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ERM
European Reference Materials

CERTIFICATION REPORT

The certification of the absorbed energy (120 J nominal) of Charpy V-notch reference test pieces for tests at 20 °C: ERM®- FA016bk

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Abstract

This certification report describes the processing and characterisation of ERM®-FA016bk, a batch of Charpy V-notch certified reference test pieces certified for the absorbed energy (KV). Sets of five of these test pieces are used for the verification of pendulum impact test machines according to ISO 148-2 (Metallic materials - Charpy pendulum impact test – Part 2: Verification of testing machines).

The absorbed energy (KV) is operationally 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®-FA016bk is made traceable to the SI, via the SI-traceable certified value of the master batch ERM®-FA016ax, by testing samples pieces of ERM®-FA016bk 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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The certification of the absorbed energy (120 J nominal) of Charpy V-notch reference test pieces for tests at 20 °C: ERM®- FA016bk

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

Summary

This certification report describes the processing and characterisation of ERM®-FA016bk, a batch of Charpy V-notch certified reference test pieces certified for the absorbed energy (*KV*). Sets of five of these test pieces are used for the verification of pendulum impact test machines according to ISO 148-2 (Metallic materials - Charpy pendulum impact test – Part 2: Verification of testing machines [1]).

The absorbed energy (*KV*) is operationally 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®-FA016bk is made traceable to the SI, via the SI-traceable certified value of the master batch ERM®-FA016ax, by testing samples pieces of ERM®-FA016bk 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 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-notch test pieces		
	Certified value 2) [J]	Uncertainty 3) [J]
Absorbed energy (*KV*) 1)	121	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 in the temperature range of (20 ± 2) °C.

2) Certified values are values that fulfil the highest standards of accuracy. The certified value of ERM®-FA016bk 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®-FA016bk and ERM®-FA016ax 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 1 set), with a coverage factor $k = 2$, corresponding to a level of confidence of about 95 %, as defined in ISO/IEC Guide 98-3, Guide to the expression of uncertainty in measurement (GUM:1995). The number of degrees of freedom of the certified uncertainty is $\nu_{RM} = 62$.

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Glossary

AISI	American Iron and Steel Institute
ASTM	American Society for Testing and Materials
BCR	Community Bureau of Reference
BELAC	Belgian accreditation body
CRM	Certified Reference Material
EC	European Commission
ERM®	European Reference Material
IEC	International Electrotechnical Commission
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-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 test pieces of the Master Batch tested during certification of the Secondary Batch
n_{SB}	Number of test pieces 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 test pieces tested to assess the homogeneity of the Secondary Batch
s_{MB}	Standard deviation of the n_{MB} results of the test pieces of the Master Batch tested for the certification of the Secondary Batch
s_{SB}	Standard deviation of the n_{SB} results of the test pieces 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 test pieces of the Master Batch tested when characterising the Secondary Batch
$\overline{X}_{SB}$	Mean KV value of the n_{SB} results of the test pieces 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 test piece is broken
ν_i	Degrees of freedom for uncertainty component i
ν_{CRM}	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 test piece of the test material using a hammer rotating around a fixed horizontal axis, is schematically presented in Figure 1.

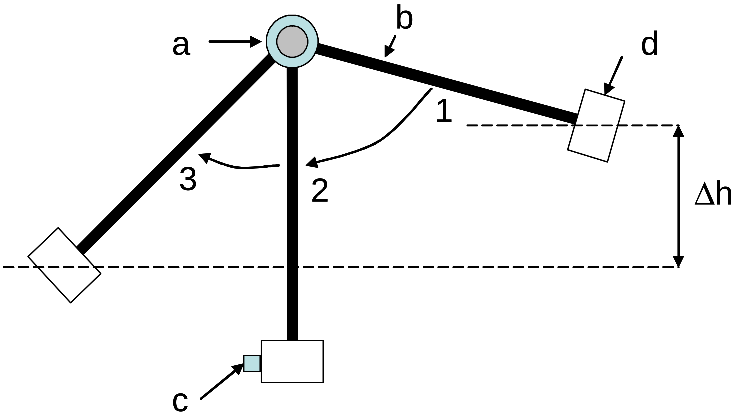

Figure 1: Schematic presentation of the Charpy pendulum impact test, showing a: the horizontal rotation axis of the pendulum, b: the stiff shaft on to which is fixed d: the hammer. The hammer is released from a well-defined height (position 1). When the hammer has reached maximum kinetic energy (shaft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

The energy absorbed by the test piece 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.

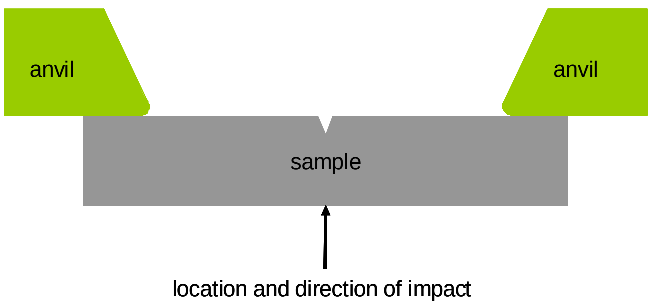

Figure 2: Schematic drawing of a V-notched Charpy test piece (top-view), indicating the place and direction of impact and the position of the anvils.

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 Directorate F – Health, Consumers and Reference Materials 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-test pieces ($\overline{X}_{\mathrm{SB}}$) 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 test pieces of the SB, is estimated by comparing the mean KV-value of a number of test pieces of the MB ($\overline{X}_{\mathrm{MB}}$), tested together with the SB test pieces 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. Procedures at the JRC allow 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 test pieces is required. This is the reason why MB and SB test pieces are made from steel with a closely-matched chemical composition nominally the same steel. Moreover, it is checked that the ratio $\frac{KV_{\mathrm{CRM}}}{KV_{\mathrm{MB}}}$ is close to 1. Procedures at the JRC allow a difference of 20 % ($KV_{\mathrm{MB}} \geq 40$ J) or 8 J ($KV_{\mathrm{MB}} < 40$ J) between KV_{CRM} and KV_{MB} to ensure that the MB and SB test pieces have a comparable interaction with the pendulum.

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

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

2 Participants

The processing of the SB (ERM®-FA016bk) test pieces was carried out by the Laboratoire national de métrologie et d'essais (LNE, FR), using AISI4340/1.6565 steel delivered by Aubert & Duval (FR). The MB test pieces (ERM®-FA016ax) used in the characterisation of the SB were provided by JRC, Directorate F (Health, Consumers and Reference Materials), Geel (BE). The homogeneity of the SB was evaluated based on data obtained at JRC using a pendulum verified according to the criteria imposed by ISO 148-2 [1]. Characterisation of the SB was carried out at JRC 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 JRC. The certification project performed was within the scope of the accreditation for the production of certified reference materials (BELAC 268-RM).

3 Processing

The ERM®-FA016bk test pieces were prepared from bars of AISI4340/1.6565 steel produced by Aubert & Duval (FR). Machining of the test pieces from these bars was performed under the supervision of LNE (see sections 3.1-3.5).

3.1 Machining of Charpy test pieces

14 bars from ingot HS360303 were cut into 1412 Charpy test pieces but not yet machined to the dimensional requirements of ISO 148-3 [9]. They were engraved to ensure identification.

3.2 Heat treatment of hot-rolled bars

The heat treatment to obtain the desired energy level of the test pieces was performed at LNE in a vacuum-furnace. 1412 test pieces of the batch were heat-treated according to the following procedure:

Step 1: normalisation treatment at 950°C for 65 minutes

Step 2: cool down in neutral gas
Step 3: austenisation treatment at 850 °C for 85 minutes
Step 4: cool down in oil
Step 5: annealing treatment at 645 °C for 120 min
Step 6: cool down in neutral gas

The test pieces were distributed over two baskets. The baskets were stacked in the furnace and each basket had several thermocouples to monitor the temperature homogeneity. The measured temperatures at all positions were within the tolerance of ± 10 °C for normalisation/austenisation treatment and ± 5 °C for annealing treatment as required by LNE**.**

3.3 Final machining of Charpy test pieces

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

3.4 Quality control

When all test pieces from the batch were fully machined, a random selection of 25 test pieces from across the whole batch 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 ± 1°, height remaining at notch root 8.00 ± 0.06 mm, radius at notch root 0.250 ± 0.025 mm, distance between the plane of symmetry of the notch and the longitudinal axis of the test piece 27.5 ± 0.2 mm). None of the test pieces was outside the ranges specified in ISO 148-3 [9].

The 25 test pieces were then impact tested using a pendulum type Tinius Olsen model 74 impact, verified in according to ISO 148-2 [1].
The tests were performed on 13/10/2014. The results are reported in the production report of LNE [10]. The average *KV* of the 25 test pieces was 121.9 J, which is within the desired energy range (112.5 J - 127.5 J). The standard deviation of the test results (*s* = 3.3 J, *RSD* = 2.7 %) was below the 4 % maximum allowed by the contract.
The variation was checked again during the characterisation tests at JRC (see Section 6).

3.5 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 the JRC Geel. After arrival (17/10/2014) the 1355 test pieces (or 271 sets) of ERM®-FA016bk were registered and stored at room temperature, pending distribution.

4 Homogeneity

The test pieces are sampled from the SBs, which should be sufficiently, but are never 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 test pieces in the SB (*test piece-to-test piece 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 five test pieces. The appropriate uncertainty contribution must be an estimate of the *set-to-set heterogeneity*, which in the case of a set of five test pieces can be calculated as $u_h = \frac{s_h}{\sqrt{5}}$.

Here, u_h is estimated from s_h, the standard deviation results obtained at JRC on 22/11/2016 (s_h = 2.57 J). This leads to $u_h = \frac{s_h}{\sqrt{5}} =$ 1.15 J (0.94 %).

As is required for a homogeneity test, the test pieces were randomly selected from the whole batch. The number of test pieces tested (30) is sufficiently large to reflect the homogeneity of the full SB (1355 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., who did not observe measurable changes of absorbed energy [11]. Additional evidence for the stability of the reference test pieces produced from AISI 4340 steel of lower energy levels (nominally 15 J, 30 J and 100 J) has been obtained during the International Master Batch (IMB) project [12]. In the IMB-project, the stability of the certified test pieces was judged from the change of the mean of means of the absorbed energy obtained on 7 reference pendulums over a three year period. None of the three regression slopes for the tested energy levels was statistically significant at the 5 % probability level. Given the large test piece-to-test piece heterogeneity and the limited number of test pieces, five, in a CRM unit, the uncertainty contribution from instability is considered to be insignificant in comparison to that of homogeneity. A dedicated isochronous study (test temperature 18 °C, reference temperature -20 °C) was organised by the JRC using batches of 30, 80 and 120 J from the same steel and showed, as expected, no change of the measured values. Uncertainties of stabilities for 120 months were calculated as 0.7 J - 2.8 J (1.8 % - 2.4 %). These uncertainties are entirely driven by the measurement precision and it was concluded that no uncertainty contribution for potential change was needed [13].

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 Fe_3C). The only driving force for instability stems from the difference in solubility of interstitial elements in the α-Fe matrix, between annealing and ambient temperature. Relaxation of residual (micro-)stress by short-range diffusion or the additional formation or growth of precipitates during the shelf-life of the certified reference test pieces is expected to proceed but slowly.

Rather than neglecting the stability issue, efforts are spent to better establish the stability of the certified values of batches of Charpy CRMs. Until such further notice, it is decided to specify a limited shelf-life. A period of 10 years is chosen, counting from the date of the characterisation tests on the SB. Since batch ERM®-FA016bk was characterised on 22/11/2016, the validity of the certificate reaches until 11/2026.

6 Characterisation

6.1 Characterisation tests

30 test pieces from ERM®-FA016bk (sets 19, 53,121, 184, 221, 246) were tested under repeatability conditions together with 25 test pieces from MB ERM®-FA016ax (sets 14, 77, 121, 175, 237), using the Wolpert PW30 machine of the JRC, Directorate F – Health, Consumers and Reference Materials, an impact pendulum yearly verified according to procedures described in ISO 148-2 [1].
Tests were performed on 22/11/2016 (laboratory temperature 20 ± 1 °C), in accordance with ISO 148-1 [2]. The measurement sequence was: SB-MB-SB-MB-SB-MB-SB-MB-SB-MB-SB. The measured absorbed energy values were corrected for friction and windage losses.

After testing, all Charpy test pieces show 'first-strike' marks: these are the marks caused by the interaction between test piece and anvils during the first and intended hammer impact. Upon fracture, the broken halve test pieces loose contact with hammer and anvils and follow one of a variety of possible trajectories, away from the pendulum, depending on the properties of both pendulum and test material. It may occur that test pieces show 'second-strike' marks. These are marks caused by a second impact of the already broken halve test pieces back onto the anvils. This phenomenon has been described by Schmieder et al. [14]. None of the broken ERM-FA016bk and none of ERM-FA016ax test pieces showed second-strike marks.

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

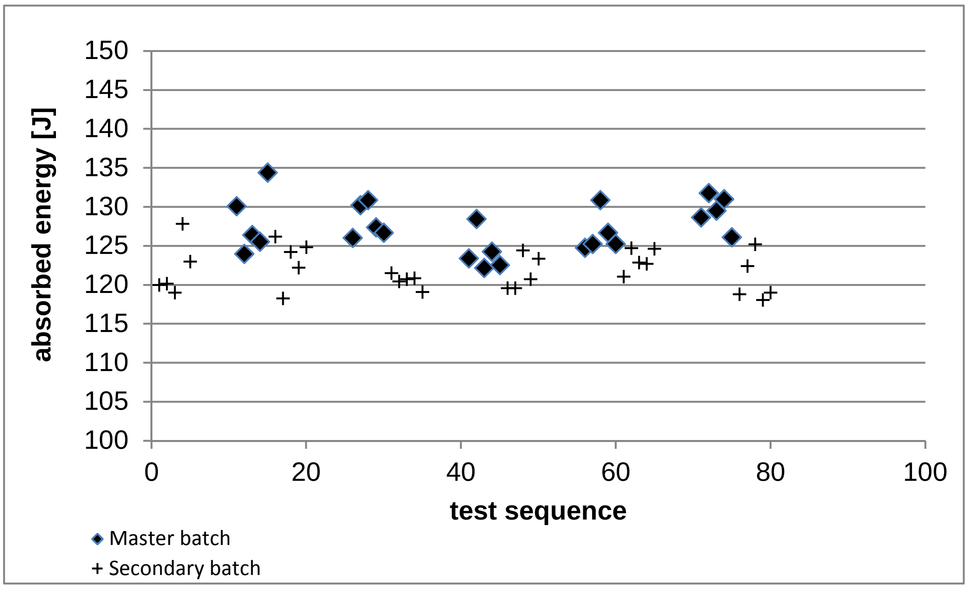

Figure 3: Absorbed energy values of 30 test pieces of ERM®-FA016bk, compared with 25 test pieces of ERM®-FA016ax; data are displayed in the actual test sequence.

Table 1: Characterisation measurements of Batch ERM®-FA016bk

	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®-FA016ax (MB)	25	127.26	3.17	2.49
ERM®-FA016bk (SB)	30	121.86	2.57	2.11

The SB-results meet the ISO 148-3 acceptance criteria for a batch of reference materials (s_{SB} ≤ 2 J for KV_{SB} < 40 J or ≤ 5 % for KV_{SB} ≥ 40 J).

6.2 Data from Master Batch ERM®-FA016ax

To calculate KV_{CRM} for ERM®-FA016bk one needs KV_{MB} of the MB used, i.e. ERM®-FA016ax. Table 2 shows the main MB-data, taken from the Certificate of Analysis of ERM®-FA016ax (Annex 2).

Table 2: 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 Table 1 and Table 2, and using Eq. 1, one readily obtains that KV_{CRM} = 121 J (rounding in accordance with uncertainty; see Table 4). The uncertainty associated with the characterisation of the SB, u_{char}, is assessed as in Eq. 2 [8], which sums the relative uncertainties of the three factors in Eq. 1:

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

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

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

Table 3: Uncertainty budget of u_{char} for ERM®-FA016bk

	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	121.86	0.47	normal	0.39	29
$\bar{x}_{MB}$		127.26	0.63	normal	0.50	24
				$u_{char,rel}$ (%)	0.97	34
				u_{char} (J)	1.17	

7 Value assignment

7.1 Certified value, combined and expanded uncertainty

As shown in 6.3, KV_{CRM} = 121 J. The uncertainty of the certified value is obtained by combining the contributions from the characterisation study, u_{char}, and from the homogeneity assessment, u_h, as is summarized in the following uncertainty budget (Table 4).

The relevant number of degrees of freedom calculated using the Welch-Satterthwaite equation [6], is sufficiently large (ν_{CRM} = 62) 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} = 2.70% or 4 J) than the verification criterion of 4 J for KV < 40 J or 10 % for KV ≥40 J for industrial pendulums [1] or even 2 J for KV < 40 J or 5 % for KV ≥40 J J for reference pendulums [9].

Table 4: Uncertainty budget of KV_{CRM} for ERM®-FA016bk

	source of uncertainty	relative value u_i (%)	degrees of freedom
u_{char}	characterisation of SB	0.97	34
u_h	homogeneity of SB	0.94	29
Combined standard uncertainty, u_{CRM} (%)		1.35	62
Combined standard uncertainty, u_{CRM} (J)		1.64	
Expanded Uncertainty, k = 2, U_{CRM} (%)		2.70	
Expanded Uncertainty, k = 2, U_{CRM} (J)		4	

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-traceable calibration tools.

The certified value of ERM®-FA016bk is made traceable to the SI-traceable 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®-FA016bk 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 measurand, and can only be obtained by following the procedures specified in ISO 148-1 [2].

9 Commutability

The samples of ERM-FA016bk are made from commercially sourced steel which behaves like any other steel of the same energy level. The material is therefore commutable with other steel samples.
The certified values are not valid when the test pieces are broken with an ASTM-type striker of 8 mm tip radius [10]

10 Instructions for use

10.1 Intended use

Test pieces of ERM®-FA016bk are '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.

10.2 Sample preparation

Special attention is drawn to the cleaning of the test pieces prior to the tests. It is mandatory to remove the oil from the test piece surface prior to testing, without damaging the edges of the test piece. 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 test piece 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 test piece, but do not use hard (e.g. steel) brushes to remove the oil from the notch.
2. Apply a second cleaning step with organic solvents. Any residual solvents shall be removed by wiping them with an absorbent tissue before proceeding to step 3.
3. Once samples are degreased, 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 test pieces to the test temperature (20 ± 2) °C. To assure thermal equilibrium is reached, move the test pieces to the test laboratory at least 3 h before the tests.

10.3 Pendulum impact tests

After cleaning, the five test pieces 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 five *KV*-values.

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Annex 1

Results of characterisation measurements of ERM®-FA016bk and ERM®-FA016ax as measured according to ISO 148-1 at JRC Geel 22/11/2016.

	Master Batch ERM®-FA016ax	**Secondary Batch ERM®-FA016bk**
	***KV* (J)**	***KV* (J)**
1	130.08	119.99
2	123.96	120.18
3	126.38	119.03
4	125.51	127.84
5	134.36	122.99
6	125.99	126.19
7	130.17	118.26
8	130.85	124.25
9	127.35	122.21
10	126.67	124.83
11	123.38	121.54
12	128.42	120.47
13	122.12	120.76
14	124.25	120.86
15	122.5	119.12
16	124.73	119.61
17	125.22	119.61
18	130.85	124.44
19	126.67	120.76
20	125.22	123.38
21	128.62	121.05
22	131.73	124.73
23	129.49	122.89
24	130.95	122.70
25	126.09	124.64
26		118.83
27		122.41
28		125.22
29		118.06
30		119.03
Mean (J)	127.26	121.86
Standard deviation (J)	3.17	2.57
***RSD* (%)**	2.49	2.11

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
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JRC
EUROPEAN COMMISSION

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 Tecnologico 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, Daejon, 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, Beproevingslaboratorium 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.

LEGAL NOTICE

NOTE

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

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European Commission

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