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European Reference Materials

## CERTIFICATION REPORT

**Certification of Charpy V-notch reference test pieces  
of 150 J nominal absorbed energy**

**Certified Reference Material ERM<sup>®</sup>-FA415i**



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

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Directorate-General Joint Research Centre  
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## **CERTIFICATION REPORT**

**Certification of Charpy V-notch reference test pieces  
of 150 J nominal absorbed energy**

**Certified Reference Material ERM<sup>®</sup>-FA415i**

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## Summary

This certification report describes the processing and characterisation of ERM<sup>®</sup>-FA415i, 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 155.1 J. The associated uncertainty (4.6 J,  $k = 2$  corresponding to a confidence level of about 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<sup>®</sup>-FA415b of the same nominal absorbed energy (150 J). The certified value is valid only for impact hammers with a 2 mm striker tip radius.



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## Glossary

AISI	American Iron and Steel Institute
ASTM	American Society for Testing and Materials
BCR	Community Bureau of Reference
CEN	European Committee for Standardization
CRM	Certified Reference Material
EC	European Commission
EN	European Norm
ERM <sup>®</sup>	Trademark European Reference Material
IMB	International Master Batch
IRMM	Institute for Reference Materials and Measurements, Joint Research Centre, European Commission
ISO	International Organisation 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 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
SB	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 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
$u_{MB}$	Standard uncertainty of $KV_{MB}$
$\bar{X}_{MB}$	Mean KV value of the $n_{MB}$ measurements on samples of the Master Batch tested when characterising the Secondary Batch
$\bar{X}_{SB}$	Mean KV value of the $n_{SB}$ results of the samples of the Secondary Batch tested for its characterisation
$\Delta h$	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
$V_{eff}$	Effective number of degrees of freedom associated with the uncertainty of the certified value

## 1. Introduction: the Charpy pendulum impact test

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

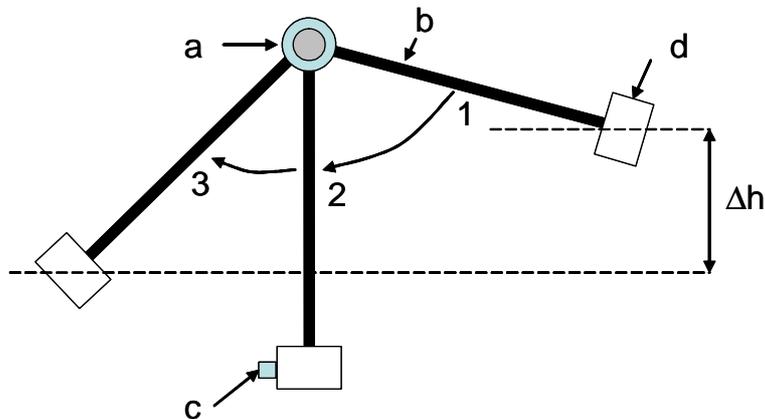


Figure 1: Schematic presentation of the Charpy pendulum impact test, showing a: the horizontal rotation axis of the pendulum, b: the stiff shaft on to which is fixed d: the hammer. The hammer is released from a well-defined height (position 1). The hammer strikes c: the test sample, when the hammer has 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 ( $\Delta h$ ) corresponds to 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 [1], ISO [2] and American [5] standards. 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.

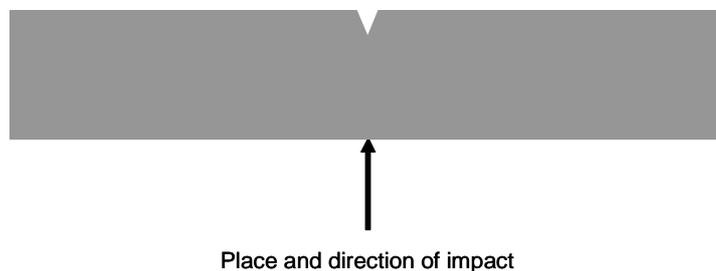


Figure 2: Schematic drawing of a V-notched Charpy sample (top-view), indicating the place and direction of impact.

## 2. The certification concept of Master Batch and Secondary Batch

### 2.1. Difference between Master and Secondary Batches

The BCR (Community Bureau of Reference) 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, involving a representative selection of qualified laboratories performing the tests in accordance with the standard procedures.

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 Varma [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 and characterisation of the SB test pieces was carried out by Cogne Acciai Speciali, Aosta (IT). The MB samples used in the characterisation of the SB were provided by IRMM, Geel (BE), where also data evaluation was performed.

## 4. Processing

The ERM<sup>®</sup>-FA415i test pieces were prepared from ASTM 565 steel. The steel was cast and rolled into bars at Cogne Acciai Speciali (see section 4.1). Heat treatment and production of the test pieces from these bars were performed later, also at Cogne Acciai Speciali (see sections 4.2, 4.3, 4.4, and 4.5).

### 4.1. Processing of hot-rolled bars

The base material consisted of ASTM 565 – Grade XM32 steel, produced at Cogne Acciai Speciali. To limit the amount of impurities potentially affecting the homogeneity of the fracture resistance, the compositional tolerances specified in Table 1 were imposed on the selected steel batch. These tolerances are stricter than generally allowed for ASTM 565.

Table 1: Adapted composition tolerances of ASTM 565 Grade XM32.

composition (mass %)						
C	S	P	Si	Mn	Cr	Ni
0.11 - 0.13	< 0.003	0.018	0.15 – 0.3	0.15 – 0.3	11.25 – 11.65	2.55 – 2.75
Mo	Cu	Al	V	W	N	
1.55 – 1.7	< 0.2	< 0.01	0.25 – 0.3	< 0.1	0.025 - 0.04	

The ingot was hot rolled, resulting in bars that were 4 m long and with a squared cross-section of 14 mm. For the ERM<sup>®</sup>-FA415i batch, steel was used from ingot number 060569. Before heat treatment, the available hot rolled bars were cut into rectangular beams of 58 mm length. These rectangular beams were machined to 55 mm length and 11 mm x 11 mm cross-section. The batch code (3, 160, I) was engraved on one end face of each sample ('3' indicates the steel type (ASTM 565); '160' indicates the target absorbed energy level; 'I' is the letter as assigned consecutively to batches of nominally the same absorbed energy).

### 4.2. Heat-treatment of test pieces

The heat treatment of the test pieces was performed in salt baths. Samples were put into steel wire baskets containing 16 samples each. The baskets were fixed at regular spacing on a frame, collecting nominally 420 samples, which were immersed into two salt baths. Prior to immersion in the salt baths, the homogeneity of the temperature in these baths was checked with a calibrated mobile thermocouple unit (temperatures observed vary < 4 °C).

The first salt bath was at 1040 °C. Samples were kept at this temperature for 30 min for an austenisation treatment. From this bath, samples were quenched into oil at 40 °C for 15 min, and further cooled down in air. After the oil-quench, the samples were annealed twice in a second salt bath, first at 705 °C, then at 670 °C, for 120 min. After this annealing treatment, the samples were cooled down in oil. The suitability of the temperatures of the second bath, which strongly affect the KV value of the resulting samples, was checked as follows.

The FA415i batch was processed together with batches FA415d-h. A selection of 24 samples of the first batch (FA415d) and 24 samples of the last batch (FA415i) were submitted to a test heat treatment (performed on January 27, 1998). To create thermal inertia conditions similar to the treatment of a full batch, the 48 selected test samples were accompanied by about 370 dummy samples. After the heat treatment, samples were machined to final dimensions. The most important dimensions (sides, depth of notch, notch-tip radius) of the 48 samples were checked against the contractual criteria deduced from ISO 148 [3] and EN 10045 [1] before they were impact tested on the Metro Com (Serial No 2005) pendulum at Cogné (February 9, 1998; laboratory temperature 22 °C, relative humidity 23 %). The average KV of the 24 samples of batch FA415i was 157.32 J (s = 3.84 J, RSD = 2.44 %). The average KV of the 24 samples of batch FA415d was 153.94 J (s = 4.89 J, RSD = 3.18 %). Since the latter is slightly lower than the original target value (160 J), it was chosen to slightly increase the second annealing temperature for the full batch to 675 °C. After the test heat treatment, 420 samples were left to constitute batch ERM<sup>®</sup>-FA415i. These samples were heat-treated on May 5, 1998.

#### **4.3. Machining of Charpy test pieces**

After the heat treatment the samples were machined to dimensional tolerances imposed in EN 10045-2 [1]. First, the cross-section was reduced to 10 mm x 10 mm, respecting the orthogonality of the four long faces of the bar. Then the surface was finished to roughness  $R_a < 0.8 \mu\text{m}$ . Finally, the V-notch was introduced using a diamond grinding tool with the appropriate V-profile.

#### **4.4. Quality control**

When all samples from the batch were fully machined, a selection of 30 samples was made from different heat treatment baskets. The dimensions of the samples were checked against the criteria specified in EN 10045-2 [1] (length  $55.0^{0.0}_{0.25}$  mm, height  $10.00 \pm 0.06$  mm, width  $10.00 \pm 0.075$  mm, notch angle  $45 \pm 1$  °, height remaining at notch root  $8.00 \pm 0.06$  mm, radius at notch root  $0.25 \pm 0.025$  mm, distance between the plane of symmetry of the notch and the longitudinal axis of the test piece  $27.50 \pm 0.10$  mm). None of the samples were outside the ranges specified in EN 10045-2 [1]. Then, characterisation tests were performed (see section 5 and Annex 1). Having confirmed the suitability of the batch with respect to the average KV and standard deviation, the dimensions of 65 samples (20 % of the batch) were checked. None of the samples were outside ranges specified in EN 10045-2 [1].

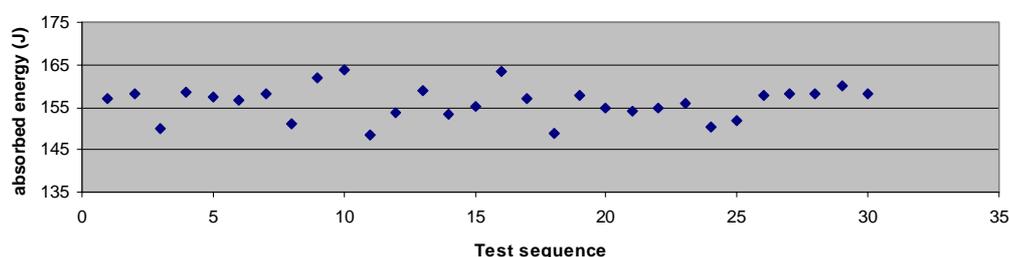
#### **4.5. Packaging and storage**

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, 78 sets were registered and stored at room temperature, pending distribution.

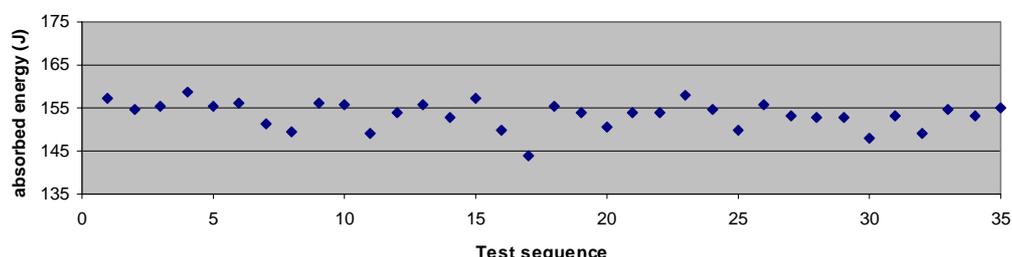
## 5. Characterisation

### 5.1. Characterisation tests

30 samples from ERM<sup>®</sup>-FA415i were tested under repeatability conditions with 35 samples from MB ERM<sup>®</sup>-FA415b (sets 1, 6, 7, 8, 10, 12, and 14), using the Metro Com (serial number 2005) machine of Cogne Acciai Speciali, an impact pendulum yearly verified according to procedures described in EN 10045-2 [1]. Tests were performed on May 19, 1998 (laboratory temperature 20 °C, relative humidity 41 %), in accordance with EN 10045-1 and ISO 148. Data obtained on individual test pieces are shown in Figure 3 and in Annex 1.



a)



b)

Figure 3: Absorbed energy values of the 30 test pieces of ERM<sup>®</sup>-FA415i and 35 test pieces of ERM<sup>®</sup>-FA415b.

Grubbs' test was performed on the result of a MB sample (sequence number 17) confirming it is not an outlier. The results of the measurements are summarised in Table 2. The RSD of the 30 SB-results (2.51 %) 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.

Table 2: Characterisation measurements of Batch ERM<sup>®</sup>-FA415i.

	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 (%)
<b>ERM<sup>®</sup>-FA415b (MB)</b>	35	153.44	3.16	2.06
<b>ERM<sup>®</sup>-FA415i (SB)</b>	30	156.13	3.91	2.51

### 5.2. Data from Master Batch ERM<sup>®</sup>-FA415b

To calculate  $KV_{CRM}$  for ERM<sup>®</sup>-FA415i one needs  $KV_{MB}$  of the MB used, i.e. ERM<sup>®</sup>-FA415b. Table 3 shows the main MB-data, taken from the Certificate of Analysis of ERM<sup>®</sup>-FA415b (Annex 2), which is the revised, ERM<sup>®</sup>-version of the originally issued certificate, based on the certification report of the MB [7].

Table 3: Data from the certification of Master Batch ERM<sup>®</sup>-FA415b [7].

	Certified absorbed energy of Master Batch	Standard uncertainty of $KV_{MB}$	Standard uncertainty of $KV_{MB}$
	$KV_{MB}$ (J)	$u_{MB}$ (J)	$u_{MB}$ (%)
<b>ERM<sup>®</sup>-FA415b</b>	152.4	1.1	0.7

### 5.3. Calculation of $KV_{CRM}$ and of $u_{char}$

From the data in Table 2 and Table 3, and using Eq. 1, one readily obtains that  $KV_{CRM} = 155.1$  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 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 for  $u_{char}$  is obtained using the Welch-Satterthwaite equation [8].

Table 4: 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	152.4	1.1	normal	1	1	0.7	8	
$\bar{X}_{SB}$	comparison of SB and MB in repeatability conditions	156.13	0.71	normal	1	1	0.46	29	
$\bar{X}_{MB}$		153.44	3.16	normal	1	1	0.35	34	
							$u_{char}$ (%)	0.91	21
							$u_{char}$ (J)	1.43	

## 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  $s_{SB}$ , the standard deviation of the results of the characterisation tests on the SB (see Table 2). The samples tested were randomly selected from the whole batch. The number of samples tested (30) is sufficient to reflect the homogeneity of the full SB (about 400 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

$u_h = \frac{s_{SB}}{\sqrt{5}} = 1.75 \text{ J}$ .  $u_h$  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 [12]. New evidence for the stability of the reference test pieces of other energy levels (nominally 15 J, 30 J and 100 J) has been obtained recently, during the International Master Batch (IMB) project [13]. 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 ( $\alpha$ -Fe and  $\text{Fe}_3\text{C}$ ). The only driving force for instability stems from the difference in solubility of interstitial elements in the  $\alpha$ -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<sup>®</sup>-FA415i was characterised in May, 1998, the certified value is valid until May, 1998.

## 8. Evaluation of results

### 8.1. Calculation of certified value, combined and expanded uncertainty

As shown in 5.2,  $KV_{CRM} = 155.1$  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 5).

Table 5: Uncertainty budget of  $KV_{CRM}$

symbol	source of uncertainty	absolute value (J)	$u_i$ (J)	degrees of freedom
$u_{char}$	characterisation of SB	1.43	1.43	21
$u_h$	homogeneity of SB	1.75	1.75	29
Combined standard uncertainty, $u_{CRM}$ (J)			2.26	49
Expanded Uncertainty, $k = 2$ , $U_{CRM}$ (J)			4.6	$\nu_{eff}$
Expanded Uncertainty, $k = 2$ , $U_{CRM}$ (%)			2.9	

The relevant number of degrees of freedom calculated using the Welch-Satterthwaite equation [8], is sufficiently large ( $\nu_{eff} = 49$ ) 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:  $U_{CRM}$  (4.6 J, or 2.9 %) is sufficiently smaller than the verification criterion of 10 % or even 5 % (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<sup>®</sup>-FA415b 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<sup>®</sup>-FA415i, 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<sup>®</sup>-FA415i 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. The certified values are not to be used when the test pieces are broken with an ASTM-type striker of 8 mm tip radius.

#### 8.4. Summary of results

The certified value and associated uncertainty are summarized in Table 6.

*Table 6: Certified value and associated uncertainty for ERM<sup>®</sup>-FA415i.*

	<b>Certified value <math>KV_{CRM}</math></b> <b>(J)</b>	<b>Expanded uncertainty (k = 2) <math>U_{CRM}</math></b> <b>(J)</b>
<b>ERM<sup>®</sup>-FA415i</b>	155.1	4.6

## **9. Instructions for use**

### **9.1. Intended use**

Samples of ERM<sup>®</sup>-FA415i 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].

### **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 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 ( $20 \pm 2$  °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.

## **10. Acknowledgements**

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## Annex 1

Results of characterisation measurements of ERM<sup>®</sup>-FA415i as measured according to EN 10045-1 and ISO 148 at Cogne Acciai Speciali, May 19, 1998.

	<b>Master Batch ERM<sup>®</sup>-FA415b</b>	<b>Secondary Batch ERM<sup>®</sup>-FA415i</b>
	<b>KV (J)</b>	<b>KV (J)</b>
1	157.25	157
2	154.5	158.25
3	155.5	150
4	158.75	158.5
5	155.25	157.5
6	156	156.5
7	151.25	158
8	149.5	151.25
9	156.25	162
10	155.75	163.75
11	149	148.5
12	154	153.75
13	155.75	159
14	152.75	153.25
15	157.25	155.25
16	150	163.5
17	144	157
18	155.25	149
19	153.75	157.75
20	150.5	154.75
21	154	154.25
22	154	155
23	158	156
24	154.75	150.5
25	150	152
26	155.75	157.75
27	153.25	158
28	152.75	158
29	152.75	160
30	148	158
31	153.25	
32	149	
33	154.5	
34	153	
35	155	
<b>Mean (J)</b>	<b>153.436</b>	<b>156.133</b>
<b>Standard deviation (J)</b>	<b>3.158</b>	<b>3.911</b>
<b>RSD (%)</b>	<b>2.058</b>	<b>2.505</b>

## Annex 2



# CERTIFICATE OF ANALYSIS

ERM<sup>®</sup> - FA415b

Steel Charpy V-notch test pieces (nominal absorbed energy <sup>1)</sup> 150 J, Master Batch)		
Parameter	Certified value <sup>2)</sup> (J)	Uncertainty <sup>3)</sup> (J)
Absorbed energy (KV) at 20 ± 2 °C, according to EN 10045-1 and ISO 148	152.4	1.1

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<sup>®</sup>-FA415b. 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. 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  $\sigma_m$  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<sup>®</sup>-FA415b was originally certified as BCR-415 B. 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<sup>®</sup> 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<sup>®</sup>, Geel, July 2005

Signed: \_\_\_\_\_

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All following pages are an integral part of the certificate.

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## 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-FA415b 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 150 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 ( $20 \pm 2$  °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.

Unlike Charpy test pieces of lower nominal impact energies, samples from ERM-FA415 batches sometimes leave debris on the Charpy pendulum anvils. Therefore, after each impact, the anvils must be checked and if debris is found, it must be removed.

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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- Lloyd's Register, Croydon (UK)
- National Institute of Standards and Technology (NIST), Boulder, CO (US)
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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-FA415b batch is called 'CRM 415 B'.

**EUR 21848 – DG Joint Research Centre, Institute for Reference Materials and Measurements –**  
Certification of Charpy V-notch reference test pieces of 150 J nominal absorbed energy – ERM<sup>®</sup>-FA415i  
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### **Abstract**

This certification report describes the processing and characterisation of ERM<sup>®</sup>-FA415i, 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 155.1 J. The associated uncertainty (4.6 J,  $k = 2$  corresponding to a confidence level of about 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<sup>®</sup>-FA415b of the same nominal absorbed energy (150 J). The certified value is valid only for impact hammers with a 2 mm striker tip radius.

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