV) is procedurally defined and refers to the impact energy required to break a V-notched test piece of standardised dimensions, as defined in ISO 148-1. The certified value of ERM®- FA013bv is made traceable to the SI, via the SI-traceable certified value of the master batch ERM®-FA013ba, by testing samples of ERM®- FA013bv and ERM®-FA013ba under repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools. The certified value is valid only for strikers with a 2 mm tip radius. The certified value is valid at (20 ± 2) °C..

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The certification of the absorbed energy (low energy) of Charpy V-notch reference test pieces for tests at 20 °C: ERM®-FA013bv

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Disclaimer

Certain commercial equipment, instruments, and materials are identified in this paper 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®-FA013bv, a batch of Charpy V-notch certified reference test pieces certified for the absorbed energy (*KV*). Sets of five of these test pieces are used for the verification of pendulum impact test machines according to ISO 148-2 (Metallic materials - Charpy pendulum impact test – Part 2: Verification of testing machines [1]).
The absorbed energy (*KV*) is procedurally defined and refers to the impact energy required to break a V-notched test piece of standardised dimensions, as defined in ISO 148-1 [2]. The certified value of ERM®- FA013bv is made traceable to the SI, via the SI-traceable certified value of the master batch ERM®-FA013ba, by testing samples of ERM®- FA013bv and ERM®-FA013ba under repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools. The certified value is valid only for strikers with a 2 mm tip radius. The certified value is valid at (20 ± 2) °C.

The certified value for *KV* (= energy required to break a V-notched test piece using a pendulum impact test machine) and the associated expanded uncertainties ($k = 2$ corresponding to a confidence level of about 95 %) calculated for the mean of a set of five test pieces, are:

Steel Charpy V-notch test pieces		
	Certified value 2) [J]	Uncertainty 3) [J]
Absorbed energy (*KV*) 1)	19.2	0.7

1) The absorbed energy (*KV*) is a method defined measurand. KV is the impact energy required to break a V-notched bar of standardised dimensions, as defined in ISO 148-1. The certified value is valid only for strikers with a 2 mm tip radius, and in the temperature range of (20 ± 2) °C.

2) The certified value of ERM®-FA013bv, and its uncertainty, is traceable to the International System of Units (SI), via the master batch ERM®-FA013ba of a similar nominal absorbed energy by testing samples of ERM®-FA013ba and ERM®-FA013bv under repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools.

3) Estimated expanded uncertainty of the mean *KV* of the 5 specimens (delivered as 1 set), with a coverage factor $k = 2$, corresponding to a level of confidence of about 95 %, as defined in ISO/IEC Guide 98-3, Guide to the expression of uncertainty in measurement (GUM:1995). The number of degrees of freedom of the certified uncertainty is $\nu_{RM} = 70$.

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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
ERM®	European Reference Material
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_{CRM}	Certified KV value of a set of 5 reference test pieces from the Secondary Batch
KV_{MB}	Certified KV value of the Master Batch test pieces
LNE	Laboratoire national de métrologie et d'essais
MB	Master Batch
n_{MB}	Number of samples of the Master Batch tested during certification of the Secondary Batch
n_{SB}	Number of samples of the Secondary Batch tested for certification
RSD	Relative standard deviation
s	Standard deviation
SB	Secondary Batch
s_h	Standard deviation of the results of the samples tested to assess the homogeneity of the Secondary Batch
s_{MB}	Standard deviation of the n_{MB} results of the samples of the Master Batch tested for the certification of the Secondary Batch
s_{SB}	Standard deviation of the n_{SB} results of the samples tested for the characterisation of the Secondary Batch
u_{CRM}	Combined standard uncertainty of KV_{CRM}
U_{CRM}	Expanded uncertainty (k = 2, confidence level of about 95 %) of KV_{CRM}
u_{char}	Standard uncertainty of the result of the characterisation tests
$u_{char,rel}$	Relative standard uncertainty of the result of the characterisation tests
u_h	Contribution to uncertainty from homogeneity
u_i	Value of uncertainty from contribution i
u_{MB}	Standard uncertainty of KV_{MB}
$u_{MB,rel}$	Relative standard uncertainty of KV_{MB}
$\overline{X}_{MB}$	Mean KV value of the n_{MB} measurements on samples of the Master Batch tested when characterising the Secondary Batch
$\overline{X}_{SB}$	Mean KV value of the n_{SB} results of the samples tested for the characterisation of the Secondary Batch
Δh	difference between the height of the centre of gravity of the pendulum prior to release and at the end of the half-swing during which the test sample is broken
ν_{RM}	Effective number of degrees of freedom associated with the uncertainty of the certified value

1 Introduction

1.1 *The Charpy pendulum impact test*

The Charpy pendulum impact test is designed to assess the resistance of a material to shock 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.

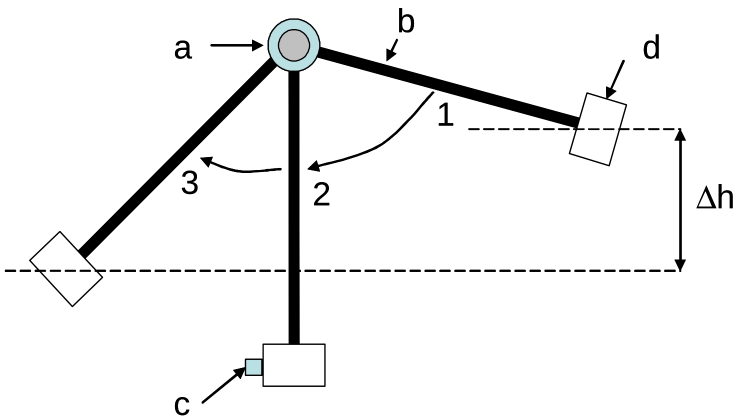

Figure 1: Schematic presentation of the Charpy pendulum impact test, showing a: the horizontal rotation axis of the pendulum, b: the stiff shaft on to which is fixed d: the hammer. The hammer is released from a well-defined height (position 1). When the hammer has reached maximum kinetic energy (shaft in vertical position 2), the hammer strikes c: the test sample, which is positioned on a support and against the pendulum anvils (not shown). 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 with a difference in potential energy, and is a measure of the energy required to break the test sample.

The energy absorbed by the test sample is very dependent 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 ISO and other international standards [1, 3]. The reference test pieces dealt with in this report comply with a V-notched test piece shape of well-defined geometry [1], schematically shown in Figure 2.

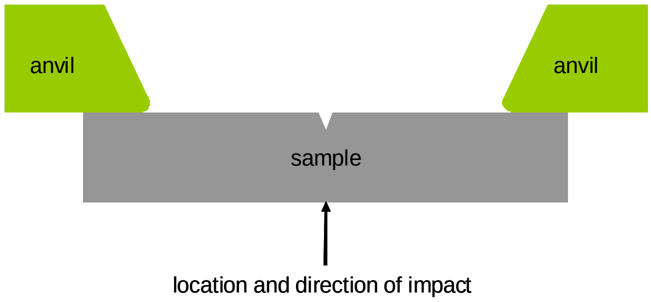

Figure 2: Schematic drawing of a V-notched Charpy test piece (top-view), indicating the place and direction of impact.

1.2 The certification concept of Master Batch and Secondary Batch

1.2.1 Difference between Master and Secondary Batches

The BCR reports by Marchandise et al. [4] and Varma [5] provide details of the certification of BCR "Master Batches" (MB) of Charpy V-notch certified reference test pieces. The certified value of a Master Batch is obtained using an international laboratory intercomparison.
This report describes the production of a "Secondary Batch" (SB) of Charpy V-notch certified reference test pieces at the Institute for Reference Materials and Measurements (IRMM) of the European Commission's (EC) Joint Research Centre (JRC). The work was performed in accordance with procedures described in the BCR reports [4] and [5]. The certification of a SB is based on the comparison of a set of SB test pieces with a set of test pieces from the corresponding MB under repeatability conditions on a single pendulum.

The BCR reports [4] and [5] 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/IEC Guide to the Expression of Uncertainty in Measurement [6]. This revised approach was developed and presented by Ingelbrecht et al. [7, 8], and is summarised below.

1.2.2 Certification of a Secondary Batch of Charpy V-notch test pieces

The certified absorbed energy of a SB of Charpy V-notch reference test pieces (KV_{CRM}) is calculated from the mean *KV*-value of a set of SB-samples ($\overline{X}_{\mathrm{SB}}$) tested on a single pendulum. This value $\overline{X}_{\mathrm{SB}}$ has to be corrected for the bias of this particular pendulum. The bias of the pendulum at the moment of testing the samples of the SB, is estimated by comparing the mean *KV*-value of a number of samples of the MB ($\overline{X}_{\mathrm{MB}}$), tested together with the SB samples under repeatability conditions, with the certified value of the MB (KV_{MB}). KV_{CRM} is then calculated as follows [8]:

$$KV_{\mathrm{CRM}} = \left[\frac{KV_{\mathrm{MB}}}{\overline{X}_{\mathrm{MB}}} \cdot \overline{X}_{\mathrm{SB}}\right] \qquad \text{Eq. 1}$$

For this approach to be reliable, the pendulum used for the tests on MB and SB in repeatability conditions, must be well performing. In other words, the ratio $\frac{KV_{\mathrm{MB}}}{\overline{X}_{\mathrm{MB}}}$ must be close to 1. IRMM allows a difference of 5 % ($KV_{\mathrm{MB}} \geq 40$ J) or 2 J ($KV_{\mathrm{MB}} < 40$ J) between KV_{MB} and $\overline{X}_{\mathrm{MB}}$, corresponding with the level of bias allowed for reference pendulums specified in ISO 148-3 [9].

Also, for reasons of commutability, a comparable response of the pendulum to the MB and SB samples is required. This is the reason why MB and SB samples are made from nominally the same steel. Moreover, it is checked that the ratio $\frac{KV_{\mathrm{CRM}}}{KV_{\mathrm{MB}}}$ is close to 1. IRMM allows a difference of 20 % ($KV_{\mathrm{MB}} \geq 40$ J) or 8 J ($KV_{\mathrm{MB}} < 40$ J) between KV_{CRM} and KV_{MB} to ensure that the MB and SB samples have a comparable interaction with the pendulum.

1.2.3 Uncertainty of the certified value of a Secondary Batch of Charpy V-notch test pieces

The uncertainty of the certified value of the SB is a combination of the uncertainties of the right-hand side factors in Eq. 1. It is clear that the MB-SB approach necessarily results in a larger uncertainty of the certified value of SB in comparison with the MB. The additional uncertainty depends on the uncertainty of the ratio $\overline{X}_{MB}/\overline{X}_{SB}$. The full measurement uncertainty of the values $\overline{X}_{MB}$ and $\overline{X}_{SB}$ is relatively large. However, when all conditions mentioned above (repeatability conditions, pendulum performance, and commutability between Secondary and Master Batch) are fulfilled, then the uncertainties of the values $\overline{X}_{MB}$ and $\overline{X}_{SB}$ have several contributions in common, in particular the uncertainty due to the bias of the pendulum. These shared uncertainty components do not contribute to the uncertainty of the ratio $\overline{X}_{MB}/\overline{X}_{SB}$, and only the standard deviations of the SB and MB results in the MB-SB comparison test need to be taken into account (see also Section 6.3). Thus, the MB-SB comparison approach can produce a value for the uncertainty of KV_{CRM} that is sufficiently small to meet the requirements of the intended use of the certified reference material (CRM).

2 Participants

The processing of the SB (ERM®- FA013bv) test pieces was carried out by the Institut für Eignungsprüfung GmbH (IfEP), Marl (DE), using AISI4340/1.6565 steel delivered by Tata Steel International Germany GmbH. The MB samples (ERM®-FA013ba) used in the characterisation of the SB were provided by IRMM, Geel (BE). The homogeneity of the SB was evaluated based on data obtained at IfEP using a pendulum verified according to the criteria imposed by ISO 148-2 [1]. Characterisation of the SB was carried out at IRMM using a pendulum verified according to the criteria imposed by ISO 148-2 [1]. The tests performed were within the scope of an ISO/IEC 17025 accreditation (BELAC 268-Test).
Data evaluation was performed at IRMM. The certification project performed was within the scope of an ISO Guide 34 accreditation (BELAC 268-RM).

3 Processing

The ERM®- FA013bv test pieces were prepared from bars of AISI 4340/1.6565 steel delivered by Tata Steel International Germany GmbH. Production of the test pieces from these bars was performed under the supervision of IfEP (see sections 3.1 - 3.5).

3.1 Machining of Charpy test pieces

The broad bars were cut into 65 discs each. 14 discs from one bar were cut into 1628 Charpy specimens and machined to the dimensional requirements of ISO 148-3 [9] and engraved to ensure identification.

3.2 Heat treatment of hot-rolled bars

The heat treatment of the specimens was performed at VTN Witten GmbH (DE) in a vacuum-furnace. 1628 specimens of the batch were heat-treated according to the following procedure:
Step 1: normalisation treatment at 900 °C for 150 minutes
Step 2: Cool down in air
Step 3: hardening at 850°C for 90 minutes
Step 4: quenching in oil
Step 5: first tempering at 280 °C for 180 minutes

Step 6: cool down in air
Step 7: second tempering at 280 °C for 180 minutes
Step 8 cool down in air.
The specimens were distributed over 3 baskets. The baskets were stacked in the furnace and each basket had three thermocouples to monitor the temperature homogeneity. The measured temperatures at all positions were within the tolerance of +/- 6 °C as required by IfEP.

3.3 Final machining of Charpy test pieces

After heat treatment, the specimens were machined to the final dimensions specified in ISO 148-3 [9] by IfEP. During this process the specimen numbers were transferred to one of the end faces. Finally the specimens were notched using a milling process.

3.4 Quality control

When all samples from the batch were fully machined, a selection of 25 samples was made. The dimensions of the 25 samples were checked on against the criteria specified in ISO 148-3 [9] (length $55.0^{+0.00}_{-0.30}$ mm, height 10.00 ± 0.06 mm, width 10.00 ± 0.07 mm, notch angle 45 ± 1°, height remaining at notch root 8.00 ± 0.06 mm, radius at notch root 0.250 ± 0.025 mm, distance between the plane of symmetry of the notch and the longitudinal axis of the test piece 27.5 ± 0.2 mm). None of the samples was outside the ranges specified in ISO 148-3 [9].

25 samples selected throughout the batch were checked for geometrical compliance and impact tested using a pendulum type Zwick PSW 750 (nominal energy 450 J), verified in September 2014 according to ISO 148-2 [1].
The tests were done on December 10, 2014. The results are reported in the production report of December 16, 2014 [10]. The average *KV* of the 25 samples was 19.64 J, which is within the desired energy range (15 J to 25 J). The standard deviation of the test results (s = 0.54 J, *RSD* = 2.77 %) was below the 1.2 J maximum allowed by the contract. The variation was checked again during the characterisation tests at IRMM (see Section 0).

3.5 Packaging and storage

Finally, the samples were cleaned and packed in sets of 5 randomised samples, in oil-filled and vacuum sealed plastic bags. These oil-filled bags, were packed in a second sealed plastic bag, and shipped to IRMM. After arrival (January 14, 2015) the 1595 samples (or 319 sets) of ERM®-FA013bv were registered and stored at room temperature, pending distribution.

4 Homogeneity

The test pieces are sampled from the SBs, which are sufficiently, but not perfectly, homogeneous. Therefore, an appropriate homogeneity contribution u_h to the uncertainty of the certified value is required. u_h is related to s_h, the standard deviation between the samples in the SB (*sample-to-sample heterogeneity*), but also depends on the number of samples over which the *KV*-value is averaged. ISO 148-2 [1] specify that the pendulum verification must be performed using 5 test pieces, which is why a CRM-unit consists of a set of 5 test pieces. The appropriate uncertainty contribution must be an estimate of the *set-to-set heterogeneity*, which in the case of a set of 5 test pieces can be calculated as $u_h = \frac{s_h}{\sqrt{5}}$.

Here, u_h is estimated from s_h, the standard deviation of results obtained at IfEP on December 10, 2014 (s_h = 0. 54 J). This leads to $u_h = \frac{s_h}{\sqrt{5}} = 0.24$ J (1.23%).

As is required for a homogeneity test, the samples were randomly selected from the whole batch. The number of samples tested (25) is sufficiently large to reflect the homogeneity of the full SB (1595 samples). It can be noted that u_h is probably a slight overestimation, since it contains also the repeatability of the instrument. However, the latter cannot be separated or separately measured.

5 Stability

The stability of the absorbed energy of Charpy V-notch certified reference test pieces was first systematically investigated for samples of nominally 120 J by Pauwels et al., who did not observe measurable changes of absorbed energy [11]. Additional evidence for the stability of the reference test pieces produced from AISI 4340 steel of lower energy levels (nominally 15 J, 30 J and 100 J) has been obtained during the International Master Batch (IMB) project [12]. In the IMB-project, the stability of the certified test pieces was judged from the change of the mean of means of the absorbed energy obtained on 7 reference pendulums over a three year period. None of the three regression slopes for the tested energy levels was statistically significant at the 5 % probability level. Given the large sample-to-sample heterogeneity and the limited number of samples (5) in a CRM unit, the uncertainty contribution from instability is considered to be insignificant in comparison to that of homogeneity. A dedicated isochronous study (test temperature 18 °C, reference temperature -20 °C) was organised by IRMM by batches of 30, 80 and 120 J from the same steel was organised by IRMM and showed, as expected, no change of the measured values. Uncertainty of stabilities for 120 months were calculated as 0.7-2.8 J (1.8-2.4 %). These uncertainties are entirely driven by the measurement precision and it was concluded that no uncertainty contribution for potential change was needed [14].

The main reason for the microstructural stability of the certified reference test pieces is 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-)stress 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.

Rather than neglecting the stability issue, efforts are spent to better establish the stability of the certified values of batches of Charpy CRMs. Until such further notice, it is decided to specify a limited shelf-life. A period of 10 years is chosen, counting from the date of the characterisation tests on the SB. Since batch ERM®-FA013bv was characterised in February, 2015, the validity of the certificate reaches until February, 2025.

6 Characterisation

6.1 Characterisation tests

30 samples from ERM®-FA013bv (sets 5, 100, 152, 188, 242 and 285) were tested under repeatability conditions together with 25 samples from MB ERM®-FA013ba (sets 77, 90, 100, 105 and 107), using the Instron Wolpert PW 30 (serial number 7300 H1527) machine of IRMM, an impact pendulum yearly verified according to procedures described in ISO 148-2 [1].
Tests were performed on February 10, 2015 (laboratory temperature 20 ± 1 °C), in accordance with ISO 148-1 [2]. The measurement sequence was: SB-MB-SB-MB-SB-MB-SB-MB-SB-MB-SB. The measured absorbed energy values were corrected for friction and windage losses.

After testing, all Charpy samples show 'first-strike' marks: these are the marks caused by the interaction between sample and anvils during the first and intended hammer impact. Upon fracture, the broken halve samples loose contact with hammer and anvils and follow one of a variety of possible trajectories, away from the pendulum, depending on the properties of both pendulum and test material. It may occur that samples show 'second-strike' marks. These are marks caused by a second impact of the already broken halve samples back onto the anvils. This phenomenon has been described by Schmieder et al. [13]. None of the broken FA013bv but three of FA013ba samples showed second-strike marks. These results of FA013ba was therefore not included in the further calculations.
The accepted data obtained on individual test pieces are shown in Figure 3 and Annex 1. The results of the measurements are summarised in Table 1.

The sequence of the samples shows three high values for the master batch. These were flagged as outliers using the Grubbs procedure but were retained, as the respective set samples met the specifications of ISO 148-3 for reference samples (standard deviation < 3 J).

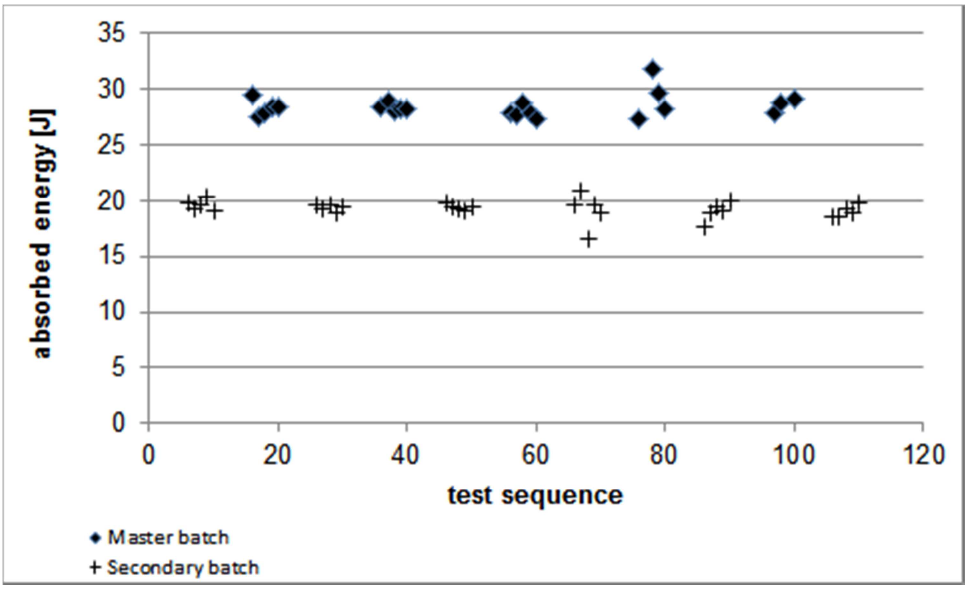

Figure 3: Absorbed energy values of 22 test pieces of ERM®-FA013ba, compared with 30 test pieces of ERM®-FA013bv; data are displayed in the actual test sequence

Table 1: Characterisation measurements of Batch ERM®-FA013bv

	Number of test pieces n_{MB} , n_{SB}	Mean value $\overline{X}_{MB}$, $\overline{X}_{SB}$ [J]	Standard deviation s_{MB} , s_{SB} [J]	Relative standard deviation RSD_{SB}, RSD_{MB} [%]
ERM®-FA013ba (MB)	22	28.46	0.98	3.45
ERM®-FA013bv (SB)	30	19.25	0.78	4.04

The SB-results meet the ISO 148-3 acceptance criteria for a batch of reference materials (s_{SB} < 2 J for KV_{SB} < 40 J).

6.2 Data from Master Batch ERM®-FA013ba

To calculate KV_{CRM} for ERM®-FA013bv one needs KV_{MB} of the MB used, i.e. ERM®-FA013ba. Table 2 shows the main MB-data, taken from the Certificate of Analysis of ERM®-FA013ba (Annex 2).

Table 2: Data from the certification of Master Batch ERM®-FA013ba

	Certified absorbed energy of Master Batch KV_{MB} (J)	Standard uncertainty of KV_{MB} u_{MB} (J)	Relative standard uncertainty of KV_{MB} $u_{MB,rel}$ (%)
ERM®-FA013ba	28.46	0.23	0.81

6.3 Calculation of KV_{CRM} and of u_{char}

From the data in Tables 2 and 3, and using Eq. 1, one readily obtains that KV_{CRM} = 19.25 J (rounding in accordance with uncertainty; see Table 4). The uncertainty associated with the characterisation of the SB, u_{char}, is assessed as in Eq. 2 [8], which sums the relative uncertainties of the three factors in Eq. 1:

$$u_{char} = KV_{CRM}\sqrt{\frac{u_{MB}^2}{KV_{MB}^2} + \frac{s_{SB}^2}{n_{SB}\cdot \overline{X}_{SB}^2} + \frac{s_{MB}^2}{n_{MB}\cdot \overline{X}_{MB}^2}} \qquad \text{Eq. 2}$$

$\overline{X}_{SB}$ and $\overline{X}_{MB}$ were obtained under repeatability conditions. Therefore, the uncertainty of the ratio $\overline{X}_{SB}/\overline{X}_{MB}$ is not affected by the contributions from reproducibility and bias of the pendulum used to compare MB and SB. Table 3 summarises the input quantities of the u_{char} uncertainty budget, their respective statistical properties, and shows how they were combined. The effective number of degrees of freedom (ν_{eff}) for u_{char} is obtained using the Welch-Satterthwaite equation from the combined uncertainty (u_c) and the individual uncertainty contributions (u_i) and their respective degrees of freedom (ν_i) (Eq. 3) [6].

$$\nu_{eff} = \frac{u_c^4}{\sum_{i=1}^{N} \frac{u_i^4}{\nu_i}} \qquad \text{Eq. 3}$$

Table 3: Uncertainty budget for u_{char} for ERM®-FA013bv

	source of uncertainty	measured value (J)	standard uncertainty (J)	probability distribution	relative uncertainty (%)	degrees of freedom
KV_{MB}	Certification of MB	28.46	0.23	normal	0.81	14
$\overline{x}_{SB}$	comparison of SB and MB in repeatability conditions	19.26	0.14	normal	0.74	29
$\overline{x}_{MB}$		28.46	0.21	normal	0.74	21
					$u_{char,rel}$ (%) 1.32	55
					u_{char} (J) 0.25	

7 Value assignment

7.1 Certified value, combined and expanded uncertainty

As shown in 6.3, KV_{CRM} = 19.25 J. The uncertainty of the certified value is obtained by combining the contributions from the characterisation study, u_{char}, and from the homogeneity assessment, u_h, as is summarized in the following uncertainty budget (Table 4).

The relevant number of degrees of freedom calculated using the Welch-Satterthwaite equation [6], is sufficiently large (ν_{RM} = 70) to justify the use of a coverage factor k = 2 to expand the confidence level to about 95 %. The obtained expanded uncertainty provides justification for the SB-MB approach followed: U_{CRM} is sufficiently smaller (U_{CRM} = 0.7 J) than the verification criterion of 4 J for industrial pendulums [1] or even 2 J for reference pendulums [9].

Table 4: Uncertainty budget of KV_{CRM} for ERM®-FA013bv

	source of uncertainty	relative value u_i (%)	degrees of freedom
u_{char}	characterisation of SB	1.32	55
u_h	homogeneity of SB	1.23	24
Combined standard uncertainty, u_{CRM} (%)		1.80	70
Combined standard uncertainty, u_{CRM} (J)		0.35	
Expanded Uncertainty, k = 2, U_{CRM} (%)		3.6	
Expanded Uncertainty, k = 2, U_{CRM} (J)		0.7	

8 Metrological traceability

The certified property is defined by the Charpy pendulum impact test procedure described in ISO 148-1 [2].

The certified value of the MB ERM®-FA013ba is traceable to the SI, since it was obtained using an interlaboratory comparison, involving a representative selection of qualified laboratories performing the tests in accordance with the standard procedures and using instruments verified and calibrated with SI-traceable calibration tools.

The certified value of ERM®-FA013bv is made traceable to the SI-traceable certified value of the MB by testing SB and MB samples in repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools. Therefore, the certified value of ERM®-FA013bv is traceable to the International System of Units (SI) via the corresponding Master Batch ERM®-FA013ba of a similar nominal absorbed energy (25 J). Absorbed energy *KV* is a method-specific value, and can only be obtained by following the procedures specified in ISO 148-1 [2].

9 Commutability

The intended use of the certified reference test pieces is the verification of Charpy impact pendulums. During the certification of the MB, different pendulums were used, each equipped with an ISO-type striker of 2 mm tip radius. Until further notice, the certified values are not to be used when the test pieces are broken with an ASTM-type striker of 8 mm tip radius [10].

10 Summary of results

The certified value and associated uncertainty are summarized in Table 5.

Table 5: Certified value and associated uncertainty for ERM®-FA013bv.

Steel Charpy V-notch test pieces		
	Certified value 2) [J]	Uncertainty 3) [J]
Absorbed energy (*KV*) 1)	19.2	0.7
1) The absorbed energy (*KV*) is a method defined measurand. KV is the impact energy required to break a V-notched bar of standardised dimensions, as defined in ISO 148-1. The certified value is valid only for strikers with a 2 mm tip radius, and in the temperature range of (20 ± 2) °C. 2) The certified value of ERM®-FA013bv, and its uncertainty, is traceable to the International System of Units (SI), via the master batch ERM®-FA013ba of a similar nominal absorbed energy by testing samples of ERM®-FA013ba and ERM®-FA013bv under repeatability conditions on an impact pendulum verified and calibrated with SI-traceable calibrated tools. 3) Estimated expanded uncertainty of the mean *KV* of the 5 specimens (delivered as 1 set), with a coverage factor k = 2, corresponding to a level of confidence of about 95 %, as defined in ISO/IEC Guide 98-3, Guide to the expression of uncertainty in measurement (GUM:1995). The number of degrees of freedom of the certified uncertainty is ν_{RM} = 70.		

11 Instructions for use

11.1 Intended use

Samples of ERM®-FA013bu correspond to the 'certified reference test pieces' as defined in ISO 148-3 [9]. Sets of five of these certified reference test pieces are intended for the indirect verification of impact testing machines with a striker of 2 mm tip radius according to procedures described in detail in ISO 148-2 [1].

The indirect verification provides an assessment of the bias of the user's Charpy pendulum impact machine. This bias assessment can be used in the calculation of the measurement uncertainty of Charpy tests on the pendulum after indirect verification. Such uncertainty calculation requires the certified value, the associated uncertainty, and in some cases also the degrees of freedom of the uncertainty, all given on page 1 of the certificate.

11.2 Sample preparation

Special attention is drawn to the cleaning of the specimens prior to the tests. 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 a 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. Before testing, bring the specimens to the test temperature (20 ± 2) °C. To assure thermal equilibrium is reached, move the specimens to the test laboratory at least 3 h before the tests.

An optional cleaning step with organic solvents may be inserted between 1 and 2. Please note that the use of ethanol is discouraged, as it results in a sticky residue with remaining traces of oil. Any residual solvents shall be removed by wiping with an absorbent tissue before proceeding to step 2.

11.3 Pendulum impact tests

After cleaning, the 5 samples constituting a CRM-unit need to be broken with a pendulum impact test machine in accordance with ISO 148-2 [1] 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. The uncertainty of the certified value applies to the mean of the 5 *KV*-values.

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Annex 1

Results of characterisation measurements of ERM®-FA013bv as measured according to ISO 148-1 at IRMM, 10 February, 2015.

	Master Batch ERM®-FA013ba	**Secondary Batch ERM®-FA013bv**
	***KV* (J)**	***KV* (J)**
1	29.54	19.87
2	27.56	19.28
3	27.82	19.63
4	28.48	20.35
5	28.48	19.04
6	28.35	19.59
7	28.87	19.23
8	28.08	19.59
9	28.21	18.88
10	28.21	19.35
11	27.95	19.82
12	27.69	19.35
13	28.74	19.23
14	27.95	19.12
15	27.3	19.35
16	27.43	19.70
17	jammed	20.90
18	31.82	16.59
19	29.67	19.70
20	28.21	18.88
21	jammed	17.61
22	27.89	18.84
23	28.81	19.42
24	jammed	19.07
25	29.21	20.01
26		18.60
27		18.60
28		19.19
29		18.95
30		19.78
Mean (J)	**28.46**	**19.25**
Standard deviation (J)	**0.98**	**0.78**
***RSD* (%)**	**3.45**	**4.04**

Annex 2

CERTIFICATE OF ANALYSIS

ERM®- FA013ba

STEEL		
	Impact toughness	
	Certified value 2) [J]	Uncertainty 3) [J]
Absorbed energy (*KV*) 1)	28.46	0.23

1) The absorbed energy (*KV*) is procedurally defined and refers to the impact energy required to break a V-notched bar of standardised dimensions, as defined in EN 10045-1 and ISO 148-1.

2) The certified value is estimated as the mean of means of absorbed energies measured at 15 laboratories. At each laboratory, 20 test pieces were broken. The instruments used by these laboratories are regularly verified with equipment that is calibrated in a manner that is traceable to the International System of Units (SI). Therefore, the certified value is traceable to the International System of Units (SI).

3) Standard uncertainty *u* of the certified mean absorbed energy of batch ERM-FA013ba, estimated as the standard deviation of the mean of the 15 laboratory mean values, corresponding with a confidence level of about 68 %.

This certificate is valid until January 2018.

NOTE

European Reference Material ERM®-FA013ba was produced and certified under the responsibility of the Institute for Reference Materials and Measurements of the European Commission's Joint Research Centre according to the principles laid down in the technical guidelines of the European Reference Materials® co-operation agreement between BAM-IRMM-LGC. Information on these guidelines is available on the internet (http://www.erm-crm.org).

Accepted as an ERM®, Geel, January 2009.

Signed:

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Joint Research Centre
Institute for Reference Materials and Measurements
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Registration No. 268-TEST
ISO Guide 34 for the
production of reference materials

All following pages are an integral part of the certificate.

Page 1 of 3

DESCRIPTION OF THE SAMPLE

A unit consists of five Charpy V-notch test pieces, which are rectangular steel bars of nominal dimensions 55 mm x 10 mm x 10 mm, with one V-notch, accurately machined to tolerances imposed in EN 10045-2 and ISO 148-3. The five specimens are packed together in a plastic bag filled with oil to prevent oxidation.

ANALYTICAL METHOD USED FOR CERTIFICATION

Charpy pendulum impact tests in accordance with EN 10045-1 and ISO 148-1, using pendulum impact machines with a 2 mm striker tip radius.

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- Universität Stuttgart, Materialprüfungsanstalt, Stuttgart (DE)* (DAP-PL-2907.02)
- VTT, Espoo (FI)

* Measurements within the scope of accreditation to ISO 17025.

SAFETY INFORMATION

Precautions need to be taken to avoid injury of the operator by broken specimens when operating the Charpy impact pendulum.

INSTRUCTIONS FOR USE

Samples of ERM-FA013ba correspond with the '(certified) BCR test pieces' as referred to in EN 10045-2 (Method for the verification of impact testing machines), as well as with the 'certified reference test pieces' as defined in ISO 148-3 (Preparation and characterisation of Charpy V reference test pieces for verification of test machines).

The ERM-FA013ba batch is one of the 'Master Batches'. Master Batch test pieces are not for sale. They are intended solely to traceably certify Secondary Batches of the same nominal absorbed energy (here 30 J). The certified value and its associated uncertainty of the Master Batch are used in the calculation of the certified value and combined and expanded uncertainty of a set of 5 specimens from a Secondary Batch.

When characterising a secondary batch, a number of Master Batch test pieces are broken under repeatability conditions together with a selection of samples from the secondary batch. 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 cleaning and conditioning procedure is considered to be 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 technically pure 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 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 (20 ± 2 °C). To assure thermal equilibrium is reached, move the specimens to the test laboratory at least 3 h before the tests.

After cleaning and equilibration, the samples need to be broken with a pendulum impact test machine operated in accordance with EN 10045-1 or ISO 148-1 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 related 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.

STORAGE

Specimens should be kept at room temperature in their original packing until used. However, the European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

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NOTE

A detailed technical report can be obtained from the Joint Research Centre, Institute for Reference Materials and Measurements on request.

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