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Institute for Reference
Materials and Measurements



CERTIFICATION REPORT

**Certification of Charpy V-notch Reference Test Pieces
of 80 J Nominal Absorbed Energy**

Certified Reference Material ERM[®]-FA015u

EUR 23263 EN - 2007



The mission of IRMM is to promote a common and reliable European measurement system in support of EU policies.

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Institute for Reference Materials and Measurements

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Certification of Charpy V-notch Reference Test Pieces of 80 J Nominal Absorbed Energy

Certified Reference Material ERM[®]-FA015u

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Abstract

This certification report describes the processing and characterisation of ERM[®]-FA015u, a batch of Charpy V-notch certified reference test pieces. Sets of five of these test pieces are used for the verification of pendulum impact test machines according to EN 10045-2 (Charpy impact test on metallic materials, Part 2. Method for the verification of impact testing machines [1]) or according to ISO 148-2 (Metallic materials - Charpy pendulum impact test – Part 2: Verification of test machines [2]). The certified value for KV (= energy required to break a V-notched test piece using a pendulum impact test machine) is 79.8 J. The associated uncertainty (2.4 J, $k = 2$ corresponding to a confidence level of 95 %) is calculated for the mean of a set of five test pieces. The certified value is traceable to the Charpy impact test method as described in EN 10045-1 [3] and ISO 148 [4], via the corresponding Master Batch ERM[®]-FA015o of the same nominal absorbed energy (80 J).

Table of Contents

Abstract	1
Table of Contents.....	3
Glossary	5
1. Introduction: the Charpy pendulum impact test	7
2. The certification concept of Master Batch and Secondary Batch	8
2.1. <i>Difference between Master and Secondary Batches.....</i>	<i>8</i>
2.2. <i>Certification of a Secondary Batch of Charpy V-notch test pieces.....</i>	<i>8</i>
3. Participants	10
4. Processing	11
4.1. <i>Processing of hot-rolled bars</i>	<i>11</i>
4.2. <i>Heat-treatment of hot-rolled bars</i>	<i>11</i>
4.3. <i>Machining of Charpy test pieces</i>	<i>11</i>
4.4. <i>Quality control.....</i>	<i>12</i>
4.5. <i>Packaging and storage</i>	<i>12</i>
5. Characterisation	13
5.1. <i>Characterisation tests</i>	<i>13</i>
5.2. <i>Data from Master Batch ERM[®]-FA015o</i>	<i>14</i>
5.3. <i>Calculation of KV_{CRM} and of u_{char}</i>	<i>14</i>
6. Homogeneity	15
7. Stability.....	16
8. Evaluation of results	17
8.1. <i>Calculation of certified value, combined and expanded uncertainty</i>	<i>17</i>
8.2. <i>Traceability</i>	<i>17</i>
8.3. <i>Commutability.....</i>	<i>17</i>
8.4. <i>Summary of results.....</i>	<i>18</i>
9. Instructions for use	19

9.1.	<i>Intended use</i>	19
9.2.	<i>Sample preparation</i>	19
9.3.	<i>Pendulum impact tests</i>	19
10.	Acknowledgements	20
11.	References	21
Annex 1	22
Annex 2	23

Glossary

AISI	American Iron and Steel Institute
ASTM	American Society for Testing and Materials
BCR	Community Bureau of Reference
CEN	European Committee for Standardization
CRM	Certified Reference Material
EC	European Commission
EN	European Norm
ERM [®]	European Reference Material
h	hour
IMB	International Master Batch
IRMM	Institute for Reference Materials and Measurements, Joint Research Centre, European Commission
ISO	International Organisation for Standardisation
JRC	Joint Research Centre
<i>k</i>	Coverage factor
<i>KV</i>	Absorbed energy = energy required to break a V-notched test piece of defined shape and dimensions when tested with a pendulum impact testing machine
<i>KV_{CRM}</i>	Certified <i>KV</i> value of a set of 5 reference test pieces from the Secondary Batch
<i>KV_{MB}</i>	Certified <i>KV</i> value of the Master Batch test pieces
LNE	Laboratoire National d'Essais
MB	Master Batch
min	minute

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 of the secondary batch tested to assess its homogeneity
s_{MB}	Standard deviation of the n_{MB} results of the samples of the Master Batch tested for the certification of the Secondary Batch
s_{SB}	Standard deviation of the n_{SB} results of the samples of the Secondary Batch tested for its characterisation
u_{CRM}	Combined standard uncertainty of KV_{CRM}
U_{CRM}	Expanded uncertainty ($k = 2$, confidence level 95 %) of KV_{CRM}
u_{char}	Standard uncertainty of the result of the characterisation tests
u_h	Homogeneity contribution to uncertainty
u_i	Value of uncertainty from contribution i
u_{MB}	Standard uncertainty of KV_{MB}
$wt\%$	Weight percent
\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 of the Secondary Batch tested for its characterisation
Δh	difference between the height of the centre of gravity of the pendulum prior to release and at end of first half-swing, after breaking the test sample
ν_{RM}	Effective number of degrees of freedom associated with the uncertainty of the certified value

1. Introduction: the Charpy pendulum impact test

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

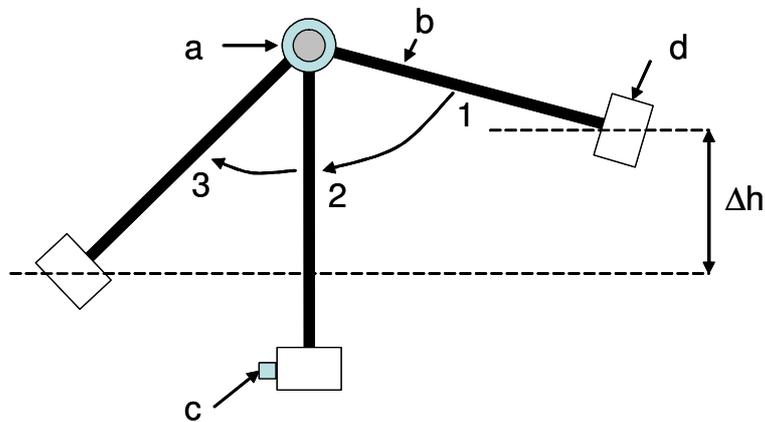


Figure 1: Schematic presentation of the Charpy pendulum impact test, showing a: the horizontal rotation axis of the pendulum, b: the stiff shaft on to which is fixed d: the hammer. The hammer is released from a well-defined height (position 1). The hammer strikes c: the test sample, when the hammer has reached maximum kinetic energy (shaft in vertical position 2). The height reached by the hammer after having broken the sample (position 3) is recorded. The difference in height between position 1 and 3 (Δh) corresponds with a difference in potential energy, 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 European, American and ISO standards [1, 2, 5]. The reference test pieces dealt with in this report comply with a V-notched test piece shape of well-defined geometry [1, 2], schematically shown in Figure 2.

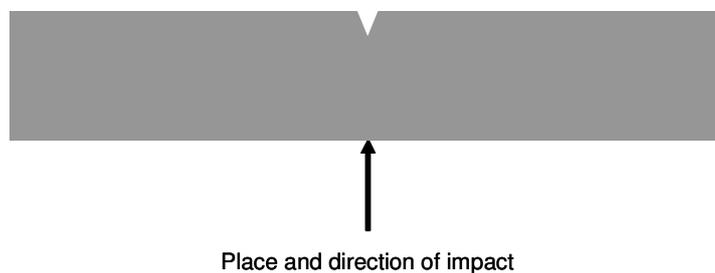


Figure 2: Schematic drawing of a V-notched Charpy sample (top-view), indicating the place and direction of impact.

2. The certification concept of Master Batch and Secondary Batch

2.1. *Difference between Master and Secondary Batches*

The BCR reports by Marchandise et al. [6] and Varma [7] provide details of the certification of the BCR “Master Batches” (MB) of Charpy V-notch certified reference test pieces. The certified value of a Master Batch is obtained using an international laboratory intercomparison.

This report describes the production of a “Secondary Batch” (SB) of Charpy V-notch certified reference test pieces at the Institute for Reference Materials and Measurements (IRMM) of the European Commission (EC) Joint Research Centre (JRC). The work was performed in accordance with procedures described in the BCR reports [6] and [7]. The certification of a SB is based on the comparison of a set of SB test pieces with a set of test pieces from the corresponding MB under repeatability conditions on a single pendulum.

While the MB-SB approach necessarily results in a larger uncertainty of the certified value of SB in comparison with the MB, the uncertainty can be kept sufficiently small to meet the requirements of the intended use of the certified reference material (CRM). Avoiding the need for an international interlaboratory comparison for each produced batch, the MB-SB approach allows cost-efficient production of certified reference test pieces.

The BCR reports [6] and [7] were published in 1991 and 1999, respectively. Since 2000, the calculation of the certified value and the estimation of its uncertainty have been updated to an approach compliant with the ISO Guide to the Expression of Uncertainty in Measurement [8]. This revised approach was developed and presented by Ingelbrecht et al. [9, 10], and is summarised below.

2.2. *Certification of a Secondary Batch of Charpy V-notch test pieces*

The certified absorbed energy of a SB of Charpy V-notch reference test pieces (KV_{CRM}) is calculated from the mean KV -value of a set of SB-samples (\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 samples of the SB, is estimated by comparing the mean KV -value of a number of samples of the MB (\bar{X}_{MB}), tested together with the SB samples under repeatability conditions, with the certified value of the MB (KV_{MB}). KV_{CRM} is then calculated as follows [10]:

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

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. IRMM now allows a difference of 5 % ($KV_{MB} > 40$ J) or 2 J ($KV_{MB} < 40$ J) between KV_{MB} and \bar{X}_{MB} , corresponding with the level of bias allowed for reference pendulums specified in EN 10045-2 [1] and ISO 148-3 [11].

Also, for reasons of commutability, a comparable response of the pendulum to the MB and SB samples is required. This is the reason why MB and SB samples are made from nominally the same steel. Moreover, it is checked that the ratio $\frac{KV_{CRM}}{KV_{MB}}$ is close to 1. IRMM now allows a difference of 20 % ($KV_{MB} > 40$ J) or 8 J ($KV_{MB} < 40$ J) between KV_{CRM} and KV_{MB} .

3. Participants

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

4. Processing

The ERM[®]-FA015u test pieces were prepared from AISI 4340 steel. The steel was cast and rolled into bars at Cogne Acciai Speciali (see section 4.1). Production of the test pieces from these bars was performed under the supervision of LNE (see sections 4.2, 4.3, 4.4, and 4.5).

4.1. Processing of hot-rolled bars

The base material consisted of AISI 4340 steel, produced at Cogne Acciai Speciali. To limit the amount of impurities potentially affecting the homogeneity of the fracture resistance, the following compositional tolerances were imposed on the selected steel batch: Mn 0.7 – 0.8, Mo 0.23 - 0.28, Ni 1.7 – 1.85, P < 0.01, Si 0.2 – 0.35, S < 0.008 (in wt %), which is stricter than generally allowed for AISI 4340. The ingot was hot rolled, resulting in bars that were 4 m long and with a squared cross-section of 11.5 mm. For the ERM[®]-FA015u batch, steel was used from ingot number 960133, billet F. A billet is a semi-finished hot-rolled product, in this case of cross-section 108.5 mm, which is between the ingot (560 mm cross-section) stage and the final required bars (11.5 mm cross-section). A full description of the processing and quality check of the steel bars is available in [12].

4.2. Heat-treatment of hot-rolled bars

The heat treatment of the hot-rolled bars was performed at Aubert&Duval, Gennevilliers (FR). 20 bars were heat-treated together. Bars were placed onto rollers which slowly move the bars back and forth inside the furnace during the heat treatment to increase the homogeneity of the resulting microstructure. The first heat treatment was an austenisation treatment performed in a furnace of 'class 10 °C' ¹ at 850 °C for 32 min. From this furnace, the bars were quenched into oil at 40 °C. After the oil-quench, the samples were annealed in a second furnace ('class 5 °C') at 585 °C for 120 min. After this annealing treatment, the samples were cooled down in air.

After heat treatment, a limited number of samples (5) were machined for a preliminary check of the obtained energy level. Results indicated an average KV-level (76.8 J) close to the desired nominal energy level (80 J).

4.3. Machining of Charpy test pieces

After the heat treatment the samples were machined to dimensional tolerances imposed in EN 10045-2 [1]. The batch code was engraved on one end face of each sample ('80' indicates the nominal absorbed energy level (80 J); 'U' is the letter code as assigned consecutively to batches of the same nominal absorbed energy). The V-notch was introduced using an electro-erosion tool.

¹ In a furnace of 'class x °C', the variation of the temperature is smaller than x °C. The furnaces used have 10 heating zones. Each zone has 3 controlling thermocouples and 3 measurement thermocouples. These are regularly calibrated. When one faulty thermocouple is detected, it is replaced by a thermocouple produced with wire from the same roll. When a roll is exhausted, all thermocouples are replaced with new ones.

4.4. Quality control

When all samples from the batch were fully machined, a selection of 25 samples was made. The dimensions of the 25 samples were checked on June 13, 2003 against the criteria specified in EN 10045-2 [1] (length $55.0^{0.0}_{0.25}$ mm, height 10.00 ± 0.06 mm, width 10.00 ± 0.075 mm, notch angle 45 ± 1 °, height remaining at notch root 8.00 ± 0.06 mm, radius at notch root 0.25 ± 0.025 mm, distance between the plane of symmetry of the notch and the longitudinal axis of the test piece 27.50 ± 0.10 mm). None of the samples were outside the ranges specified in EN 10045-2 [1].

The 25 samples checked for geometrical compliance were impact tested on June 13, 2003 on the Tinius Olsen 358 Joules pendulum - which is one of the French reference pendulums - at LNE. The results are reported in certificate LNE n° C111076/CQPE/3 [13]. The average *KV* of the 25 samples was 79.2 J, sufficiently close to the target value (80 J). The standard deviation of the test results ($s = 1.63$ J, $RSD = 2.1$ %) was smaller than the maximum allowed 3 %. The variation was checked again during the certification tests at IRMM (see section 5).

4.5. Packaging and storage

Finally, the samples were cleaned and packed in sets of 5, in oil-filled and closed plastic bags. These oil-filled bags, together with a label, again were packed in a sealed plastic bag, and shipped to IRMM. After arrival (July 2003), the 1175 samples (or 235 sets) were registered and stored at room temperature, pending distribution.

5. Characterisation

5.1. Characterisation tests

30 samples from ERM[®]-FA015u (sets 13, 76, 129, 181, 204 and 224) were tested under repeatability conditions with 25 samples from MB ERM[®]-FA015o (sets 22, 35, 43, 54 and 69), using the Instron Wolpert PW 30 (serial number 7300 H1527) machine of IRMM, an impact pendulum yearly verified according to procedures described in EN 10045-2 [1] and ISO 148-2 [2]. Tests were performed on January 29, 2008 (laboratory temperature 19 ± 1 °C), in accordance with EN 10045-1 and ISO 148. The measured absorbed energy values were corrected for friction and windage losses. Data obtained on individual test pieces are shown in Figure 3 and in Annex 1. The results of the measurements are summarised in Table 1.

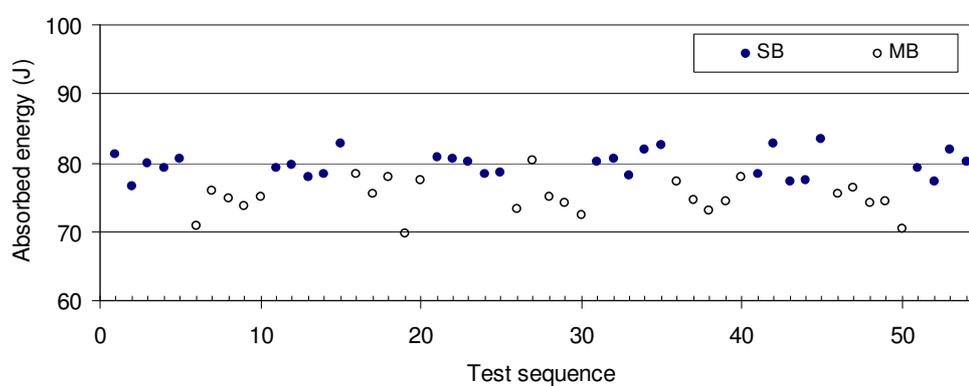


Figure 3: Absorbed energy values of the 30 test pieces of ERM[®]-FA015u and 25 test pieces of ERM[®]-FA015o displayed in the actual test sequence.

Table 1: Results of characterisation of Batch ERM[®]-FA015u by comparison with Master Batch ERM[®]-FA015o.

	Number of test pieces	Mean value	Standard deviation	Relative standard deviation
	n_{MB} or n_{SB}	\bar{X}_{MB} or \bar{X}_{SB} (J)	s_{MB} or s_{SB} (J)	RSD (%)
ERM[®]-FA015o (MB)	25	74.91	2.53	3.4
ERM[®]-FA015u (SB)	30	79.78	1.82	2.3

The RSD of the 30 SB-results (2.3 %) meets the EN 10045-2 and ISO 148-3 acceptance criteria for a batch of reference materials ($RSD < 5$ %), as well as the more stringent acceptance criterion ($RSD < 3$ %) contractually fixed between IRMM and its sample supplier.

5.2. Data from Master Batch ERM[®]-FA015o

To calculate KV_{CRM} for ERM[®]-FA015u one needs KV_{MB} of the MB used, i.e. ERM[®]-FA015o. Table 2 shows the main MB-data, taken from the Certificate of Analysis of ERM[®]-FA015o (Annex 2), which is the revised, ERM-version of the originally issued certificate, based on the certification report of the MB [6].

Table 2: Data from the certification of Master Batch ERM[®]-FA015o [6].

	Certified absorbed energy of Master Batch	Standard uncertainty of KV_{MB}	Standard uncertainty of KV_{MB}
	KV_{MB} (J)	u_{MB} (J)	u_{MB} (%)
ERM[®]-FA015o	74.9	0.6	0.8

5.3. 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 $KV_{CRM} = 79.8$ J. The uncertainty associated with the characterisation of the SB, u_{char} , is assessed as in Eq. 2 [10], which sums the relative uncertainties of the three factors 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 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 for u_{char} is obtained using the Welch-Satterthwaite equation [8].

Table 3: Uncertainty budget for u_{char}

symbol	source of uncertainty	measured value (J)	standard uncertainty (J)	probability distribution	divisor	sensitivity coefficient	relative uncertainty (%)	degrees of freedom	
KV_{MB}	certification of MB	74.9	0.6	normal	1	1	0.80	8	
\bar{X}_{SB}	comparison of SB and MB in repeatability conditions	79.78	0.33	normal	1	1	0.42	29	
\bar{X}_{MB}		74.91	0.51	normal	1	1	0.68	24	
							u_{char} (%)	1.13	26
							u_{char} (J)	0.90	

6. Homogeneity

The test pieces constituting a CRM unit are sampled from the SB, which is sufficiently, but not perfectly, homogeneous. Therefore, a separate homogeneity contribution u_h to the uncertainty of the certified value is required.

Here, u_h is estimated from s_h , the standard deviation (1.63 J) of results obtained at LNE on June 13, 2003. As is required for a homogeneity test, the samples were randomly selected from the whole batch. The number of samples tested (25) is largely sufficient to reflect the homogeneity of the full SB (1175 samples).

The effect of s_{SB} on the uncertainty of the certified value depends on the number of samples over which the KV -value is averaged. EN 10045-2 [1] and in ISO 148-2 [2] specify that the pendulum verification must be performed using 5 test pieces. Therefore, a CRM-unit consists of 5 test pieces, and

$u_h = \frac{s_h}{\sqrt{5}} = 0.73 \text{ J}$. 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.

7. Stability

The stability of the absorbed energy of Charpy V-notch certified reference test pieces was first systematically investigated for samples of nominally 120 J by Pauwels et al., who did not observe measurable changes of absorbed energy [14]. New 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 recently, during the International Master Batch (IMB) project [15]. In the IMB-project, the stability of the certified test pieces is deduced from the unchanged value of the mean of means of the absorbed energy obtained on 7 reference pendulums over a three year period. Given the large sample-to-sample heterogeneity, the ageing effects are undetectable when testing limited numbers of samples, and the uncertainty contribution from instability is considered to be insignificant.

The main reason for the microstructural stability of the certified reference test pieces is the annealing treatment to which the samples were subjected after the austenisation treatment. Annealing is performed at temperatures where the equilibrium phases are the same as the (meta-)stable phases at ambient temperature (α -Fe and Fe_3C). The only driving force for instability stems from the difference in solubility of interstitial elements in the α -Fe matrix, between annealing and ambient temperature. Relaxation of residual (micro-)stress by short-range diffusion or the additional formation or growth of precipitates during the shelf-life of the certified reference test pieces is expected to proceed but slowly.

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[®]-FA015u was characterised in January, 2008, the validity of the certificate stretches until January, 2018.

8. Evaluation of results

8.1. Calculation of certified value, combined and expanded uncertainty

As shown in 5.2, $KV_{CRM} = 79.8$ 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).

Table 4: Uncertainty budget of KV_{CRM}

symbol	source of uncertainty	absolute value (J)	u_i (J)	degrees of freedom
u_{char}	characterisation of SB	0.90	0.90	26
u_h	homogeneity of SB	0.73	0.73	24
Combined standard uncertainty, u_{CRM}			1.16 J	48
Expanded Uncertainty, $k = 2$, U_{CRM}			2.4 J	ν_{RM}

The relevant number of degrees of freedom calculated using the Welch-Satterthwaite equation [8], is sufficiently large ($\nu_{RM} = 48$) 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 than the verification criterion of 10 % (8 J) or even 5 % (4 J) for reference pendulums.

8.2. Traceability

The absorbed energy KV is a method-specific value, and can only be obtained by following the procedures specified in EN 10045-1 [3] and ISO 148 [4]. The certified value of the MB ERM[®]-FA015o is traceable to these standard procedures as it was obtained using an interlaboratory comparison, involving a representative selection of qualified laboratories performing the tests in accordance with the standard procedures.

The certified value of ERM[®]-FA015u, is made traceable to the certified value of the MB using tests on SB and MB samples in repeatability conditions. Therefore the certified value of ERM[®]-FA015u is traceable to the Charpy impact test as described in EN 10045-1 and ISO 148.

8.3. Commutability

The intended use of the certified reference test pieces is the verification of Charpy impact pendulums. During the certification of the MB, 9 different pendulums were used, each equipped with a 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, although the expected differences can be small for materials with relatively low absorbed energy such as the ERM[®]-FA015 batches of 80 J nominal absorbed energy [14].

8.4. Summary of results

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

Table 5: Certified value and associated uncertainties for ERM[®]-FA015u.

	Certified mean value for set of 5 test pieces KV_{CRM} (J)	Combined standard uncertainty u_{CRM} (J)	Expanded uncertainty (k = 2) U_{CRM} (J)
ERM[®]-FA015u	79.8	1.16	2.4

9. Instructions for use

9.1. Intended use

Samples of ERM[®]-FA015u correspond with the '(certified) BCR test pieces' as referred to in EN 10045-2 [1], as well as with the 'certified reference test pieces' as defined in ISO 148-3 [11]. Sets of five of these certified reference test pieces are intended for the indirect verification of impact testing machines with a striker of 2 mm tip radius according to procedures described in detail in EN 10045-2 [1] and ISO 148-2 [2].

The indirect verification provides an assessment of the bias of the user's Charpy pendulum impact machine. This bias assessment can be used in the calculation of the measurement uncertainty of Charpy tests on the pendulum after indirect verification. Such uncertainty calculation requires the certified value, the associated uncertainty, and in some cases also the degrees of freedom of the uncertainty, all given on page 1 of the certificate.

9.2. Sample preparation

Special attention is drawn to cleaning of the specimens prior to the tests. It is mandatory to remove the oil from the sample surface prior to testing, without damaging the edges of the sample. Between the moment of removing the protective oil layer and the actual test, corrosion can occur. This must be avoided by limiting this period of time, while keeping the sample clean.

The following procedure is considered a good practice.

1. First use absorbent cleaning-tissue to remove the excess oil. Pay particular attention to the notch of the sample, but do not use hard (e.g. steel) brushes to remove the oil from the notch.
2. Submerge the samples in ethanol for about 5 min. Use of ultrasonication is encouraged, but only if the edges of the samples are prevented from rubbing against each other. To reduce the consumption of solvent, it is allowed to make a first cleaning step with detergent, immediately prior to the solvent step.
3. Once samples are removed from the solvent, only manipulate the samples wearing clean gloves. This is to prevent development of corrosion between the time of cleaning and the actual test.
4. Before testing, bring the specimens to the test temperature (20 ± 2 °C). To assure thermal equilibrium is reached, move the specimens to the test laboratory at least 3 h before the tests.

9.3. Pendulum impact tests

After cleaning, the 5 samples constituting a CRM-unit need to be broken with a pendulum impact test machine in accordance with EN 10045-2 [1] or ISO 148-2 [2] standards. Prior to the tests, the anvils must be cleaned. It must be noted that Charpy test pieces sometimes leave debris on the Charpy pendulum anvils. Therefore, the anvils must be checked regularly and if debris is found, it must be removed. The uncertainty of the certified value applies to the mean of the 5 KV-values.

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11. References

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Annex 1

Results of characterisation measurements of ERM[®]-FA015u as measured according to EN 10045-1 and ISO 148 at IRMM, January 29, 2008.

	Master Batch ERM[®]-FA015o	Secondary Batch ERM[®]-FA015u
	<i>KV(J)</i>	<i>KV(J)</i>
1	70.84	81.23
2	75.90	76.60
3	74.84	79.79
4	73.69	79.26
5	75.09	80.51
6	78.37	79.15
7	75.54	79.69
8	77.84	77.91
9	69.80	78.44
10	77.48	82.73
11	73.16	80.69
12	80.22	80.58
13	75.09	80.04
14	74.03	78.26
15	72.47	78.62
16	77.27	80.11
17	74.63	80.47
18	73.06	78.16
19	74.45	81.90
20	77.98	82.62
21	75.58	78.40
22	76.46	82.87
23	74.17	77.34
24	74.35	77.52
25	70.35	83.41
26		79.29
27		77.34
28		81.79
29		80.18
30		78.58
Mean (J)	74.91	79.78
Standard deviation (J)	2.53	1.82
RSD (%)	3.4	2.3

Annex 2



CERTIFICATE OF ANALYSIS

ERM[®] - FA015o

Steel Charpy V-notch test pieces (nominal absorbed energy ¹⁾ 80 J, Master Batch)		
Parameter	Certified value ²⁾ (J)	Uncertainty ³⁾ (J)
Absorbed energy (KV) at 20 ± 2 °C, according to EN 10045-1 and ISO 148	74.9	0.6

1) The term absorbed energy is defined in EN 10045-1 and ISO 148 and refers to the impact energy required to break a V-notched bar of standardised dimensions.

2) Mean absorbed energy of test pieces from batch ERM[®]-FA015o. The certified value was obtained as the mean of means of absorbed energies measured at 9 laboratories. At each laboratory, 10 test pieces were broken. The certified value is traceable to the Charpy impact test method as described in EN 10045-1 and ISO 148. Therefore, the certified value is valid only for impact hammers with a 2 mm striker tip radius.

3) Half-width of the 68 % confidence interval of the mean absorbed energy defined in 2), estimated as $\frac{\sigma_m}{\sqrt{9}}$, with σ_m the standard deviation of the mean of the mean values obtained at the 9 participating laboratories.

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

NOTE

European Reference Material ERM[®]-FA015o was originally certified as BCR-015 O. It was produced and certified under the responsibility of IRMM according to the principles laid down in the technical guidelines of the European Reference Materials[®] co-operation agreement between BAM-IRMM-LGC. Information on these guidelines is available on the Internet (<http://www.erm-crm.org>).

Accepted as an ERM[®], Geel, July 2005

Signed: _____

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All following pages are an integral part of the certificate.

Page 1 of 3

DESCRIPTION OF THE SAMPLE

A unit consists of five 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 10045-1 and ISO 148. The five specimens are packed together in a plastic bag filled with oil to prevent oxidation.

INSTRUCTIONS FOR USE

The ERM-FA0150 batch is intended to be used as a 'Master Batch'. Master Batch test pieces are not for sale. They are used to traceably certify Secondary Batches of Charpy V-notch reference test pieces of the same type of steel with the same nominal absorbed energy (here 80 J).

To characterise a secondary batch, a selection of samples from the secondary batch have to be broken under repeatability conditions together with Master Batch test pieces. The certified value of the Master Batch and its associated uncertainty are used in the calculation of the certified value and its combined and expanded uncertainty of a set of 5 specimens from a Secondary Batch. Sets of 5 samples of Secondary Batches are distributed as certified reference test pieces for the verification of Charpy impact test machines in accordance with EN 10045-2 and ISO 148-2.

Special attention is drawn to cleaning and conditioning of the specimens prior to testing. The following procedure is recommended:

1. Wipe excess oil from the specimens with cellulose paper.
2. Immerse the specimens in a clean bath of degreasing solvent for about 5 min.
3. Wipe the specimens with cellulose paper and let dry.
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 12 h before the tests.

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

The cleaned samples need to be broken with an impact pendulum in accordance with EN 10045-1 or ISO 148 standards.

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.

After testing, the user is recommended to inspect the traces/imprints left behind by the anvils and hammer on the two halves of the broken specimen. Asymmetry of these marks can indicate problems with the machine geometry or the positioning of the sample prior to impact. If so desired, broken samples can be stored for later inspection of the anvil and striker marks.

STORAGE

Specimens should be kept at ambient temperature in their original packing until used. The European Commission cannot be held responsible for changes that happen during storage of the material at the customer's premises, especially of opened samples.

SAFETY INFORMATION

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

METHOD USED FOR CERTIFICATION

Charpy pendulum impact tests in accordance with EN 10045-1 and ISO 148, using pendulum impact machines with a 2 mm striker tip radius.

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NOTE

A detailed technical report of the Master Batch certification project ("The certification of two new Master Batches of V-notch Charpy impact toughness specimens in accordance with EN 10045-2: 1992", R. K. Varma, bcr information, EUR 18947 EN, 1999) can be obtained from IRMM on explicit request. In this report, the ERM-FA015o batch is called 'CRM 015 O'.

European Commission

EUR 23263 EN – Joint Research Centre – Institute for Reference Materials and Measurements

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Abstract

This certification report describes the processing and characterisation of ERM[®]-FA015u, a batch of Charpy V-notch certified reference test pieces. Sets of five of these test pieces are used for the verification of pendulum impact test machines according to EN 10045-2 (Charpy impact test on metallic materials, Part 2. Method for the verification of impact testing machines [1]) or according to ISO 148-2 (Metallic materials - Charpy pendulum impact test – Part 2: Verification of test machines [2]). The certified value for KV (= energy required to break a V-notched test piece using a pendulum impact test machine) is 79.8 J. The associated uncertainty (2.4 J, $k = 2$ corresponding to a confidence level of 95 %) is calculated for the mean of a set of five test pieces. The certified value is traceable to the Charpy impact test method as described in EN 10045-1 [3] and ISO 148 [4], via the corresponding Master Batch ERM[®]-FA015o of the same nominal absorbed energy (80 J).

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