European Commission

ERM
European Reference Materials

CERTIFICATION REPORT

The certification of the absorbed energy (80 J nominal) of Charpy V-notch reference test pieces for tests at 20 °C: ERM ®-FA015z

EUR 27578- 2015

European Commission
Joint Research Centre
Institute for Reference Materials and Measurements (IRMM)

Contact information
Reference materials sales
Address: Institute for Reference Materials and Measurements, Retieseweg 111, 2440 Geel, Belgium
E-mail: jrc-irmm-rm-distribution@ec.europa.eu
Tel.: +32 (0)14 571 705

JRC Science Hub
https://ec.europa.eu/jrc

JRC98551

EUR 27578 EN

ISBN 978-92-79-53883-4 (PDF)
ISSN 1831-9424 (online)
doi: 10.2787/96434

Luxembourg: Publications Office of the European Union, 2015

Printed in Belgium

Abstract
This certification report describes the processing and characterisation of ERM®-FA015z, 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-notched test piece of standardised dimensions, as defined in ISO 148-1. The certified value of ERM®-FA015z is made traceable to the SI, via the SI-traceable certified value of the master batch ERM®-FA015v, by testing samples of ERM®-FA015z and ERM®-FA015v 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.

CERTIFICATION REPORT

The certification of the absorbed energy (80 J nominal) of Charpy V-notch reference test pieces for tests at 20 °C: ERM ®-FA015z

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

European Commission, Joint Research Centre
Institute for Reference Materials and Measurements (IRMM)
Geel, Belgium

Disclaimer

Summary

This certification report describes the processing and characterisation of ERM®-FA015z, 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-notched test piece of standardised dimensions, as defined in ISO 148-1 [2]. The certified value of ERM®-FA015z is made traceable to the SI, via the SI-traceable certified value of the master batch ERM®-FA015v, by testing samples of ERM®-FA015z and ERM®-FA015v 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-notched 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, is:

Steel Charpy V-notch test pieces		
	Certified value 2) [J]	Uncertainty 3) [J]
Absorbed energy (*KV*) 1)	79.4	2.3

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®-FA015z, and its uncertainty, are traceable to the International System of Units (SI), via the master batch ERM®-FA015v of a similar nominal absorbed energy by testing samples of ERM®-FA015v and ERM®-FA015z under repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools.

3) Estimated expanded uncertainty of the mean *KV* of the 5 specimens (delivered as 1 set), with a coverage factor $k = 2$, corresponding to a level of confidence of about 95 %, as defined in ISO/IEC Guide 98-3, Guide to the expression of uncertainty in measurement (GUM:1995). The number of degrees of freedom of the certified uncertainty is $\nu_{RM} = 66$.

Table of contents

Glossary

AISI	American Iron and Steel Institute
ASTM	American Society for Testing and Materials
BCR	Community Bureau of Reference
CRM	Certified Reference Material
EC	European Commission
ERM®	European Reference Material
IMB	International Master Batch
IRMM	Institute for Reference Materials and Measurements
ISO	International Organization for Standardization
JRC	Joint Research Centre
k	Coverage factor
KV	Absorbed energy = energy required to break a V-notched test piece of defined shape and dimensions when tested with a pendulum impact testing machine
KV_{CRM}	Certified KV value of a set of 5 reference test pieces from the Secondary Batch
KV_{MB}	Certified KV value of the Master Batch test pieces
LNE	Laboratoire national de métrologie et d'essais
MB	Master Batch
n_{MB}	Number of samples of the Master Batch tested during certification of the Secondary Batch
n_{SB}	Number of samples of the Secondary Batch tested for certification
RSD	Relative standard deviation
s	Standard deviation
SB	Secondary Batch
s_h	Standard deviation of the results of the samples tested to assess the homogeneity of the Secondary Batch
s_{MB}	Standard deviation of the n_{MB} results of the samples of the Master Batch tested for the certification of the Secondary Batch
s_{SB}	Standard deviation of the n_{SB} results of the samples tested for the characterisation of the Secondary Batch
u_{CRM}	Combined standard uncertainty of KV_{CRM}
U_{CRM}	Expanded uncertainty (k = 2, confidence level of about 95 %) of KV_{CRM}
u_{char}	Standard uncertainty of the result of the characterisation tests
$u_{char,rel}$	Relative standard uncertainty of the result of the characterisation tests
u_h	Contribution to uncertainty from homogeneity
u_i	Value of uncertainty from contribution i
u_{MB}	Standard uncertainty of KV_{MB}
$u_{MB,rel}$	Relative standard uncertainty of KV_{MB}
$\overline{X}_{MB}$	Mean KV value of the n_{MB} measurements on samples of the Master Batch tested when characterising the Secondary Batch
$\overline{X}_{SB}$	Mean KV value of the n_{SB} results of the samples tested for the characterisation of the Secondary Batch
Δh	difference between the height of the centre of gravity of the pendulum prior to release and at the end of the half-swing during which the test sample is broken
ν_{RM}	Effective number of degrees of freedom associated with the uncertainty of the certified value
ν_ι	Degrees of freedom
IEC	International Electrotechnical Commission
BELAC	Belgian Accreditation Body

1 Introduction

1.1 The Charpy pendulum impact test

The Charpy pendulum impact test is designed to assess the resistance of a material to shock loading. The test, which consists of breaking a notched bar of the test material using a hammer rotating around a fixed horizontal axis, is schematically presented in Figure 1.

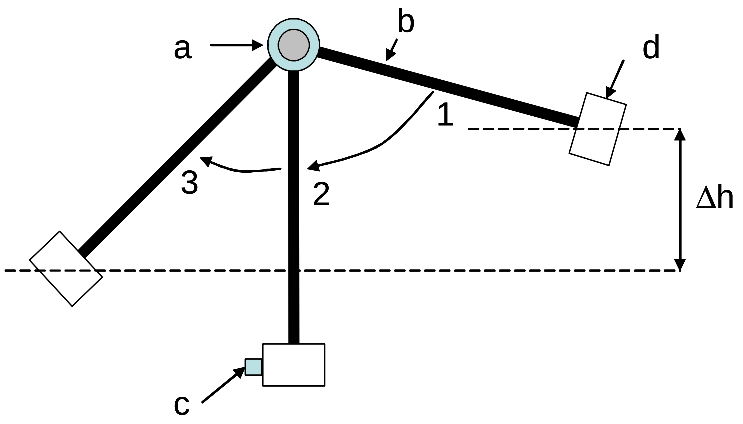

Figure 1: Schematic presentation of the Charpy pendulum impact test, showing a: the horizontal rotation axis of the pendulum, b: the stiff shaft on to which is fixed d: the hammer. The hammer is released from a well-defined height (position 1). When the hammer has reached maximum kinetic energy (shaft in vertical position 2), the hammer strikes c: the test sample, which is positioned on a support and against the pendulum anvils (not shown). The height reached by the hammer after having broken the sample (position 3) is recorded. The difference in height between position 1 and 3 (Δh) corresponds with the difference in potential energy, and is a measure of the energy required to break the test sample.

The energy absorbed by the test sample is very dependent on the impact pendulum construction and its dynamic behaviour. Methods to verify the performance of an impact pendulum require the use of reference test pieces as described in ISO and other international standards [1, 3]. The reference test pieces dealt with in this report comply with a V-notched test piece of well-defined geometry [1], schematically shown in Figure 2.

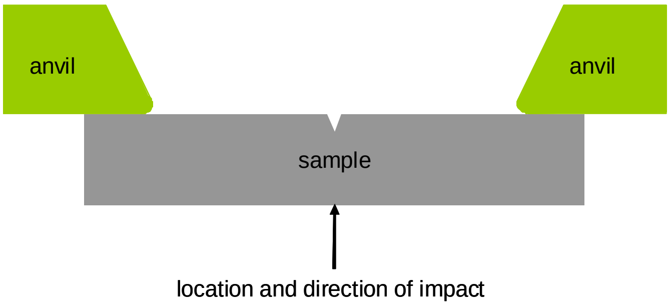

Figure 2: Schematic drawing of a V-notched Charpy test piece (top-view), indicating the place and direction of impact.

1.2 The certification concept of Master Batch and Secondary Batch

1.2.1 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 Institute for Reference Materials and Measurements (IRMM) of the European Commission's (EC) Joint Research Centre (JRC). The work was performed in accordance with procedures described in the BCR reports [4] and [5]. The certification of a SB is based on the comparison of a set of SB test pieces with a set of test pieces from the corresponding MB under repeatability conditions on a single pendulum.

The BCR reports [4] and [5] were published in 1991 and 1999, respectively. Since 2000, the calculation of the certified value and the estimation of its uncertainty have been updated to an approach compliant with the ISO/IEC Guide to the Expression of Uncertainty in Measurement [6]. This revised approach was developed and presented by Ingelbrecht et al. [7, 8], and is summarised below.

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

The certified absorbed energy of a SB of Charpy V-notch reference test pieces (KV_{CRM}) is calculated from the mean *KV*-value of a set of test pieces from the SB($\overline{X}_{\mathrm{SB}}$) 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. The certified value of the MB is used then to calculate KV_{CRM} as follows [8]:

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

For this approach to be reliable, the pendulum used for the tests on MB and SB in repeatability conditions must be well performing. In other words, the ratio $\frac{KV_{\mathrm{MB}}}{\overline{X}_{\mathrm{MB}}}$ must be close to 1. IRMM allows a difference of 5 % ($KV_{\mathrm{MB}} \geq 40$ J) or 2 J ($KV_{\mathrm{MB}} < 40$ J) between KV_{MB} and $\overline{X}_{\mathrm{MB}}$, corresponding 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 samples are made from nominally the same steel. Moreover, it is checked that the ratio $\frac{KV_{\mathrm{CRM}}}{KV_{\mathrm{MB}}}$ is close to 1. IRMM allows a difference of 25 % ($KV_{\mathrm{MB}} \geq 40$ J) or 10 J ($KV_{\mathrm{MB}} < 40$ J) between KV_{CRM} and KV_{MB}.

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 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 $\overline{X}_{MB}/\overline{X}_{SB}$. The 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®-FA015z) test pieces was carried out by the Laboratoire national de métrologie et d'essais (LNE), using AISI 4340 steel delivered by Aubert&Duval (FR) . The MB samples (ERM®-FA015v) used in the characterisation of the SB were provided by IRMM, Geel (BE). The homogeneity of the SB was evaluated based on data obtained at LNE, within the scope of an ISO/IEC 17025 accreditation, using a pendulum verified according to the criteria imposed by ISO 148-2 [1]. Also the characterisation of the SB was carried out at IRMM also 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 IRMM. The certification was performed within the scope of an ISO Guide 34 accreditation (BELAC 268-RM).

3 Processing

The ERM®-FA015z test pieces were prepared from bars of AISI 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®-FA015z batch, steel was used from ingot number HS360303.

3.2 Machining of Charpy test pieces

The ingot was divided into 14 billets. All 14 bars from one billet 3 were cut into 1429 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 1429 specimens was performed at Aubert&Duval (FR) in a vacuum-furnace, according to the following procedure:
Step 1: normalisation treatment at 950 °C for 65 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 neutral gas;
Step 3: 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 4: quenching in oil.

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]. 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 at LNE using an electro-erosion tool.

3.5 Quality control

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

The 25 samples selected throughout the batch and checked for geometrical compliance were also impact tested using a pendulum type Tinius Olsen model 74 , verified according to ISO 148-2 [1]. The tests were performed on 13.01.2015. The results are reported in the LNE production report of January 2015 [10]. The average *KV* of the 25 samples was 78.2 J, which is within the desired energy interval (75 J - 85 J). The standard deviation of the test results (*s* = 1.8 J, *RSD* = 2.3 %) was below the 4 % maximum allowed by the contract. The variation was checked again during the characterisation tests at IRMM (see Section 6).

3.6 Packaging and storage

Finally, the samples were cleaned and packed as sets of 5 randomised samples, in oil-filled and vacuum sealed plastic bags. These oil-filled bags, were packed in a second sealed plastic bag, and shipped to IRMM. After arrival (22.01.2015) the 1295 samples (or 259 sets) of ERM®-FA015z were registered and stored at room temperature.

4 Homogeneity

The test pieces are 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 samples in the SB (*sample-to-sample heterogeneity*), but also depends on the number of samples over which the *KV*-value is averaged. ISO 148-2 [1] specifies that the pendulum verification must be performed using 5 test pieces, which is why a CRM-unit consists of a set of 5 test pieces. The appropriate uncertainty contribution must be an estimate of the *set-to-set heterogeneity*, which in the case of a set of 5 test pieces can be calculated as $u_h = \frac{s_h}{\sqrt{5}}$.

Here, u_h is estimated from s_h, the standard deviation of results obtained at LNE on 17.02.2015 (s_h = 1.8 J). This leads to $u_h = \frac{s_h}{\sqrt{5}}$ = 0.80 J or (1.03 %).

As is required for a homogeneity test, the samples were randomly selected from the whole batch. The number of samples tested (25) is sufficiently large to reflect the homogeneity of the full SB (1295 samples). It can be noted that u_h is probably a slight overestimation, since it contains also the repeatability of the instrument. However, the latter cannot be separated or separately measured.

5 Stability

The stability of the absorbed energy of Charpy V-notch certified reference test pieces was first systematically investigated for samples of nominally 120 J by Pauwels et al. [11]. Additional evidence for the stability of the reference test pieces produced from AISI 4340 steel of lower energy levels (nominally 15 J, 30 J and 100 J) has been obtained during the International Master Batch (IMB) project [12]. In the IMB-project, the stability of the certified test pieces was judged from the change of the mean of means of the absorbed energy obtained on 7 reference pendulums over a three year period. None of the three regression slopes for the tested energy levels was statistically significant at the 5 % probability level. Given the large sample-to-sample heterogeneity and the limited number of samples (5) in a CRM unit, the uncertainty contribution from instability is considered to be insignificant in comparison to that of homogeneity. A dedicated isochronous study (test temperature 18 °C, reference temperature -20 °C) on batches of 30, 80 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 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 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, but proceeds slowly.

Rather than neglecting the stability issue, 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®-FA015z was characterised in June 2015, the validity of the certificate reaches until June 2025.

6 Characterisation

6.1 Characterisation tests

30 samples from ERM®-FA015z (sets 1, 73, 126, 176, 202, 259) were tested under repeatability conditions together with 25 samples from MB ERM®-FA015v (sets 6, 52, 110, 191, 239), using the IRMM Instron Wolpert PW-30 machine (UK). The pendulum is annually verified according to procedures described in ISO 148-2 [1].
Tests were performed on 11 and 12 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.

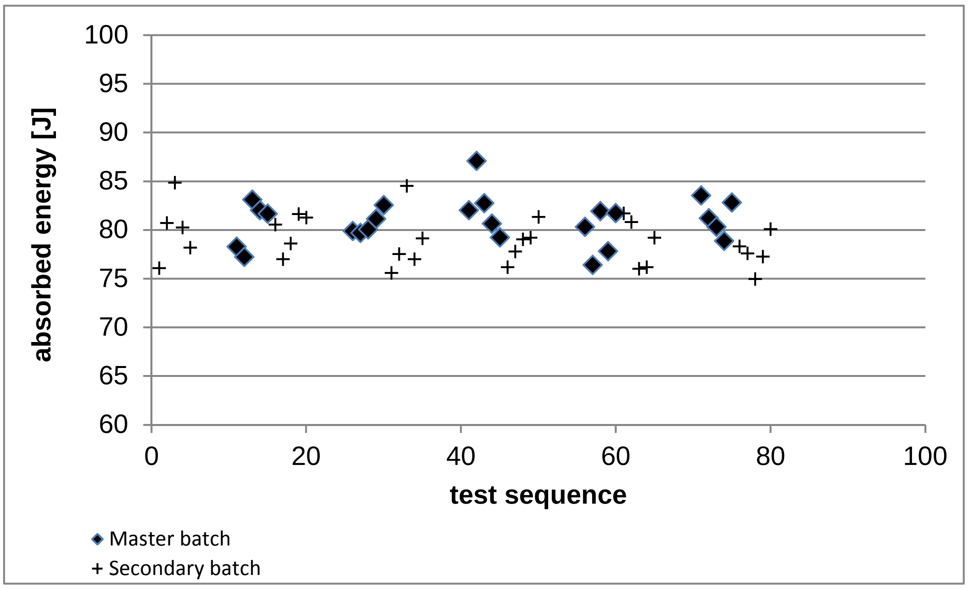

Figure 3: Absorbed energy values of 25 test pieces of ERM®-FA015v, compared with 30 test pieces of ERM®-FA015z; data are displayed in the actual test sequence

The sequence of the samples shows one high value for the master batch. This value was flagged as outlier using the Grubbs procedure. As the technical evaluation of the results did not indicate any technical flaws, there is no reason to exclude this value from the analysis data set. Moreover, the respective set of samples met the specification of ISO 148-3 for reference samples having standard deviation < 3 J.

Table 2: Characterisation measurements of Batch ERM®-FA015z

	Number of test pieces n_{MB} , n_{SB}	Mean value $\overline{X}_{MB}$, $\overline{X}_{SB}$ [J]	Standard deviation s_{MB} , s_{SB} [J]	Relative standard deviation RSD_{SB}, RSD_{MB} [%]
ERM®-FA015v (MB)	25	80.88	2.29	2.83
ERM®-FA015z (SB)	30	78.97	2.49	3.15

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

6.2 Data from Master Batch ERM®-FA015v

To calculate KV_{CRM} for ERM®-FA015z one needs KV_{MB} of the MB used, i.e. ERM®-FA015v. Table 3 shows the main MB-data, taken from the Certificate of Analysis of ERM®-FA015v (Annex 2).

Table 3: Data from the certification of Master Batch ERM®-FA015v

	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®-FA015v	81.32	0.51	0.63

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} = 79.4 J (to be rounded in accordance with the 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 squares of the relative uncertainties of the three factors in Eq. 1:

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

$\overline{X}_{SB}$ 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 4 summarises the input quantities of the u_{char} uncertainty budget, their respective statistical properties, and shows how they were combined. The effective number of degrees of freedom (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].

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

Table 4: Uncertainty budget for u_{char} for ERM®-FA015z

	source of uncertainty	measured value [J]	standard uncertainty [J]	probability distribution	relative uncertainty [%]	degrees of freedom
KV_{MB}	Certification of MB	81.32	0.51	normal	0.63	13
$\overline{x}_{SB}$	comparison of SB and MB in repeatability conditions	78.97	0.45	normal	0.58	29
$\overline{x}_{MB}$		80.88	0.46	normal	0.57	24
relative standard uncertainty, $u_{char,rel}$ [%]					1.02	54
standard uncertainty, u_{char} [J]					0.81	

7 Value assignment

7.1 Certified value, combined and expanded uncertainty

As shown in 6.3, the value of KV_{CRM} is 79.4 J (rounded in accordance to the expanded 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 (ν_{RM} = 66) 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} = 2.31 J) than the verification criterion of 10 % for industrial pendulums [1] or even 5 % for reference pendulums [9].

Table 5: Uncertainty budget of KV_{CRM} for ERM®-FA015z

	source of uncertainty	relative value u_i [%]	degrees of freedom
u_{char}	characterisation of SB	1.02	54
u_h	homogeneity of SB	1.03	24
Relative combined standard uncertainty, u_{CRM} [%]		1.45	66
Combined standard uncertainty, u_{CRM} [J]		1.15	
Relative expanded Uncertainty, k = 2, U_{CRM} [%]		2.90	
Expanded Uncertainty, k = 2, U_{CRM} [J]		2.31	

Because of inherent imprecision of uncertainty values, the value of the U_{CRM} is rounded to 2.3 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®-FA015v 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 calibrated tools.

The certified value of ERM®-FA015z is made traceable to the SI-traceable certified value of the MB by testing SB and MB samples under repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools. Therefore, the certified value of ERM®-FA015z is traceable to the International System of Units (SI) via the corresponding Master Batch ERM®-FA015v 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 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®-FA015z

Steel Charpy V-notch test pieces		
	Certified value 2) [J]	Uncertainty 3) [J]
Absorbed energy (*KV*) 1)	79.4	2.3

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

2) The certified value of ERM®-FA015z, and its uncertainty, are traceable to the International System of Units (SI), via the master batch ERM®-FA015v of similar nominal absorbed energy by testing samples of ERM®-FA015v and ERM®-FA015z under repeatability conditions on an impact pendulum verified and calibrated with SI-traceable tools.

3) Estimated expanded uncertainty of the mean *KV* of the 5 specimens (delivered as 1 set), with a coverage factor $k = 2$, corresponding to a level of confidence of about 95 %, as defined in ISO/IEC Guide 98-3, Guide to the expression of uncertainty in measurement (GUM:1995). The number of degrees of freedom of the certified uncertainty is $\nu_{RM} = 66$.

11 Instructions for use

11.1 Intended use

Samples of ERM®-FA015z 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 samples 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.

Acknowledgements

The authors wish to thank to R. Jakopic, A. Held, R. Koeber and H. Emons (all IRMM) for reviewing of the certification report.

References

1. *ISO 148-2: Metallic materials - Charpy pendulum impact test - Part 2: Verification of testing machines*, International Organization for Standardization, Geneva (CH), 2008
2. *ISO 148-1: Metallic materials - Charpy pendulum impact test - Part 1: Test method,* International Organization for Standardization, Geneva (CH), 2009
3. *ASTM E23 - 07ae1 Standard Test Methods for Notched Bar Impact Testing of Metallic Materials*, ASTM International, West Conshohocken, PA (USA), 2007
4. Marchandise H., Perez-Sainz A., Colinet E., *Certification of the impact toughness of V-notch Charpy specimens*, in *BCR information series*, Community Bureau of Reference - BCR, Brussels (BE), 1991
5. Varma R.K., *The certification of two new master batches of V-notch Charpy impact toughness specimens in accordance with EN 10045-2: 1992*, CRM's 015 and 415, EUR Report 18947 EN - European Communities, Luxembourg - 1999 - ISBN 92-828-2244-3
6. *ISO/IEC Guide 98-3:2008, Uncertainty of measurement - Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*, International Organization for Standardization, Geneva (CH), 2008
7. Ingelbrecht, C. and Pauwels J., *EC Reference Materials for Impact Toughness - Traceability and uncertainty*. Presentation at Eurachem Eurolab symposium on Reference Materials for Technologies in the New Millennium, Berlin, May 22-23, 2000
8. Ingelbrecht, C., Pauwels, J., and Gyppaz, D., *Charpy specimens from BCR for machine verification according to EN 10045-2*. Poster presentation at Charpy Centenary Conference, October 2-5, Poitiers (FR), 2001
9. *ISO 148-3: Metallic materials - Charpy pendulum impact test - Part 3: Preparation and characterization of Charpy V-notch test pieces for indirect verification of pendulum impact machines*, International Organization for Standardization, Geneva (CH), 2008
10. Characterisation report: Charpy V-notch reference test pieces ERM-FA016bh, Laboratoire national de metrologie at d'essais (DE), December 2013
11. Pauwels, J., Gyppaz, D., Varma, R., Ingelbrecht, C., *European certification of Charpy specimens: reasoning and observations*, in *Pendulum Impact testing: A Century of Progress*. Seattle, Washington: American Society for Testing and Materials, 1999
12. McCowan, C.N., Roebben, G., Yamaguchi, Y., Lefrançois, S., Splett, J. D., Takagi, S., Lamberty, A., *International Comparison of Impact Reference Materials,* J. ASTM International, Vol. 3(2), 2004
13. Schmieder, A. K., Purtscher P. T., Vigliotti, D. P., The role of strike marks on the reproducibility of Charpy impact test results, in *Pendulum impact machines: procedures and specimens for verification*, ASTM STP 1248, ed. Siewert, T. A. and Schmieder A. K., American Society for Testing and Materials, Philadelphia (USA), 1995
14. Lamberty, A, Roebben, G, Dean, A, Linsinger T, *Study of the stability of Charpy V-notch reference test pieces for tests at 20 °C (ERM®-FA013ba, ERM®-FA015v and ERM®-FA016ax) during long-term storage at 18°C, EUR 26348 EN*, Luxembourg: Publications Office of the European Union, 2015

Annex 1

Results of characterisation measurements of ERM®-FA015z as measured according to ISO 148-1 at IRMM, 11 and 12 June 2015.

	Master Batch ERM®-FA015v	**Secondary Batch ERM®-**FA015z
	***KV* (J)**	***KV* (J)**
1	78.26	76.11
2	77.20	80.72
3	83.09	84.86
4	82.01	80.26
5	81.65	78.19
6	79.86	80.58
7	79.69	77.03
8	80.04	78.62
9	81.12	81.65
10	82.55	81.29
11	82.01	75.62
12	87.07	77.56
13	82.73	84.53
14	80.65	77.03
15	79.22	79.15
16	80.29	76.21
17	76.39	77.80
18	81.90	79.05
19	77.80	79.22
20	81.72	81.36
21	83.52	81.72
22	81.19	80.83
23	80.29	76.04
24	78.87	76.21
25	82.80	79.22
26		78.34
27		77.63
28		74.99
29		77.27
30		80.11
Mean (J)	80.88	78.97
Standard deviation (J)	2.29	2.49
***RSD* (%)**	2.83	3.15

European Commission

European Reference Materials

Annex 2

CERTIFICATE OF ANALYSIS

ERM®- FA015v

STEEL		
	Impact toughness	
	Certified value [2] [J]	Uncertainty [3] [J]
Absorbed energy (*KV*) [1]	81.32	0.51

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 16 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-FA015v, estimated as the standard deviation of the mean of the 16 laboratory mean values, corresponding with a confidence level of about 68 %.

This certificate is valid until January 2018.

NOTE

European Reference Material ERM®-FA015v 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

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-FA015v 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-FA015v 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.

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 27572 EN – Joint Research Centre – Institute for Reference Materials and Measurements

Title: **CERTIFICATION REPORT The certification of the absorbed energy (80 J nominal) of Charpy V-notch reference test pieces for tests at 20 °C: ERM®-FA015z**

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

Luxembourg: Publications Office of the European Union

2015 – 20 pp. – 21.0 x 29.7 cm

EUR – Scientific and Technical Research series – ISSN 1831-9424

ISBN 978-92-79-53883-4

doi: 10.2787/96434

As the Commission's in-house science service, the Joint Research Centre's mission is to provide EU policies with independent, evidence-based scientific and technical support throughout the whole policy cycle.

Working in close cooperation with policy Directorates-General, the JRC addresses key societal challenges while stimulating innovation through developing new methods, tools and standards, and sharing its know-how with the Member States, the scientific community and international partners.

Key policy areas include: environment and climate change; energy and transport; agriculture and food security; health and consumer protection; information society and digital agenda; safety and security, including nuclear; all supported through a cross-cutting and multi-disciplinary approach.

LA-NA-27578-EN-N

Publications Office

doi: 10.2787/96434

ISBN: 978-92-79—53883-4