



CERTIFICATION REPORT

**The certification of the absorbed energy
(120 J nominal) of Charpy V-notch reference test pieces for
tests at 20 °C: ERM[®]- FA016bi**



European Commission
Joint Research Centre
Directorate F – Health, Consumers and Reference Materials

Contact information

Reference materials sales
Address: Retieseweg 111, 2440 Geel, Belgium
E-mail: jrc-rm-distribution@ec.europa.eu
Tel.: +32 (0)14 571 705

JRC Science Hub

<https://ec.europa.eu/jrc>

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Abstract

This certification report describes the processing and characterisation of ERM[®]-FA016bi, a batch of Charpy V-notch 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).

The absorbed energy (KV) is operationally defined and refers to the impact energy required to break a V-notch test piece of standardised dimensions, as defined in ISO 148-1. The certified value of ERM[®]-FA016bi is made traceable to the SI, via the SI-traceable certified value of the master batch ERM[®]-FA016ax, by testing samples of ERM[®]-FA016bi 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 and at (20 ± 2) °C.



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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[®] - FA016bi

Ts. Gerganova, G. Roebben, A. Dean, T. Linsinger

European Commission, Joint Research Centre
Directorate F – Health, Consumers and Reference Materials
Geel, Belgium

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Summary

This certification report describes the processing and characterisation of ERM®-FA016bi, a batch of Charpy V-notch 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 operationally defined and refers to the impact energy required to break a V-notch test piece of standardised dimensions, as defined in ISO 148-1 [2]. The certified value of ERM®-FA016bi is made traceable to the SI, via the SI-traceable certified value of the master batch ERM®-FA016ax, by testing samples of ERM®-FA016bi 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 and at (20 ± 2) °C.

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

Steel Charpy V-notched test pieces		
	Certified value ²⁾ [J]	Uncertainty ³⁾ [J]
Absorbed energy (<i>KV</i>) ¹⁾	126	4

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

2) The certified value of ERM®-FA016bi, and its uncertainty, are traceable to the International System of Units (SI), via the master batch ERM®-FA016ax of a similar nominal absorbed energy by testing test pieces of ERM®-FA016ax and ERM®-FA016bi 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 test pieces (delivered as one 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 effective degrees of freedom of the certified uncertainty is $\nu_{RM} = 59$.

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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
ISO	International Organization for Standardization
JRC	Joint Research Centre
k	Coverage factor
KV	Absorbed energy = energy required to break a V-notch 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}
\bar{X}_{MB}	Mean KV value of the n_{MB} measurements on samples of the Master Batch tested when characterising the Secondary Batch
\bar{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.

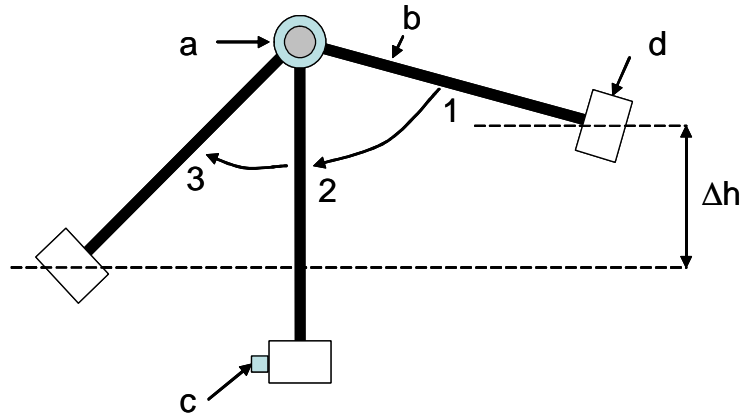


Figure 1: Schematic presentation of the Charpy pendulum impact test, showing a: the horizontal rotation axis of the pendulum, b: the stiff shaft on to which is fixed d: the hammer. The hammer is released from a well-defined height (position 1). When the hammer has reached maximum kinetic energy (shaft in vertical position 2), the hammer strikes c: the test piece, 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 the difference in potential energy, and is a measure of the energy required to break the test piece.

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 of well-defined geometry [2 and 9], schematically shown in Figure 2.

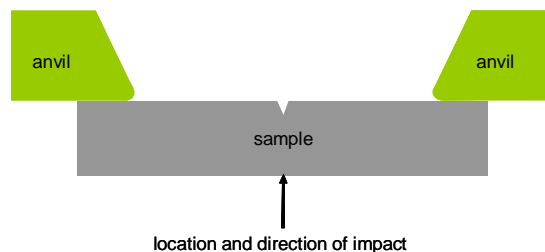


Figure 2: Schematic drawing of a V-notched Charpy test piece (top-view), indicating the place and direction of impact.

1.2 The certification concept of Master Batch and Secondary Batch

1.2.1 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 inter-laboratory comparison.

This report describes the production of a “Secondary Batch” (SB) of Charpy V-notch certified reference test pieces at the JRC-Geel 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] and according to ISO/IEC 17025. 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 from test pieces of the SB (\bar{X}_{SB}) tested on a single pendulum. This value \bar{X}_{SB} has to be corrected for the bias of this particular pendulum. The bias of the pendulum at the moment of testing the test pieces of the SB, is estimated by comparing the mean KV -value of a number of test pieces of the MB (\bar{X}_{MB}), tested together with the SB test pieces under repeatability conditions. The certified value of the MB is used then to calculate KV_{CRM} as follows [8]:

$$K_{V_{CRM}} = \left[\frac{K_{MB}}{\bar{X}_{MB}} \cdot \bar{X}_{SB} \right] \quad \text{Eq. 1}$$

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 $\frac{KV_{MB}}{\bar{X}_{MB}}$ must be close to 1. JRC-Geel allows a difference of 5 % ($KV_{MB} \geq 40$ J) or 2 J ($KV_{MB} < 40$ J) between KV_{MB} and \bar{X}_{MB} , corresponding to the level of bias allowed for reference pendulums specified in ISO 148-3 [9].

Also, the interaction of the pendulum with a SB sample must be similar to the interaction with a MB sample. This is the reason why MB and SB test pieces are made from nominally the same steel. Moreover, it is checked that the ratio $\frac{KV_{CRM}}{KV_{MB}}$ is close to 1. JRC-Geel allows a difference of 25 % ($KV_{MB} \geq 40$ J) or 10 J ($KV_{MB} < 40$ J) between KV_{CRM} and KV_{MB} .

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

The uncertainty of the certified value of the SB is a combination of the uncertainties of the factors in Eq. 1. The MB-SB approach necessarily results in a larger uncertainty of the certified value of SB in comparison with the MB. The additional uncertainty contribution depends on the uncertainty of the ratio $\bar{X}_{MB}/\bar{X}_{SB}$. The measurement uncertainty of the values \bar{X}_{MB} and \bar{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 \bar{X}_{MB} and \bar{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 $\bar{X}_{MB}/\bar{X}_{SB}$, 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[®]-FA016bi) test pieces was carried out by the Laboratoire national de métrologie et d'essais (LNE), using AISI-SAE 4340 steel delivered by Aubert&Duval (FR). The MB test pieces (ERM[®]-FA016ax) used in the characterisation of the SB were provided by JRC-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 and ISO/IEC 17025 [1]. Also the characterisation of the SB was carried out at JRC using a pendulum verified according to 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 JRC. The certification was performed within the scope of an ISO Guide 34 accreditation (BELAC 268-RM).

3 Processing

The ERM[®]-FA016bi test pieces were prepared from bars of AISI-SAE 4340 steel delivered by Aubert&Duval (FR). Production of the test pieces from these bars was performed under the supervision of LNE at FLEURY GF, France as described in [10].

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) [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 x 12 mm. For the ERM[®]-FA016bi batch, steel was used from ingot number HS360303.

3.2 Machining of Charpy test pieces

The ingot was divided into 14 billets. 18 bars from one billet were cut into 1413 Charpy specimens and machined to the dimensional requirements of ISO 148-3 [9] and engraved to ensure identification.

3.3 Heat treatment of hot-rolled bars

The heat treatment of the specimens was performed at Aubert&Duval (FR) in a vacuum-furnace. 1413 specimens of the batch were heat-treated according to the following procedure:

Step 1: austenisation treatment at 850 °C for 85 minutes in a furnace of 'class 10 °C' (the variation of the temperature in the furnace is smaller than 10 °C)

Step 2: cool down in oil

Step 3: annealing treatment at 640 °C for 120 minutes in a furnace of 'class 5 °C' (the variation of the temperature in the furnace is smaller than 5 °C)

Step 4: quenching in neutral gas

3.4 Final machining of Charpy test pieces

After heat treatment, the specimens were machined to the final dimensions specified in ISO 148-3 [9] by LNE. During this process the specimen numbers were engraved on both sides of the notch on one of the long faces of the sample. Finally the specimens were notched using an electro-erosion tool.

3.5 Quality control

When all samples from the batch were fully machined, a selection of 25 test pieces was made. The dimensions of the 25 test pieces were checked 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 $(45 \pm 1)^\circ$, 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 test pieces was outside the intervals specified in ISO 148-3 [9].

The 25 test pieces selected throughout the batch and checked for geometrical compliance were also impact tested using a pendulum type Tinius Olsen model 74 (nominal energy 120 J), verified according to ISO 148-2 [1].

The tests were performed on 11.07.2014. The results are reported in the production report of 11.07.2014 [10]. The average *KV* of the 25 test pieces was 125.8 J, which is within the desired energy interval (112.5 – 127.5 J). The standard deviation of the test results ($s = 2.6$ J, $RSD = 2.1$ %) was below the 4 % maximum allowed by the contract. The variation was checked again during the characterisation tests at JRC-Geel (see Section 6).

3.6 Packaging and storage

Finally, the test pieces were cleaned and packed in sets of 5 randomised test pieces, in oil-filled and vacuum sealed plastic bags. These oil-filled bags, were packed in a second sealed plastic bag, and shipped to JRC-Geel. After arrival (22.07.2014) a total of 1345 test pieces (or 269 sets) of ERM[®]-FA016bi were registered and stored at room temperature.

4 Homogeneity

The test pieces were sampled from the SBs and analysed to estimate the homogeneity uncertainty contribution u_h to the uncertainty of the certified value. u_h is related to s_h , the standard deviation between the test pieces in the SB (*sample-to-sample heterogeneity*), but also depends on the number of test pieces 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 at LNE on 11.07.2014 ($s_h = 2.6$ J). This leads to $u_h = \frac{s_h}{\sqrt{5}} = 1.16$ J or (0.92 %).

As is required for a homogeneity test, the test pieces were randomly selected from the whole batch. The number of test pieces tested (25) is sufficiently large to reflect the homogeneity of the full SB (1345 test pieces). 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 test pieces of nominally 120 J by Pauwels et al. [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 test pieces (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) on batches of 30 J, 80 J and 120 J from the same steel showed, as expected, no change of the measured values. Uncertainty of stabilities for 120 months were calculated and varied between 0.7 J and 2.8 J (1.8 % to 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 test pieces 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 Fe₃C). 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 but proceed slowly.

Rather than neglecting the stability issue, efforts are made 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[®]-FA016bi was characterised in June 2015, the validity of the certificate reaches until June 2025.

6 Characterisation

6.1 Characterisation tests

30 test pieces from ERM[®] FA016bi (sets 3, 50, 103, 179, 248, 270) were tested under repeatability conditions together with 25 test pieces from MB ERM[®]-FA016ax (sets 10, 115, 144, 157, 225), using the JRC-Geel Instron Wolpert PW-30 machine (UK). The pendulum is annually verified according to procedures described in ISO 148-2 [1].

Tests were performed on 16, 22 and 24 of June 2015 (laboratory temperature 20 ± 2 °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 Annex 1. The results of the measurements are summarised in Table 2.

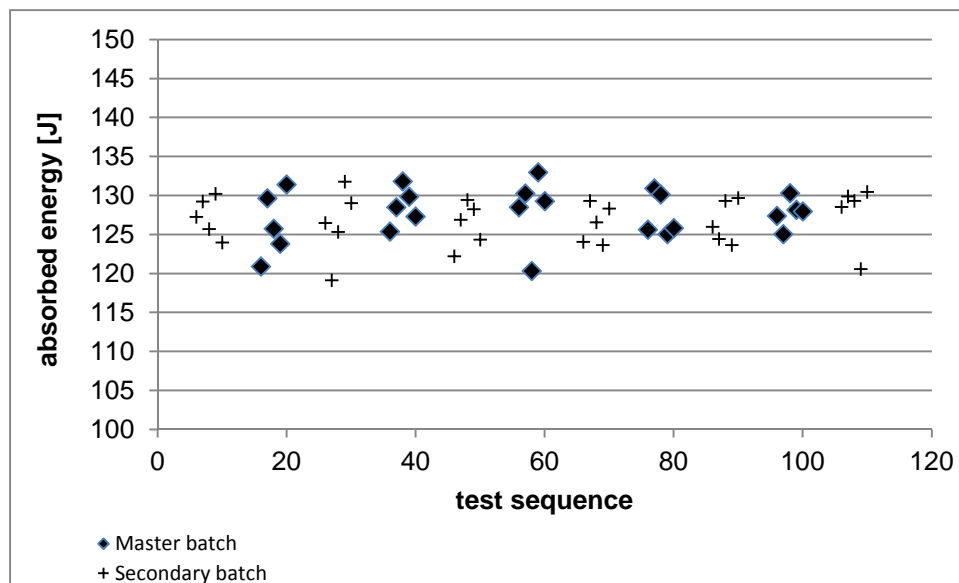


Figure 3: Absorbed energy values of 25 test pieces of ERM[®]-Fa016ax, compared with 30 test pieces of ERM[®]-Fa016bi; data are displayed in the actual test sequence

Table 2: Characterisation measurements of Batch ERM® FA016bi

	Number of test pieces n_{MB}, n_{SB}	Mean value $\bar{X}_{MB}, \bar{X}_{SB}$ [J]	Standard deviation s_{MB}, s_{SB} [J]	Relative standard deviation RSD_{SB}, RSD_{MB} [%]
ERM®-FA016ax (MB)	25	127.69	3.21	2.51
ERM®-FA016bi (SB)	30	126.81	3.11	2.46

The SB-results meet the ISO 148-3 acceptance criteria for a batch of reference materials ($RSD_{SB} < 5\%$). Also the difference between \bar{X}_{MB} and \bar{X}_{SB} the indicator used to assess the similarity of master batch and secondary batch behaviour, is smaller than the allowed 25 % (see Section 1.2.2).

6.2 Data from Master Batch ERM®-Fa016ax

To calculate KV_{CRM} for ERM®-FA016bi 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 $KV_{CRM} = 126$ J (rounded in accordance with the expanded uncertainty; see Table 5)). 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:

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

\bar{X}_{SB} and \bar{X}_{MB} were obtained under repeatability conditions. Therefore, the uncertainty of the ratio $\bar{X}_{SB} / \bar{X}_{MB}$ 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 number of the effective 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].

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

Table 4: Uncertainty budget for u_{char} for ERM® FA016bi

	source of uncertainty	measured value [J]	standard uncertainty [J]	probability distribution	relative uncertainty [%]	degrees of freedom
KV_{MB}	Certification of MB	126.82	0.93	normal	0.73	13
\bar{X}_{SB}	comparison of SB and MB in repeatability conditions	126.81	0.57	normal	0.45	29
\bar{X}_{MB}		127.69	0.64	normal	0.50	24
<i>standard uncertainty, $u_{char,rel}$ [%]</i>					1.00	37
<i>standard uncertainty, u_{char} [J]</i>					1.25	

7 Value assignment

7.1 Certified value, combined and expanded uncertainty

As shown in 6.3, $KV_{CRM} = 126$ J was rounded in accordance with the uncertainty. 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 summarised in the following uncertainty budget (Table 5).

The relevant number of degrees of freedom calculated using the Welch-Satterthwaite equation [6], is sufficiently large ($\nu_{RM} = 59$) 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 ($U_{CRM} = 3.42$ J) than the verification criterion of 10 % for industrial pendulums [1] or even 5 J for reference pendulums [9].

Table 5: Uncertainty budget of KV_{CRM} for ERM®-FA06bi

	source of uncertainty	relative value u_i [%]	degrees of freedom
u_{char}	characterisation of SB	1.00	37
u_h	homogeneity of SB	0.92	24
Combined standard uncertainty, u_{CRM} [%]			59
Combined standard uncertainty, u_{CRM} [J]			
Expanded Uncertainty, $k = 2$, U_{CRM} [%]			
Expanded Uncertainty, $k = 2$, U_{CRM} [J]			

Because of inherent imprecision of uncertainty values, the value of the U_{CRM} is rounded to 4 J. Correspondingly, the value of KV_{CRM} is rounded to 126 J.

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®-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-traceably calibrated tools.

The certified value of ERM®-FA016bi is made traceable to the SI-traceably certified value of the MB by testing SB and MB test pieces in repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools. Therefore, the certified value of ERM®-FA016bi is traceable to the International System of Units (SI) via the corresponding Master Batch ERM®-FA016ax of a similar nominal absorbed energy. Absorbed energy KV is an operationally-defined 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 6.

Table 6: Certified value and associated uncertainty for ERM®-FA016bi

Steel Charpy V-notch test pieces		
	Certified value ²⁾ [J]	Uncertainty ³⁾ [J]
Absorbed energy (KV) ¹⁾	126	4
<p>1) The absorbed energy (KV) is an operationally defined measurand. KV is the impact energy required to break a V-notched test piece of standardised dimensions, as defined in ISO 148-1. The certified value is valid only for strikers with a 2 mm tip radius, and at temperatures of (20 ± 2) °C.</p> <p>2) The certified value of ERM®-FA016bi, and its uncertainty, are traceable to the International System of Units (SI), via the master batch ERM®-Fa016ax of a similar nominal absorbed energy by testing test pieces of ERM®-Fa016ax and ERM®-FA016bi under repeatability conditions on an impact pendulum verified and calibrated with SI- traceably calibrated tools.</p> <p>3) Estimated expanded uncertainty of the mean KV of the 5 test pieces (delivered as one 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 effective degrees of freedom of the certified uncertainty is $\nu_{RM} = 59$.</p>		

11 Instructions for use

11.1 Intended use

Test pieces of ERM[®]-FA016bi 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 that 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. 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 test pieces constituting a CRM-unit need to be broken with a pendulum impact test machine in accordance with ISO 148-2 [1]. 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[®]-FA016bi as measured according to ISO 148-1 at JRC-Geel, 16, 22 and 24 June 2015.

	Master Batch ERM [®] -FA016ax	Secondary Batch ERM [®] -FA016bi
	<i>KV (J)</i>	<i>KV (J)</i>
1	120.92	127.32
2	129.66	129.27
3	125.77	125.77
4	123.83	130.25
5	131.42	124.02
6	125.38	126.55
7	128.49	119.18
8	131.81	125.38
9	129.86	131.81
10	127.32	129.08
11	128.49	122.27
12	130.25	126.94
13	120.34	129.47
14	132.98	128.30
15	129.27	124.41
16	125.65	124.09
17	130.90	129.34
18	130.12	126.62
19	125.06	123.70
20	125.84	128.37
21	127.40	126.03
22	125.06	124.48
23	130.32	129.34
24	128.17	123.70
25	127.98	129.73
26		128.56
27		129.93
28		129.34
29		120.60
30		130.51
Mean (J)	127.69	126.81
Standard deviation (J)	3.21	3.11
RSD (%)	2.51	2.46

Annex 2

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: _____



Prof. Dr. Hendrik Emons
European Commission
Joint Research Centre
Institute for Reference Materials and Measurements
Retieseweg 111
B-2440 Geel, Belgium



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)
- Bodycote Materials Testing, Emmen (NL)* (RvA testen L085)
- Bodycote Materials Testing, Spijkenisse (NL)* (RvA testen L085)
- Bundesanstalt für Materialforschung und -prüfung (BAM), Abteilung V Werkstofftechnik, Berlin (DE)* (DAP-PL-2614.16)
- Centro de Apoio Tecnológico a Industria Metalomechanica (CATIM), Laboratório de Ensaios, Porto (PT)* (IPAC L009)
- Cogne Acciai Speciali, Aosta (IT)
- Fraunhofer Gesellschaft, Institut für Werkstoffmechanik, Freiburg (DE)
- European Commission Joint Research Centre (JRC), Institute for Reference Materials and Measurements, Geel (BE) (BELAC 268-Test)
- Korea Research Institute of Standards and Science, Strength Evaluation Group, Daejeon, Korea
- 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
- SCK-CEN, Labo Reactormaterialenonderzoek, Mol (BE)* (BELAC 015-Test)
- SIRRIS, Beproevinglaboratorium Gent, Zwijnaarde (BE)* (BELAC 232-Test)
- U.S. Steel Košice, Labortest, Košice (SK)* (SNAS 026/S012)
- 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

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

European Commission – Joint Research Centre
Institute for Reference Materials and Measurements (IRMM)
Retieseweg 111, B - 2440 Geel (Belgium)
Telephone: +32-(0)14-571.722 - Fax: +32-(0)14-590.406

European Commission

EUR 28586 EN – Joint Research Centre – Directorate F – Health, Consumers and Reference Materials

Title: **CERTIFICATION REPORT: The certification of the absorbed energy (120 J nominal) of Charpy V-notch reference test pieces for tests at 20 °C: ERM®-FA016bi**

Author(s): Ts. Gerganova, G. Roebben, A. Dean, T. Linsinger

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