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CERTIFICATION REPORT

Certification of Charpy V-notch Reference Test Pieces of 120 J Nominal Absorbed Energy

Certified Reference Materials ERM®-FA016aw and ERM®-FA016az

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Certification of Charpy V-notch Reference Test Pieces of 120 J Nominal Absorbed Energy

Certified Reference Materials ERM®-FA016aw and ERM®-FA016az

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Summary

This certification report describes the processing and characterisation of ERM®-FA016aw and ERM®-FA016az, two batches of Charpy V-notch certified reference test pieces. Sets of five of these test pieces are used for the verification of pendulum impact test machines according to EN 10045-2 (Charpy impact test on metallic materials, Part 2. Method for the verification of impact testing machines [1]) or according to ISO 148-2 (Metallic materials - Charpy pendulum impact test – Part 2: Verification of test machines [2]). The certified values for *KV* (absorbed energy = energy required to break a V-notched test piece using a pendulum impact test machine) are 125.7 J for ERM®-FA016aw and 121.6 J for ERM®-FA016az. The associated expanded uncertainties (4.0 J for ERM®-FA016aw and 4.3 J for ERM®-FA016az, $k = 2$ corresponding to a confidence level of 95 %) are calculated for the mean of a set of five test pieces. The certified values are traceable to the Charpy impact test method as described in EN 10045-1 [3] and ISO 148 [4], via the corresponding master batch ERM®-FA016c of the same nominal absorbed energy (120 J).

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Glossary

AISI	American Iron and Steel Institute
ASTM	American Society for Testing and Materials
BCR	Community Bureau of Reference
CEN	European Committee for Standardization
CRM	Certified Reference Material
EC	European Commission
EN	European Norm
Eq.	Equation
ERM®	European Reference Material trademark
g	Gravitation acceleration
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
m	Mass of pendulum
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
RSD_{MB}	Relative standard deviation of the n_{MB} results of the samples of the master batch tested for the certification of the secondary batch
RSD_{SB}	Relative standard deviation of the n_{SB} results of the samples of the secondary batch tested for its characterisation
s	Standard deviation
SB	Secondary Batch
s_{h}	Standard deviation of the results of the samples of the secondary batch tested to assess its homogeneity
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 of the secondary batch tested for its characterisation
u_{CRM}	Combined standard uncertainty of KV_{CRM}
U_{CRM}	Expanded uncertainty (k = 2, confidence level 95 %) of KV_{CRM}
u_{char}	Standard uncertainty of the result of the characterisation tests
u_{h}	Standard uncertainty component from homogeneity
u_i	Standard uncertainty component corresponding to effect i
u_{MB}	Standard uncertainty of KV_{MB}
$\overline{X}_{\mathrm{MB}}$	Mean KV value of the n_{MB} measurements on samples of the master batch tested when characterising the secondary batch
$\overline{X}_{\mathrm{SB}}$	Mean KV value of the n_{SB} results of the samples of the secondary batch tested for its characterisation
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
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 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.

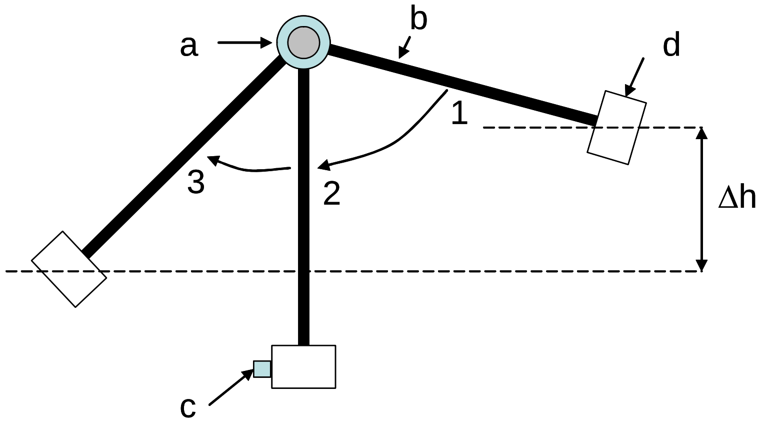


Figure 1: Schematic presentation of the Charpy pendulum impact test, showing a: the horizontal rotation axis of the pendulum, b: the stiff shaft onto which is fixed d: the hammer, of mass m. The hammer is released from a defined height (position 1). The hammer strikes c: the test sample, when the hammer 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 with a difference in potential energy (= $m \times g \times \Delta h$, with g = gravitation 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 shape of well-defined geometry [1, 2], schematically shown in Figure 2.

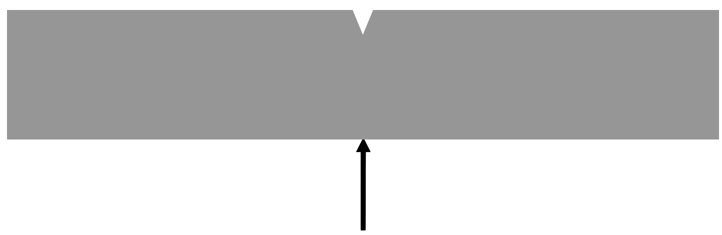


Figure 2: Schematic drawing of a V-notched Charpy sample (top-view when sample is in place for test), indicating the place and direction of impact.

2 The certification concept of master batch and secondary batch

2.1 Difference between master and secondary batches

The BCR reports by Marchandise et al. [6] and Varma [7] provide details of the certification of the 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 Joint Research Centre (JRC) of the European Commission (EC). The work was performed in accordance with procedures described in the BCR reports [6] and [7]. 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.

Since the uncertainty of the certified value of the MB contributes to the uncertainty of the certified value of the SB, the latter is necessarily larger than the former. Nevertheless, as will be shown also in this report, the uncertainty can be kept sufficiently small to meet the requirements of the intended use of the certified reference material (CRM). Avoiding the need for an international interlaboratory comparison for each produced batch, the MB-SB approach allows cost-efficient production of certified reference test pieces.

The BCR reports [6] and [7] 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 to Expression of Uncertainty in Measurement [8]. This revised approach was developed and presented by Ingelbrecht *et al.* [9, 10] and is summarised below.

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 [10]:

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

For this approach to be reliable, the pendulum used for the tests on MB and SB in repeatability conditions, must be well performing. This can be checked by comparing the certified value of the MB, KV_{MB}, with the results obtained on the MB samples when comparing SB and MB, $\overline{X}_{\mathrm{MB}}$. IRMM allows a difference of 5 % (if $KV_{\mathrm{MB}} > 40$ J) or 2 J (if $KV_{\mathrm{MB}} < 40$ J) between KV_{MB} and $\overline{X}_{\mathrm{MB}}$, corresponding with the level of bias allowed for reference pendulums specified in EN 10045-2 [1] and ISO 148-3 [11].

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 steel with nominally the same chemical composition, and similar heat treatments. These precautions have to result in a ratio $\frac{KV_{\mathrm{CRM}}}{KV_{\mathrm{MB}}}$ close to 1. IRMM allows a difference of 20 % ($KV_{\mathrm{MB}} > 40$ J) or 8 J ($KV_{\mathrm{MB}} < 40$ J) between KV_{CRM} and KV_{MB}.

3 Participants

The processing of the SB test pieces was carried out by the Laboratoire National de Métrologie et d'Essais (LNE), Trappes (FR), using steel bars produced at Cogne Acciai Speciali, Aosta (IT). The MB samples used in the characterisation of the SB were provided by IRMM, Geel (BE). Characterisation of the SB was carried out at IRMM using a pendulum verified according to the criteria imposed by EN 10045-2 [1] and ISO 148-2 [2]. Data evaluation was performed at IRMM.

4 Processing

The ERM®-FA016aw and ERM®-FA016az test pieces were prepared from AISI 4340 steel. The steel was cast and rolled into bars at Cogne Acciai Speciali (see section 4.1). Production of the test pieces from these bars was performed under the supervision of LNE (see Sections 4.2, 4.3, 4.4, and 4.5).

4.1 Processing of hot-rolled bars

The base material consisted of AISI 4340 steel, produced at Cogne Acciai Speciali. 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 (in mass %), which is stricter than generally allowed for AISI 4340. The ingot was hot rolled, resulting in bars that were 4 m long and with a squared cross-section of 11.5 mm. For the ERM®-FA016aw and ERM®-FA016az batches, steel was used from ingot number 960133, billet I. A billet is a semi-finished hot-rolled product, in this case of cross-section 108.5 mm, which is between the ingot (560 mm cross-section) stage and the final required bars (11.5 mm cross-section). A full description of the processing and quality check of the steel bars is available in [12].

4.2 Heat treatment of hot-rolled bars

The heat treatment of the hot-rolled bars was performed at Aubert & Duval, Gennevilliers (FR). For each batch 22 bars 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 minutes. From this furnace, the bars were quenched into oil at 40 °C. After the oil-quench, the samples were annealed in a second furnace ('class 5 °C') at 630 °C for 120 minutes. After this annealing treatment, the samples were cooled down in air.

[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.

After heat treatment, a limited number of samples (5 or 8) were machined for a preliminary check of the obtained energy level. Results obtained at Aubert & Duval indicated average *KV*-levels (113.2 J for ERM®-FA016aw and 111.8 J ERM®-FA016az) close to the desired nominal energy level (120 J).

4.3 Machining of Charpy test pieces

After the heat treatment the samples were machined to dimensional tolerances imposed in EN 10045-2 [1] and ISO 148-3 [11]. The batch code (e.g. AW120 with '120' indicating the nominal absorbed energy level (120 J) and 'AW' 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 twice on the top face of the sample, once on both sides of the notch. The V-notch was introduced using an electro-erosion tool.

4.4 Quality control

When all samples from a batch were fully machined, a randomised selection of 25 samples was made. The dimensions of the 25 samples were checked on April 2, 2007 (ERM®-FA016aw) and on September 10, 2007 (ERM®-FA016az) 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.

The samples checked for geometrical compliance were impact tested on April 3, 2007 (ERM®-FA016aw) and on September 11, 2007 (ERM®-FA016az) on the Tinius Olsen 358 Joules pendulum - which is one of the French reference pendulums - at LNE. The results are reported in certificates LNE No. F031180/CQPE/3 [13] and F120699/CQPE/4 [14]. The average KV of the 25 samples was 125.7 J for ERM®-FA016aw and 122.0 J for ERM®-FA016az, sufficiently close to the target value (120 J) and to the certified value of the master batch (see Section 2.2). The standard deviation of the test results (s = 3.5 J, RSD = 2.8 % for ERM®-FA016aw and s = 3.5 J, RSD = 2.9 % for ERM®-FA016az) was smaller than the maximum allowed 3 %. The variation was checked again during the certification tests at IRMM (see Section 5).

4.5 Packaging and storage

Finally, the samples were packed in sets of 5, in oil-filled and closed plastic bags. The samples are closely packed in the bag to eliminate the possibility that the corners or edges of one bar scratch the other bars. These oil-filled bags, together with a label, again were packed in a sealed plastic bag, and shipped to IRMM. After arrival (May, 2007 for ERM®-FA016aw and November, 2007 for ERM®-FA016az), the 255 sets of ERM®-FA016aw samples and the 260 sets of ERM®-FA016az samples were registered and stored at room temperature, pending further characterisation, certification and distribution.

5 Characterisation

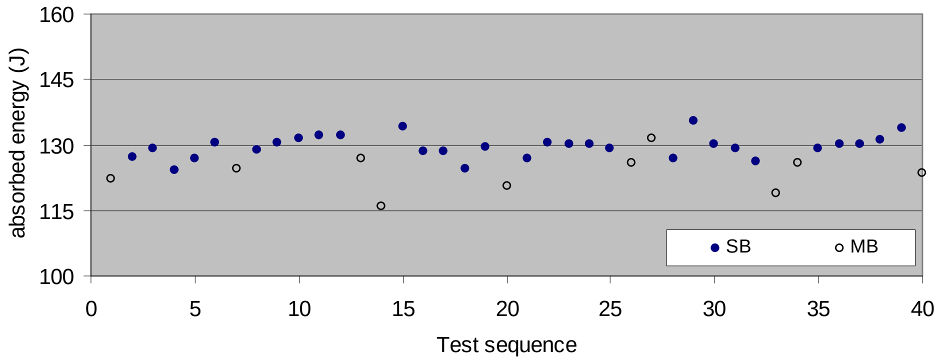
5.1 Characterisation tests

30 samples from ERM®-FA016aw (sets 1, 60, 135, 201, 239 and 255) and 30 samples from ERM®-FA016az (sets 1, 74, 128, 180, 231 and 258) were tested under repeatability conditions with 10 samples[1] from MB ERM®-FA016c (sets 61 and 62), using the Instron Wolpert PW 30 (serial number 7300 H1527) machine of IRMM, an impact pendulum yearly verified according to procedures described in EN 10045-2 [1] and ISO 148-2 [2]. Tests were performed on April 23, 2008 (laboratory temperature 20.3 – 20.7 °C), in accordance with EN 10045-1 and ISO 148. The measured absorbed energy values were corrected for friction and windage losses. Data obtained on individual test pieces are shown in Figure 3 a) and b) and in Annex 1. The results of the measurements are summarised in Table 1.

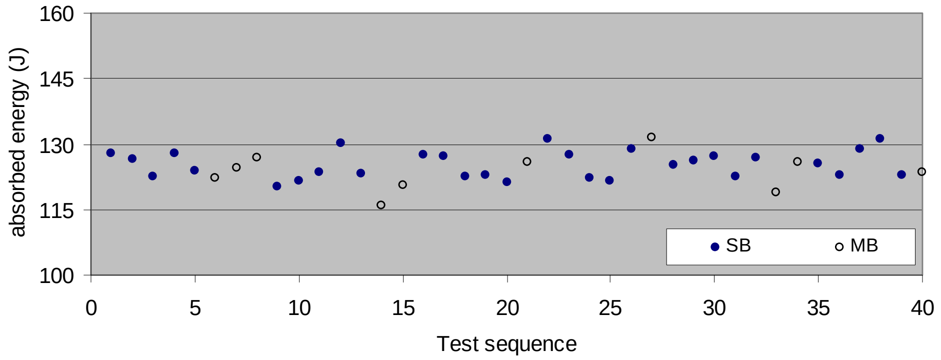
Table 1: Characterisation measurements of Batches ERM®-FA016aw and ERM®-FA016az.

	Number of test pieces	**Mean value**	**Standard deviation**	**Relative standard deviation**
	n_{MB} , n_{SB}	$\overline{X}_{MB}$, $\overline{X}_{SB}$ [J]	s_{MB} , s_{SB} [J]	RSD_{SB}, RSD_{MB} [%]
ERM®-FA016c (MB)	10	123.58	4.39	3.55
ERM®-FA016aw (SB)	30	129.57	2.59	2.00
ERM®-FA016az (SB)	30	125.31	3.11	2.48

[1] The number of MB samples (10) was lower than in previous SB-MB characterisation exercises (25); see also Annex 2, Instructions for use. However, the good homogeneity of the ERM®-FA016aw and ERM®-FA016az batches justifies the lower number of MB samples, as the resulting expanded uncertainty of the certified value (see Section 8.4) is not larger than for previous Secondary Batches.



a)



b)

Figure 3: Absorbed energy values of 10 test pieces of ERM®-FA016c, compared with a) 30 test pieces of ERM®-FA016aw and b) with 30 test pieces of ERM®-FA016az; data are displayed in the actual test sequence.

The relative standard deviations of the 30 SB-results (2.00 % for ERM®-FA016aw and 2.48 % for ERM®-FA016az) meet the EN 10045-2 [1] and ISO 148-3 [11] acceptance criteria for a batch of reference materials (RSD_{SB} < 5 %), as well as the more stringent acceptance criterion (RSD_{SB} < 3 %) contractually fixed between IRMM and its sample supplier. 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 % (see Section 2.2).

5.2 Data from master batch ERM®-FA016c

To calculate KV_{CRM} for ERM®-FA016aw and for ERM®-FA016az one needs KV_{MB} of the MB used, i.e. ERM®-FA016c. Table 2 shows the main MB-data, taken from the Certificate of Analysis of ERM®-FA016c (Annex 2), which is the revised, ERM-version of the originally issued certificate, based on the certification report of the MB [6]. The certified value was obtained from an interlaboratory comparison with 14 laboratories.

The values KV_{MB} (Table 2) and $\overline{X}_{MB}$ (Table 1) are less than 5 % different, confirming that the pendulum used for the characterisation of the secondary batch is functioning with a sufficiently low bias (see Section 2.2).

Table 2: Data from the certification of master batch ERM®-FA016c [6].

	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} [%]
ERM®-FA016c	119.9	0.6	0.5

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

From the data in Table 1 and Table 2, and using Eq. 1, one readily obtains that KV_{CRM} = 125.7 J for ERM®-FA016aw and 121.6 J for ERM®-FA016az (rounding in accordance with uncertainty; see Table 4). The relative uncertainty contribution associated with the characterisation of the SB, u_{char}, is assessed as in Eq. 2 [10], which sums the relative uncertainties of the three factors appearing 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 3 summarises the input quantities of the u_{char} uncertainty budget, their respective statistical properties, and shows how they were combined. The standard uncertainty of KV_{MB} is taken from Table 2, the standard uncertainty values of $\overline{X}_{\mathrm{SB}}$ and $\overline{X}_{\mathrm{MB}}$ are calculated as the ratio of their standard deviation to the square root of the number of samples tested (from Table 1). The effective number of degrees of freedom for u_{char} is obtained using the Welch-Satterthwaite equation [8].

Table 3: Uncertaity budgets for uchar for ERM®-FA016aw and for ERM®-FA016az

FA016 aw	measured value [J]	source of uncertainty	standard uncertainty value [J]	probability distribution	divisor [1]	sensitivity coefficient [2]	relative standard uncertainty [%]	degrees of freedom
KV_{MB}	119.9	certification of MB	0.6	normal	1	1	0.50	13
$\overline{X}_{SB}$	129.57	comparison of SB and MB under repeatability conditions	0.47	normal	1	1	0.36	29
$\overline{X}_{MB}$	123.58		1.39	normal	1	1	1.12	9
						u_{char} (%)	1.28	14
						u_{char} (J)	1.61	

FA016 az	measured value [J]	source of uncertainty	standard uncertainty value [J]	probability distribution	divisor [1]	sensitivity coefficient [2]	relative standard uncertainty [%]	degrees of freedom
KV_{MB}	119.9	certification of MB	0.6	normal	1	1	0.50	13
$\overline{X}_{SB}$	125.31	comparison of SB and MB under repeatability conditions	0.57	normal	1	1	0.45	29
$\overline{X}_{MB}$	123.58		1.39	normal	1	1	1.12	9
						u_{char} (%)	1.31	16
						u_{char} (J)	1.59	

[1] Divisor: number used to calculate standard uncertainty from non-standard-uncertainty expression of uncertainty (e.g.: coverage factor to adapt expanded uncertainty to standard uncertainty, or factor to transform bounds of rectangular distribution into standard uncertainty of equivalent normal distribution).
[2] Sensitivity coefficient: used to multiply an input quantity to express it in terms of the output quantity.

6 Homogeneity

The test pieces constituting a CRM unit are sampled from the SB, which is sufficiently homogeneous (s_{SB} < 5 %, as required in EN 10045-2 [1] and ISO 148-3 [11]), but not perfectly homogeneous. Therefore, as for most reference materials, a separate homogeneity contribution u_h to the uncertainty of the certified value is required. Here, u_h is estimated from s_h, the standard deviation (2.59 J for ERM®-FA016aw and 3.11 J for ERM®-FA016az) of the results obtained at IRMM on April 23, 2008. As is required for a homogeneity test, the samples were randomly selected from the whole batch. The number of samples tested (30) is largely sufficient to reflect the homogeneity of the full SB (1275 samples for ERM®-FA016aw and 1300 samples for ERM®-FA016az).

The effect of s_h on the uncertainty of the certified value depends on the number of samples over which the *KV*-value is averaged. EN 10045-2 [1] and in ISO 148-2 [2] specify that pendulum 'indirect verification' with CRMs must be performed using 5 test pieces. Therefore, a CRM-unit consists of 5 test pieces, and $u_h = \frac{s_h}{\sqrt{5}} = 1.16$ J for ERM®-FA016aw and $u_h = \frac{s_h}{\sqrt{5}} = 1.39$ J for ERM®-FA016az. u_h is probably a slight overestimation, since it contains also the repeatability of the instrument. However, the latter cannot be separated from u_h or separately measured.

7 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-)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.

Given the large sample-to-sample heterogeneity, the ageing effects are difficult to detect when testing limited numbers of samples. Dedicated efforts have therefore been spent to quantify the stability of the certified values of batches of Charpy CRMs. 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 over a period of 1.5 years, even with exposure to 90 ^{o}C [15]. 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 recently, during the International master batch (IMB) project [16]. 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 insignificant. Nevertheless, until 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 batches ERM®-FA016aw and ERM®-FA016az were characterised in April, 2008, the validity of the certificate stretches until April, 2018. This validity may be extended as further evidence of stability becomes available.

8 Evaluation of results

8.1 Calculation of certified value, combined and expanded uncertainty

As shown in Section 5.3, KV_{CRM} = 125.7 J for ERM®-FA016aw and KV_{CRM} = 121.6 J for ERM®-FA016az. 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. The absolute values of these contributions are quadratically summed, and the approach is summarised in the uncertainty budget shown in Table 4.

The relevant number of degrees of freedom calculated using the Welch-Satterthwaite equation [8], is sufficiently large (ν_{eff} = 28 for ERM®-FA016aw and ν_{eff} = 37 for ERM®-FA016az) 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 (3.2 %) than the verification criterion of 10 % (for industrial pendulums [1, 2]) or even 5 % (for reference pendulums [1, 11]).

Table 4: Uncertainty budgets of KV_{CRM} for ERM®-FA016aw and for ERM®-FA016az.

FA016aw	source of uncertainty	absolute value [J]	divisor [1]	Sensitivity coefficient[2]	u_i [J]	degrees of freedom
u_{char}	characterisation of SB	1.61	1	1	1.61	14
u_h	homogeneity of SB	1.16	1	1	1.16	29
combined standard uncertainty, u_{CRM}					1.99	28
expanded uncertainty, k = 2, U_{CRM}					4.0	

FA016az	source of uncertainty	absolute value [J]	divisor [1]	Sensitivity coefficient[2]	u_i [J]	degrees of freedom
u_{char}	characterisation of SB	1.59	1	1	1.59	16
u_h	homogeneity of SB	1.39	1	1	1.39	29
combined standard uncertainty, u_{CRM}					2.12	37
expanded uncertainty, k = 2, U_{CRM}					4.3	

[1] Divisor: number used to calculate standard uncertainty from non-standard-uncertainty expression of uncertainty (e.g.: coverage factor to adapt expanded uncertainty to standard uncertainty, or factor to transform bounds of rectangular distribution into standard uncertainty of equivalent normal distribution).

[2] Sensitivity coefficient: used to multiply an input quantity to express it in terms of the output quantity.

8.2 Traceability

The absorbed energy *KV* is a method-specific value, and can only be obtained by following the procedures specified in EN 10045-1 [3] and ISO 148 [4]. The certified value of the MB ERM®-FA016c is traceable to these standard procedures as it was 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 ERM®-FA016aw and of ERM®-FA016az are made traceable to the certified value of the MB using tests on SB and MB samples under repeatability conditions. Therefore the certified values of ERM®-FA016aw and of ERM®-FA016az are traceable to the Charpy impact test as described in EN 10045-1 [3] and ISO 148 [4].

8.3 Commutability

During the certification of the MB, 14 different pendulums were used, each equipped with a ISO-type striker of 2 mm tip radius [6]. 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.

8.4 Summary of results

The certified value and associated uncertainties are summarised in Table 5.

Table 5: Certified values and associated uncertainties for ERM®-FA016aw and for ERM®-FA016az.

	Certified mean value for set of 5 test pieces KV_{CRM} **[J]**	**Combined standard uncertainty** u_{CRM} **[J]**	**Expanded uncertainty (k = 2)** U_{CRM} **[J]**
ERM®-FA016aw	125.7	1.99	4.0
ERM®-FA016az	121.6	2.12	4.3

9 Instructions for use

9.1 Intended use

Samples of ERM®-FA016aw and of ERM®-FA016az correspond with the ‘(certified) BCR test pieces’ as referred to in EN 10045-2 [1], as well as with the ‘certified reference test pieces’ as defined in ISO 148-3 [11]. 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 EN 10045-2 [1] and ISO 148-2 [2]. The indirect verification provides a punctual assessment of the bias of the user’s Charpy pendulum impact machine.

9.2 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 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 hours before the tests.

9.3 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 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.

The comparison of the indirect verification results with the certified value and uncertainty must be based on the mean of the 5 measured *KV* values, because the calculation of the uncertainty of the certified value is based on this sample size.

10 Acknowledgements

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Annex 1

Results of characterisation measurements of ERM®-FA016aw and ERM®-FA016az as measured according to EN 10045-1 [3] and ISO 148 [4] at IRMM, April 23, 2008.

	master batch **ERM®-FA016c**	**secondary batch** **ERM®-FA016aw**	**secondary batch** **ERM®-FA016az**
	***KV* [J]**	***KV* [J]**	***KV* [J]**
1	122.31	127.05	127.95
2	124.57	129.19	126.39
3	126.78	124.13	122.70
4	116.00	126.85	127.95
5	120.63	130.36	123.86
6	125.75	129.00	120.25
7	131.40	130.56	121.60
8	118.77	131.34	123.54
9	125.95	132.12	130.17
10	123.61	132.32	123.16
11		134.27	127.44
12		128.41	127.12
13		128.41	122.64
14		124.59	122.84
15		129.46	121.09
16		126.73	131.21
17		130.43	127.51
18		130.24	122.25
19		130.24	121.48
20		129.07	128.68
21		126.92	125.17
22		135.32	126.34
23		130.24	127.12
24		129.26	122.64
25		126.34	126.92
26		129.07	125.68
27		130.04	123.03
28		130.04	128.68
29		131.21	131.21
30		133.76	122.71
Mean [J]	**123.58**	**129.57**	**125.31**
Standard deviation [J]	**4.39**	**2.59**	**3.11**
***RSD* [%]**	**3.55**	**2.00**	**2.48**

Annex 2

CERTIFICATE OF ANALYSIS

ERM®- FA016c

CHARPY V test pieces (Master Batch)		
Parameter	Absorbed energy at 20 ± 2 °C	
	Certified value [1] J	Uncertainty [2] J
KV according to EN 10045-1 and ISO 148	119.9	0.6

1) Mean absorbed energy of test pieces from batch ERM FA016c, estimated as the mean of means of absorbed energies measured at 14 laboratories. At each laboratory, 5 test pieces were broken. The certified value is traceable to the Charpy impact test method as described in EN 10045-1 and ISO 148.

2) Standard uncertainty *u* of the certified mean absorbed energy of batch ERM FA016c, estimated as the standard deviation of the mean of the 14 laboratory mean values, corresponding with a level of confidence of about 68 %.

This certificate is valid until September 2009; this validity may be extended as further evidence of stability becomes available.

NOTE

European Reference Material ERM®-FA016c was originally certified as BCR-016 C. It was produced and certified under the responsibility of IRMM 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, September 2004

Signed:

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All following pages are an integral part of the certificate.

Page 1 of 3

DESCRIPTION OF THE SAMPLE

A unit consists of five test pieces, which are rectangular steel bars of nominal 55 mm x 10 mm x 10 mm, with one V-notch, accurately machined to dimensions imposed in EN-10045-1 and ISO 148. The five specimens are packed together in a plastic bag filled with oil to prevent oxidation.

INSTRUCTIONS FOR USE

Samples of ERM FA016c 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 FA016c 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 120 J). The certified value and associated uncertainty of the Master Batch are used in the calculation of the combined uncertainty of the certified value of a set of 5 specimens from a Secondary Batch. Sets of 5 samples of Secondary Batches are distributed as certified reference test pieces for the verification of Charpy impact test machines in accordance with EN 10045-2 and ISO 148-2.

When characterising a secondary batch, (a minimum of) 25 Master Batch test pieces are broken in repeatability conditions together with a selection of samples from the secondary batch. Special attention is drawn to cleaning of the specimens prior to testing. The following procedure is recommended:

1. Wipe excess oil from the specimens with cellulose paper.

2. Immerse the specimens in a clean bath of degreasing solvent for about five minutes.

3. Wipe the specimens with cellulose paper and allow to dry in still air.

4. Before testing, allow the specimens to equilibrate to laboratory temperature for about 12 hours.

After cleaning, the user must avoid touching the specimens with the fingers (wear clean gloves). Vigorous cleaning methods affecting the roughness of the specimen surface or possibly causing deformation/indentation of the specimen edges should be avoided, as this can result in obtaining erroneous data.

Unlike Charpy test pieces of lower nominal impact energies, samples from ERM FA016 batches sometimes leave debris on the Charpy pendulum anvils. Therefore, after each impact, the anvils must be checked and if debris is found, it must be removed.

STORAGE

Specimens should be kept in their original packing until they are used.

SAFETY INFORMATION

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

METHOD USED FOR CERTIFICATION

Charpy pendulum impact tests in accordance with EN 10045-1 and ISO 148.

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NOTE

A detailed technical report of the Master Batch certification project ("Certification of the impact toughness of V-notch Charpy specimens", H. Marchandise, A. Perez-Sainz, E. Colinet, bcr information, XII/323/91-EN, 1991) can be obtained from IRMM on explicit request. In this report, the ERM-FA016c batch is called '120 C' (120 J is the nominal energy of the ERM FA016 batches).

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Abstract

This certification report describes the processing and characterisation of ERM®-FA016aw and ERM®-FA016az, two batches of Charpy V-notch certified reference test pieces. Sets of five of these test pieces are used for the verification of pendulum impact test machines according to EN 10045-2 (Charpy impact test on metallic materials, Part 2. Method for the verification of impact testing machines [1]) or according to ISO 148-2 (Metallic materials - Charpy pendulum impact test – Part 2: Verification of test machines [2]). The certified values for *KV* (absorbed energy = energy required to break a V-notched test piece using a pendulum impact test machine) are 125.7 J for ERM®-FA016aw and 121.6 J for ERM®-FA016az. The associated expanded uncertainties (4.0 J for ERM®-FA016aw and 4.3 J for ERM®-FA016az, $k = 2$ corresponding to a confidence level of 95 %) are calculated for the mean of a set of five test pieces. The certified values are traceable to the Charpy impact test method as described in EN 10045-1 [3] and ISO 148 [4], via the corresponding master batch ERM®-FA016c of the same nominal absorbed energy (120 J).

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