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The certification of the absorbed energy (120 J nominal) of Charpy V-notch reference test pieces for tests at 20 °C: ERM®-FA016bg

Certified Reference Material ERM®-FA016bg

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CERTIFICATION REPORT

The certification of the absorbed energy (120 J nominal) of Charpy V-notch reference test pieces for tests at 20 °C: ERM®-FA016bg

Certified Reference Material ERM®-FA016bg

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Summary

This certification report describes the processing and characterisation of ERM®-FA016bg, a batch of Charpy V-notch certified reference test pieces certified for the absorbed energy *KV* (= energy required to break a V-notched test piece using a pendulum impact test machine). 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 (or *method* 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®- FA016bg is made traceable to the SI, via the SI-traceable certified value of the master batch ERM®-FA016ax, by testing samples of ERM®-FA016bg and ERM®-FA016ax 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* 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)	117	5

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®-FA016bg, and its uncertainty, are traceable to the International System of Units (SI), via the master batch ERM®-FA016ax of the same nominal absorbed energy (120 J) by testing samples of ERM®-FA016ax and ERM®-FA016bg 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 the 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, ν_{RM} = 55.

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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.

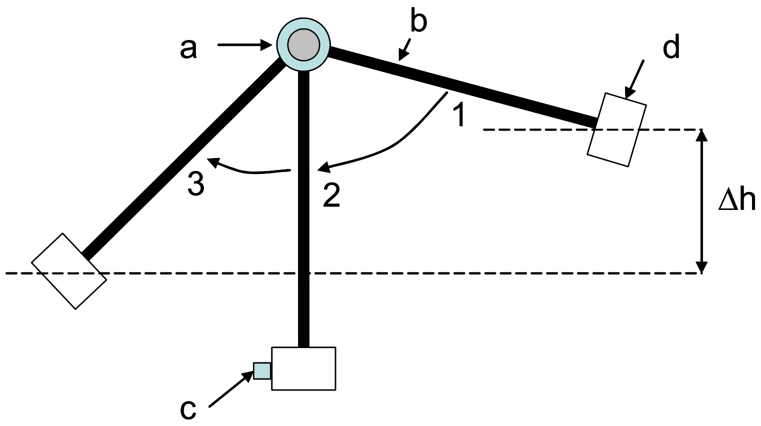

Figure 1: Schematic presentation of the Charpy pendulum impact test, showing a: the horizontal rotation axis of the pendulum, b: the stiff shaft on to which is fixed d: the hammer. The hammer is released from a well-defined height (position 1). When the hammer has reached maximum kinetic energy (shaft 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.

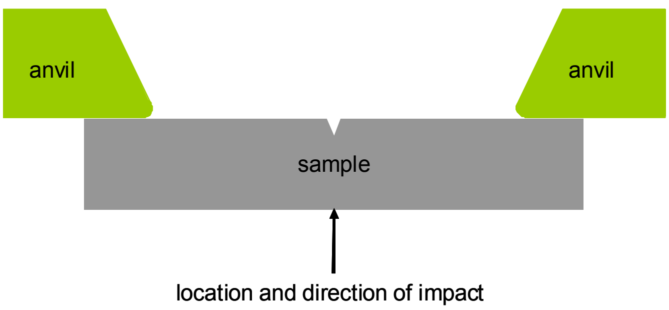

Figure 2: Schematic drawing of a V-notched Charpy test piece (top-view), indicating the place and direction of impact.

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 now allows a difference of 20 % (if

$KV_{MB} \geq$ 40 J) or 8 J (if KV_{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 5.3). 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®-FA016bg) test pieces was carried out by the Laboratoire national de métrologie et d'essais (LNE), Trappes (FR), using steel bars produced at Aubert & Duval (FR).
The MB samples (ERM®-FA016ax) 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 LNE 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®-FA016bg test pieces were prepared from AISI 4340 steel. The steel was cast and rolled into bars at Aubert & Duval (FR) (see section 3.1). Production of the test pieces from these bars was performed under the supervision of LNE (see sections 3.2, 3.3, 3.4 and 3.5).

3.1 Processing of hot-rolled bars

The base material consisted of AISI 4340 steel. To limit the amount of impurities potentially affecting the homogeneity of the fracture resistance, the following compositional tolerances specified in Table 1 were imposed on the selected steel batch. These tolerances are stricter than generally allowed for AISI 4340 steel.

Table 1: Adapted composition tolerances of AISI 4340

Composition (mass fraction, in g/kg)						
C	S	P	Si	Mn	Cr	Ni
1.1 – 1.3	< 0.03	<0.18	1.5 – 3	7.5 – 9	112.5 – 116.5	25.5 – 27.5
Mo	Cu	Al	V	W	N	
15.5 – 17	< 2	< 0.1	2.5 – 3	< 1	0.25 - 0.4	

The ingot was hot rolled, resulting in bars that were 6 m long and with a squared cross-section of 12 mm x12 mm. For the ERM®-FA016bg batch, steel was used from ingot number HS360303.

3.2 Heat treatment of hot-rolled bars

The heat treatment of the hot-rolled bars was performed at Aubert & Duval. The 15 bars for ERM®-FA016bg were heat-treated together. 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] at 850 °C for 30 min. From this furnace, the bars were quenched into oil at 40 °C. After the oil-quench, the samples were annealed in a furnace of 'class 5 °C' at 630 °C for 120 min. After this annealing treatment, the samples were cooled down in air.

3.3 Machining of Charpy test pieces

After heat treatment, a limited number of samples (5) were machined for a preliminary check of the obtained energy level. Results indicated an average *KV* (116.2 J) which is within the desired energy range (112.5 J to 127.5 J).
The samples were machined to dimensional tolerances imposed in ISO 148-3 [9]. The batch code (indicating the nominal energy level and the letter code assigned to the batch) and the individual sample code (indicating the bar and the sequence in the bar from which the sample originates) were engraved twice on one long face of each sample, on both sides of the notch, for fully traceable sample coding also after fracture. The V-notch was introduced using an electro-erosion tool.

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 April 15, 2013 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

[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.

longitudinal axis of the test piece 27.5 ± 0.2 mm). All samples were within the criteria specified in ISO 148-3.
The 25 samples checked for geometrical compliance were impact tested on the Tinius Olsen, model 74 impact (serial number 126 385) pendulum - which is one of the French reference pendulums - at LNE on April 15, 2013. The results are reported in the certificate LNE n° L011137/DE/5 and [10]. The average *KV* of the 25 samples was 116.82 J, which is within the desired energy range (112.5 J to 127.5 J). The standard deviation of the test results (s = 4.52 J, *RSD* = 3.87 %) was acceptable. The variation was checked again during the characterisation tests at IRMM (see section 6).

3.5 Packaging and storage

Finally, the samples were cleaned and packed in sets of 5, in oil-filled and closed plastic bags. These oil-filled bags, together with a label, again were packed in a sealed plastic bag, and shipped to IRMM. After arrival (June 3, 2013) the 1445 samples (or 289 sets) of ERM®-FA016bg were registered and stored at (18 ± 5) °C, 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] specifies 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 during the characterisation tests at IRMM on August 7, 2013 (s_h = 4.39 J). This leads to $u_h = \frac{s_h}{\sqrt{5}} = 2.02$ J (1.67 %).

As is required for a homogeneity test, the samples were randomly selected from the whole batch. The number of samples tested (30) is sufficiently large to reflect the homogeneity of the full SB (1445 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 0.05 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.

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.

In the meanwhile 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®-FA016bg was characterised in August, 2013, the validity of the certificate expires in August, 2023.

6 Characterisation

6.1 Characterisation tests

30 samples from ERM®-FA016bg (sets 41, 62, 105, 125, 163 and 282) were tested under conditions of intermediate precision with 25 samples from MB ERM®-FA016ax (sets 8, 104, 191, 223 and 247), 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 August 7, 2013 (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.

The accepted data obtained on individual test pieces are shown in Figure 3 and in Annex 1. The results of the measurements are summarised in Table 2

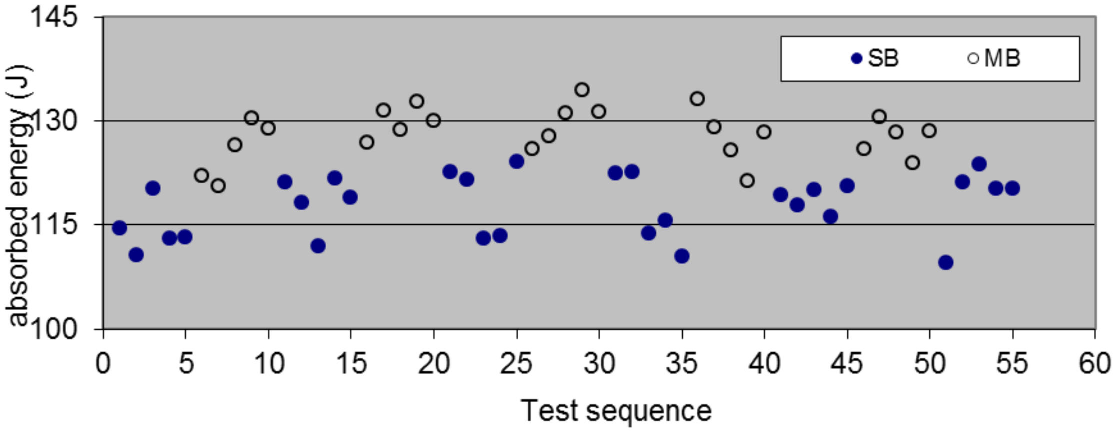

Figure 3: Absorbed energy values of 25 test pieces of ERM®-FA016ax, compared with 30 test pieces of ERM®-FA016bg; data are displayed in the actual test sequence

Table 2: Characterisation measurements of Batch ERM®-FA016bg

	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®-FA016ax (MB)	25	128.17	3.58	2.80
ERM®-FA016bg (SB)	30	117.76	4.39	3.73

The SB-results meet the ISO 148-3 acceptance criteria for a batch of reference materials (RSD_{SB} < 5 %). Also the difference between $\overline{X}_{MB}$ and $\overline{X}_{SB}$, the indicator used to assess the similarity of master batch and secondary batch behaviour, is smaller than the allowed 20 % J (see Section 1.2.2).

6.2 Data from Master Batch ERM®-FA016ax

To calculate KV_{CRM} for ERM®-FA016bg one needs KV_{MB} of the MB used, i.e. ERM®-FA016ax. Table 3 shows the main MB-data, taken from the Certificate of Analysis of ERM®-FA016ax (Annex 2).

Table 3: Data from the certification of Master Batch ERM®-FA016ax

	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®-FA016ax	126.82	0.93	0.73

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} = 117 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:

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

$\overline{X}_{\mathrm{SB}}$ and $\overline{X}_{\mathrm{MB}}$ were obtained under repeatability conditions. Therefore, the uncertainty of the ratio $\overline{X}_{\mathrm{SB}} / \overline{X}_{\mathrm{MB}}$ is not affected by the contributions from reproducibility and bias of the pendulum used to compare MB and SB. Table 4 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 for u_{char} is obtained using the Welch-Satterthwaite equation [6].

Table 4: Uncertainty budget for u_{char} for ERM®-FA016bg

FA016bg	source of uncertainty	measured value (J)	standard uncertainty (J)	probability distribution	divisor	sensitivity coefficient	relative uncertainty (%)	degrees of freedom
KV_{MB}	Certification of MB	126.82	0.93	normal	1	1	0.73	13
$\overline{X}_{\mathrm{SB}}$	comparison of SB and MB in repeatability conditions	117.76	0.80	normal	1	1	0.68	29
$\overline{X}_{\mathrm{MB}}$		128.17	0.72	normal	1	1	0.56	24
						$u_{\mathrm{char,rel}}$ (%)	1.15	51
						u_{char} (J)	1.34	

7 Value assignment

7.1 Certified value, combined and expanded uncertainty

As shown in 6.3, KV_{CRM} = 117 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 5).

The relevant number of degrees of freedom calculated using the Welch-Satterthwaite equation [6], is sufficiently large (ν_{RM} = 55) 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} = 4.1 %) than the verification criterion of 10 % (for industrial pendulums [1]) or even 5 % (for reference pendulums [9]). Rounding the absolute value (in J) of U_{CRM} in accordance with the IRMM procedure leads to a final value of U_{CRM} = 5 J.

Table 5: Uncertainty budget of KV_{CRM} for ERM®-FA016bg

FA016bg	source of uncertainty	relative value u_i (%)		degrees of freedom
u_{char}	characterisation of SB	1.15		51
u_h	homogeneity of SB	1.67		29
Combined standard uncertainty, u_{CRM} (%)			2.03	55
Combined standard uncertainty, u_{CRM} (J)			2.36	
Expanded Uncertainty, k = 2, U_{CRM} (%)			4.05	
Expanded Uncertainty, k = 2, U_{CRM} (J)			5	

8 Metrological traceability and commutability

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®-FA016ax 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®-FA016bg 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®-FA016bg is traceable to the International System of Units (SI) via the corresponding Master Batch ERM®-FA016ax of the same nominal absorbed energy (120 J). Absorbed energy *KV* is a method-specific value, and can only be obtained by following the procedures specified in ISO 148-1 [2].

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 [11].

9 Instructions for use

9.1 Safety information

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

9.2 Intended use

Samples of ERM®-FA016bg 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 148-1 [2].

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.

9.3 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. Submerge the samples in ethanol for about 5 min. 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.

9.4 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.

9.5 Storage

Specimens should be kept at room temperature (18 ± 5) °C in their original packing until used. 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.

Acknowledgements

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Annex 1

Results of characterisation measurements of ERM®-FA016bg as measured according to ISO 148-1 at IRMM, August 07, 2013.

	Master Batch ERM®-FA016ax	**Secondary Batch ERM®-FA016bg**
	***KV* (J)**	***KV* (J)**
1	122.14	114.58
2	120.58	110.72
3	126.43	120.20
4	130.35	113.03
5	128.98	113.23
6	126.82	121.17
7	131.52	118.25
8	128.78	111.88
9	132.70	121.75
10	129.96	119.03
11	126.02	122.73
12	127.85	121.56
13	131.13	113.03
14	134.47	113.42
15	131.33	124.09
16	133.09	122.53
17	129.17	122.73
18	125.85	113.80
19	121.36	115.54
20	128.39	110.53
21	126.04	119.27
22	130.54	117.87
23	128.39	120.00
24	123.89	116.12
25	128.58	120.58
26		109.57
27		121.24
28		123.77
29		120.27
30		120.27
Mean (J)	**128.17**	**117.76**
Standard deviation (J)	**3.58**	**4.39**
***RSD* (%)**	**2.80**	**3.73**

Annex 2

Institute for Reference Materials and Measurements

ERM European Reference Materials

CERTIFICATE OF ANALYSIS

ERM®- FA016ax

STEEL		
	Impact toughness	
	Certified value [2)] [J]	Uncertainty [3)] [J]
Absorbed energy (*KV*) [1)]	126.82	0.93

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 14 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-FA016ax, estimated as the standard deviation of the mean of the 14 laboratory mean values, corresponding with a confidence level of about 68 %.

This certificate is valid until January 2018.

NOTE

European Reference Material ERM®-FA016ax 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
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JRC EUROPEAN COMMISSION

BELAC Registration No. 268-TEST
ISO Guide 34 for the
production of reference materials

All following pages are an integral part of the certificate.

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.

PARTICIPANTS

- Aubert&Duval, Les Ancizes and Gennevilliers (FR)
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- Laboratoire National de Métrologie et d'Essais, Charpy Laboratory, Trappes (FR)* (COFRAC SMH 2-1287)
- National Institute of Standards and Technology (NIST), Materials Reliability Division, Boulder, USA
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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-FA016ax 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-FA016ax 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. Because the certified value of the Master Batch, and its uncertainty, are intermediate values, they have not been rounded according to normal rounding procedures. Instead one additional digit is preserved.

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

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European Commission

EUR 26133 EN – Joint Research Centre – Institute for Reference Materials and Measurements

Title: The certification of the absorbed energy (120 J nominal) of Charpy V-notch reference test pieces for tests at 20°C: ERM®-FA016bg

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Abstract

This certification report describes the processing and characterisation of ERM®-FA016bg, a batch of Charpy V-notch certified reference test pieces certified for the absorbed energy *KV* (= energy required to break a V-notched test piece using a pendulum impact test machine). 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).

The absorbed energy (*KV*) is *procedurally* defined (or *method* 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®- FA016bg is made traceable to the SI, via the SI-traceable certified value of the master batch ERM®-FA016ax, by testing samples of ERM®- FA016bg and ERM®-FA016ax 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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