CERTIFICATION REPORT

The certification of the absorbed energy (80 J nominal) of Charpy V-notch reference test pieces for tests at 20 °C: ERM®-FA015af

EUR 31294 EN

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Abstract

This certification report describes the processing and characterisation of ERM®-FA015af, a batch of Charpy V-notch alloy steel certified reference test pieces certified for the average absorbed energy (KV). Sets of five of these test pieces are used for the indirect verification of pendulum impact test machines according to ISO 148-2.
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-FA015af is made traceable to the International System of Units (SI), via the SI-traceable certified value of the master batch ERM®-FA015v, by testing test pieces of ERM-FA015af and ERM-FA015v under repeatability conditions on a Charpy reference pendulum verified and calibrated with SI-traceably calibrated tools. The certified value is valid only for strikers with a 2 mm tip radius. The certified value is valid at (20 ± 2) °C.
The certified value for KV and the associated expanded uncertainty (k = 2 corresponding to a confidence level of about 95 %) were calculated in accordance with ISO 17034:2016 and ISO Guide 35:2017 for the mean of a set of five test pieces. The uncertainty includes contributions related to possible inhomogeneity and characterisation. Before release of the certified reference material, the certification project was subjected to peerreview.

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The certification of the absorbed energy (80 J nominal) of Charpy V-notch reference test pieces for tests at 20 °C: ERM®-FA015af

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Disclaimer

Summary

This certification report describes the processing and characterisation of ERM®-FA013cf, a batch of Charpy V-notch alloy steel certified reference test pieces certified for the average absorbed energy (*KV*). Sets of five of these test pieces are used for the indirect verification of pendulum impact test machines according to ISO 148-2 [1].
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-FA013cf is made traceable to the International System of Units (SI), via the SI-traceable certified value of the master batch ERM®-FA013ba [3], by testing test pieces of ERM-FA013cf and ERM-FA013ba under repeatability conditions on a Charpy reference pendulum verified and calibrated with SI-traceably calibrated tools. The certified value is valid only for strikers with a 2 mm tip radius. The certified value is valid at (20 ± 2) °C.

The certified value for *KV* and the associated expanded uncertainty (k = 2 corresponding to a confidence level of about 95 %) were calculated in accordance with ISO 17034:2016 [4] and ISO Guide 35:2017 [5] for the mean of a set of five test pieces. The uncertainty includes contributions related to possible inhomogeneity and characterisation. Before release of the certified reference material, the certification project was subjected to peer-review. The following value was assigned:

Steel Charpy V-notch test pieces

	Certified value [2] [J]	Uncertainty [3] [J]
Absorbed energy (*KV*) [1]	20.4	0.7

1) Impact energy required to break a V-notched test piece of standardised dimensions, as defined in ISO 148-1:2016. The certified value is valid only for strikers with a 2 mm tip radius, and in the temperature range of (20 ± 2) °C.

2) Certified values are values that fulfil the highest standards of accuracy. The given value represents the arithmetic mean of 30 individual results obtained by direct comparison of ERM-FA013cf with ERM-FA013ba, a master batch of similar nominal absorbed energy, under repeatability conditions on an impact pendulum verified and calibrated according to ISO 148-2:2016. The certified value and its uncertainty refer to the mean KV of the five test pieces delivered as one set. The certified value and its uncertainty are traceable to the International System of Units (SI).

3) The uncertainty of the certified value is the expanded uncertainty with a coverage factor k = 2 corresponding to a level of confidence of 95 %, estimated in accordance with ISO 17034:2016 and ISO Guide 35:2017. The certified uncertainty has 57 effective degrees of freedom.

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Glossary

AISI	American Iron and Steel Institute
ASTM	ASTM International (American standardisation organisation)
BCR	Community Bureau of Reference
BELAC	Belgian accreditation body
CRM	Certified Reference Material
EC	European Commission
ERM®	Trademark owned by the European Commission; used by the JRC for reference materials
IEC	International Electrotechnical Commission
IMB	International Master Batch
IfEP	Institut für Eignungsprüfung GmbH
ISO	International Organization for Standardization
JRC	Joint Research Centre of the European Commission
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 five reference test pieces from the Secondary Batch
KV_{MB}	Certified KV value of the Master Batch test pieces
MB	Master Batch
n_{MB}	Number of test pieces of the Master Batch tested during certification of the Secondary Batch
n_{SB}	Number of test pieces of the Secondary Batch tested for certification
RSD	Relative standard deviation
RSD_{MB}	Relative standard deviation of the n_{MB} results of the test pieces of the Master Batch tested for the certification of the Secondary Batch
RSD_{SB}	Relative standard deviation of the n_{SB} results of the test pieces for the characterisation of the Secondary Batch
s	Standard deviation
SB	Secondary Batch
s_{h}	Standard deviation of the results of the test pieces tested to assess the homogeneity of the Secondary Batch
s_{MB}	Standard deviation of the n_{MB} results of the test pieces of the Master Batch tested for the certification of the Secondary Batch
s_{SB}	Standard deviation of the n_{SB} results of the test pieces tested for the characterisation of the Secondary Batch
u_{CRM}	Combined standard uncertainty of the certified value
U_{CRM}	Expanded uncertainty of the certified value ; an additional index "rel" is added when appropriate
u_{char}	Standard uncertainty of the material characterisation; an additional index "rel" is added when appropriate
u_{h}	Standard uncertainty related to a possible between-unit inhomogeneity; an additional index "rel" is added when appropriate
u_{i}	Value of uncertainty from contribution i
u_{MB}	Standard uncertainty of KV_{MB}; an additional index "rel" is added when appropriate
$\overline{X}_{\mathrm{MB}}$	Mean KV value of the n_{MB} measurements on test pieces of the Master Batch tested when characterising the Secondary Batch
$\overline{X}_{\mathrm{SB}}$	Mean KV value of the n_{SB} results of the test pieces tested for the characterisation of the Secondary Batch
Δh	difference between the height of the centre of gravity of the pendulum prior to release and at the end of the half-swing during which the test piece is broken
ν_{i}	Degrees of freedom for uncertainty component i
ν_{CRM}	Effective degrees of freedom of the uncertainty of the certified value

1 Introduction

1.1 Background

1.1.1 The Charpy pendulum impact test

The Charpy pendulum impact test is designed to assess the resistance of a material to shock loading. The test consists of breaking a notched test piece of the test material using a hammer rotating around a fixed horizontal axis and is schematically presented in Figure1.

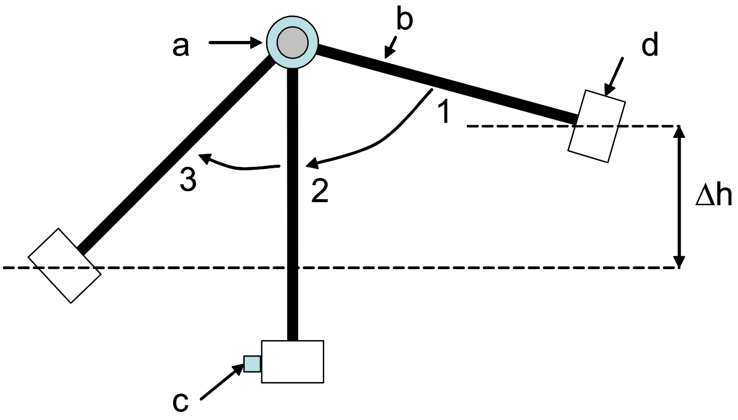

Figure 1: Schematic presentation of the Charpy pendulum impact test, showing a: the horizontal rotation axis of the pendulum, b: the stiff shaft on to which is fixed d: the hammer. The hammer is released from a well-defined height (position 1). When the hammer has reached maximum kinetic energy (shaft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

The energy absorbed by the test piece is very dependent on the impact pendulum construction and its dynamic behaviour. Methods to verify the performance of an impact pendulum require the use of reference test pieces as described in ISO and other international documentary standards [1, 6]. The reference test pieces dealt with in this report comply with a V-notched test piece shape of well-defined geometry [2], schematically shown in Figure 2.

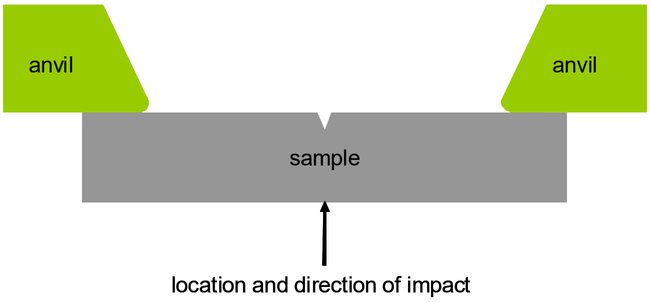

Figure 2: Schematic drawing of a V-notched Charpy test piece (top-view), indicating the place and direction of impact and the position of the anvils.

1.1.2 The certification concept of Master Batch and Secondary Batch

1.1.2.1 Difference between Master and Secondary Batches

The BCR reports by Marchandise et al. [7] and Varma [8] 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 interlaboratory comparison (ILC).

This report describes the production of a "Secondary Batch" (SB) of Charpy V-notch certified reference test pieces at the Directorate F – Health, Consumers and Reference Materials of the European Commission's (EC) Joint Research Centre (JRC). The work was performed in accordance with procedures described in the BCR reports [7] and [8]. 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 [7] and [8] 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 ISO 17034:2016 [4]/ ISO Guide 35:2017 [5] and the ISO/IEC Guide to the Expression of Uncertainty in Measurement [9], respectively. This revised approach was developed and presented by Ingelbrecht et al. [10, 11], and is summarised below.

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

The certified absorbed energy of a SB of Charpy V-notch reference test pieces (KV_{CRM}) is calculated from the mean KV-value of a set of SB test pieces ($\overline{X}_{SB}$) tested on a single pendulum. This value $\overline{X}_{SB}$ has to be corrected for the bias of this particular pendulum. The bias of the pendulum at the moment of testing the test pieces of the SB, is estimated by comparing the mean KV-value of a number of test pieces of the MB ($\overline{X}_{MB}$), tested together with the SB test pieces under repeatability conditions, with the certified value of the MB (KV_{MB}). KV_{CRM} is then calculated as follows [11]:

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

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 $\frac{KV_{MB}}{\overline{X}_{MB}}$ must be close to 1. Procedures at the JRC allow a difference of 5 % ($KV_{MB} \geq 40$ J) or 2 J ($KV_{MB} < 40$ J) between KV_{MB} and $\overline{X}_{MB}$, corresponding with the level of bias allowed for reference pendulums specified in ISO 148-3 [12].

For reasons of commutability, a comparable response of the pendulum to the MB and SB test pieces is required. This is the reason why MB and SB test pieces are made from steel with a closely-matched chemical composition. Moreover, it is checked that the ratio $\frac{KV_{CRM}}{KV_{MB}}$ is close to 1. Procedures at the JRC allow a difference of 20 % ($KV_{MB} \geq 40$ J) or 10 J ($KV_{MB} < 40$ J) between KV_{CRM} and KV_{MB} to ensure that the MB and SB test pieces have a comparable interaction with the pendulum.

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

The uncertainty of the certified value of the SB is a combination of the uncertainties of the right-hand side factors in Equation 1. It is clear that the MB-SB approach necessarily results in a larger uncertainty of the certified value of the SB in comparison with the MB. The additional uncertainty depends on the uncertainty of the ratio $\overline{X}_{\mathrm{MB}}/\overline{X}_{\mathrm{SB}}$. The full measurement uncertainty of the values $\overline{X}_{\mathrm{MB}}$ and $\overline{X}_{\mathrm{SB}}$ 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 $\overline{X}_{\mathrm{MB}}$ and $\overline{X}_{\mathrm{SB}}$ 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 $\overline{X}_{\mathrm{MB}}/\overline{X}_{\mathrm{SB}}$, 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).

1.2 Choice of the material

Steel is one of the most frequently used materials worldwide. The impact toughness of structural steel is one important property for planning buildings and equipment. Consequently, testing of the impact toughness of the steel used is required in legislation, for example the EU legislation on construction products (Regulation 305/2011) and pressure equipment (Directive 2014/68/EU). Steel is therefore the material most often tested by ISO 148 and is therefore the material of choice for a CRM.

The validity of the MB-SB approach to characterisation requires that both MB and SB have similar breaking behaviour, meaning that similar amounts of energy are absorbed by actually breaking the sample and by ductile deformation. Apart from the energy level itself, the chemical composition contributes to the amount of ductile deformation. As the MB is a AISI 4340/1.6565 type steel, also a AISI 4340/1.6565 was chosen for the SB.

1.3 Outline of the CRM project

The production of a CRM as defined in ISO 17034 [4] is a project comprising planning, processing of the material, homogeneity and stability testing, characterisation and assigning of the property values and finally distribution and post-certification monitoring to control stability.

The characterisation principle applied is characterisation by direct comparison with an existing CRM (primary batch or MB), as described in 1.1.2.

The uncertainty of the certified value was estimated in compliance with ISO 17034 [4], which implements the basic principles of ISO/IEC Guide 98 (GUM) [9].

The CRM project, including the certification approach and the evaluation of the obtained measurement data, was subjected to peer-review.

2 Participants

2.1 Project management and data evaluation

European Commission, Joint Research Centre, Directorate F – Health, Consumers and Reference Materials, Unit F.6 (Reference Materials), Geel, BE
(accredited to ISO 17034:2016 for production of certified reference materials, BELAC No. 268-RM)

2.2 Processing

Institut für Eignungsprüfung GmbH (IfEP), Marl (DE)
(accredited to DIN EN ISO Guide 34 for production of certified reference materials, DAkkS No. D-RM-11183-01-00)

2.3 Homogeneity measurements

Institut für Eignungsprüfung GmbH (IfEP), Marl (DE)

2.4 Characterisation measurements

European Commission, Joint Research Centre, Directorate F – Health, Consumers and Reference Materials, Geel, BE
(measurements under the scope of ISO/IEC 17025:2017 accreditation BELAC No. 268-TEST)

3 Material processing and process control

3.1 Origin of the starting material

The ERM-FA013cf test pieces were prepared from bars of AISI 4340/1.6565 steel produced by Tata Steel International Germany GmbH. One broad bar was cut into 36 discs which were subsequently normalised for 150 min at 900 °C..

3.2 Processing

3.2.1 Machining of Charpy test pieces

Machining of the test pieces was performed by IfEP. 12 discs from one bar were cut into 1679 Charpy test pieces and engraved to ensure identification but not yet machined to the dimensional requirements of ISO 148-3 [12].

3.2.2 Heat treatment of hot-rolled bars

The heat treatment to obtain the desired energy level of the test pieces was performed at VTN Witten GmbH (DE) in a vacuum-furnace. 1679 test pieces of the batch were heat-treated according to the following procedure:

Step 1: hardening at 850°C for 90 minutes
Step 2: quenching in oil
Step 3: first tempering at 280 °C for 180 minutes
Step 4: cool down in air
Step 5: second tempering at 280 °C for 180 minutes
Step 6 cool down in air.

The test pieces were distributed over three baskets. The baskets were stacked in the furnace equipped with a thermocouple to monitor the temperature. The measured temperature was within the tolerance of ± 6 °C as required by IfEP**.**

3.2.3 Final machining of Charpy test pieces

After heat treatment, the test pieces were machined to the final dimensions specified in ISO 148-3 [12] by IfEP. During this process the test piece numbers were engraved on one of the end faces. Finally the test pieces were notched using a milling process.

3.2.4 Packaging and storage

The test pieces were cleaned and packed in sets of five randomised test pieces, wrapped in anti-corrosion papers, packed in sealed plastic bags and shipped to the JRC (Geel, BE). After arrival, the 1565 test pieces (or 313 sets) of ERM-FA013cf were registered and stored at room temperature, pending distribution.

3.3 Process control

When all test pieces from the batch were fully machined, a random selection of 25 test pieces from across the whole batch was made. The dimensions of the 25 test pieces were checked against the criteria specified in ISO 148-3 [12]: 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. None of the test pieces was outside the ranges specified in ISO 148-3 [12].

25 test pieces were then impact tested using a pendulum type Zwick PSW 750 (nominal energy 450 J), verified in 2017 according to ISO 148-2 [1].
The tests were performed on 31/07/2018. The results are reported in the production report of IfEP [13]. The average *KV* of the 25 test pieces was 20.0 J, which is within the desired energy range (15 J - 25 J). The standard deviation of the test results (s = 0.50 J, *RSD* = 2.50 %) was below the 1.2 J acceptance criterion.
The variation was checked again during the characterisation tests at JRC (see Section 6).

4 Homogeneity

A key requirement for any reference material produced as a batch of units is equivalence between those units. In this respect, it is relevant whether the variation between units is significant compared to the uncertainty of the certified value, but it is not relevant whether this variation between units is significant compared to the variation of measurement results. Consequently, ISO 17034 [4] requires RM producers to quantify the between-unit variation. This aspect is covered in between-unit homogeneity studies.

The test pieces are sampled from the SBs, which should be sufficiently, but are never perfectly, homogeneous. Therefore, an appropriate homogeneity contribution u_h to the uncertainty of the certified value is required. u_h is related to s_h, the standard deviation between the test pieces in the SB (*test piece-to-test piece heterogeneity*), but also depends on the number of test pieces over which the *KV*-value is averaged. ISO 148-2 [1] specifies that the pendulum verification must be performed using five test pieces, which is why a CRM unit consists of a set of five test pieces. The appropriate uncertainty contribution must be an estimate of the *set-to-set heterogeneity* (u_h), which in the case of a set of five test pieces can be calculated as

$$u_h = \frac{s_h}{\sqrt{5}} \qquad \text{Equation 2}$$

For the assessment of homogeneity, the measurements performed at Institut für Eignungsprüfung GmbH (IfEP), Marl (DE) on 31/07/2018 during process control were used. As required for a homogeneity test, the test pieces were randomly selected from the whole batch. The number of test pieces tested (25) is sufficiently large to reflect the homogeneity of the full SB (1565 test pieces). The results are depicted in Annex 1.

Here, u_h is estimated from s_h, the standard deviation results obtained (s_h = 0.50 J). This leads to $u_h = \frac{s_h}{\sqrt{5}}$ = 0.22 J (1.12 %).

It can be noted that u_h is probably a slight overestimation, since it contains also the repeatability of the instrument. However, the latter cannot be separated or separately measured.

5 Stability

The stability of the absorbed energy of Charpy V-notch certified reference test pieces was first systematically investigated for test pieces of nominally 120 J by Pauwels et al., who did not observe measurable changes of absorbed energy [14]. 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) was obtained during the International Master Batch (IMB) project [15]. 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 seven reference pendulums over a three year period. None of the three regression slopes for the tested energy levels was statistically significant at the 5 % probability level. Given the large test piece-to-test piece heterogeneity and the limited number of test pieces, five in a CRM unit, the uncertainty contribution from instability is considered insignificant in comparison to that of homogeneity. A dedicated isochronous study (test temperature 18 °C, reference temperature -20 °C) was organised by the JRC using batches of 30 J, 80 J and 120 J from the same steel and showed, as expected, no significant change of the measured values. Uncertainty of stabilities for 120 months were calculated as 0.7 J - 2.8 J (1.8 % - 2.4 %). These uncertainties are entirely driven by the measurement precision and it was concluded that no uncertainty contribution for potential change was needed [16].

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

Rather than neglecting the stability issue, efforts are spent to better establish the stability of the certified values of batches of Charpy CRMs. Until such further notice, it is decided to specify a limited shelf-life. A period of 10 years is chosen, counting from the date of the characterisation tests on the SB. Since batch ERM-FA013cf was characterised on 03/03/2022, the certificate is valid until 3/2032.

6 Characterisation

The material characterisation is the process of determining the property value of a reference material. This was based on a direct comparison between the SB and an existing CRM (MB). This approach is valid as long as the two materials are certified for the same measurand and as long as the two materials are very similar.

The first condition is fulfilled as both MB and SB are certified for absorbed energy according to ISO-148-3. The second condition is fulfilled as the SB and MB are from equivalent steel types with relative low differences in the absorbed energy between SB and MB. Therefore, the measurement of the MB can indeed correct for undetected biases in the measurement of the SB.

6.1 Study setup

30 test pieces from ERM-FA013cf (sets 16, 82, 126, 164, 245, 274) were tested under repeatability conditions together with 25 test pieces from MB ERM-FA013ba [3] (sets 177, 194, 196, 244, 249), using the Instron Wolpert PW30 machine of the JRC, Directorate F – Health, Consumers and Reference Materials, a Charpy reference pendulum yearly verified according to procedures described in ISO 148-2 [1].

6.2 Measurement procedure used

Tests were performed on 03/03/2022 (laboratory temperature 20.0 ± 0.3 °C), in accordance with ISO 148-1:2016 [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 air resistance losses.

6.3 Evaluation of results

6.3.1 Technical evaluation

The results of the MB samples are within the range of acceptable bias allowed for reference pendulums specified in ISO 148-3 [12] (±5 % ($KV_{\mathrm{MB}} \geq 40$ J) or ±2 J ($KV_{\mathrm{MB}} < 40$ J). This proves that the pendulum used for characterisation was functioning properly.

After testing, all Charpy test pieces show 'first-strike' marks: these are the marks caused by the interaction between test piece and anvils during the first and intended hammer impact. Upon fracture, the broken halve test pieces loose contact with hammer and anvils and follow one of a variety of possible trajectories, away from the pendulum, depending on the properties of both pendulum and test material. It may occur that test pieces show 'second-strike' marks. These are marks caused by a second impact of the already broken halve test pieces back onto the anvils. This phenomenon has been described by Schmieder et al. [17]. None of the broken ERM-FA013cf or ERM-FA013ba test pieces showed second-strike marks.

Accumulation of debris from consecutive tests can lead to a trend in the measurement sequence. The results of MB and SB were tested for a linear trend using regression analysis. The regression of result versus position in the analytical sequence was significant on a 95 % confidence level, but not on a 99 % significance level. As any real trend should be also affect the measurements of the masterbatch (where no trend was seen), this trend was regarded an analytical artefact and all data were retained.

The accepted data obtained on individual test pieces are shown in Figure 3 and Annex 2.

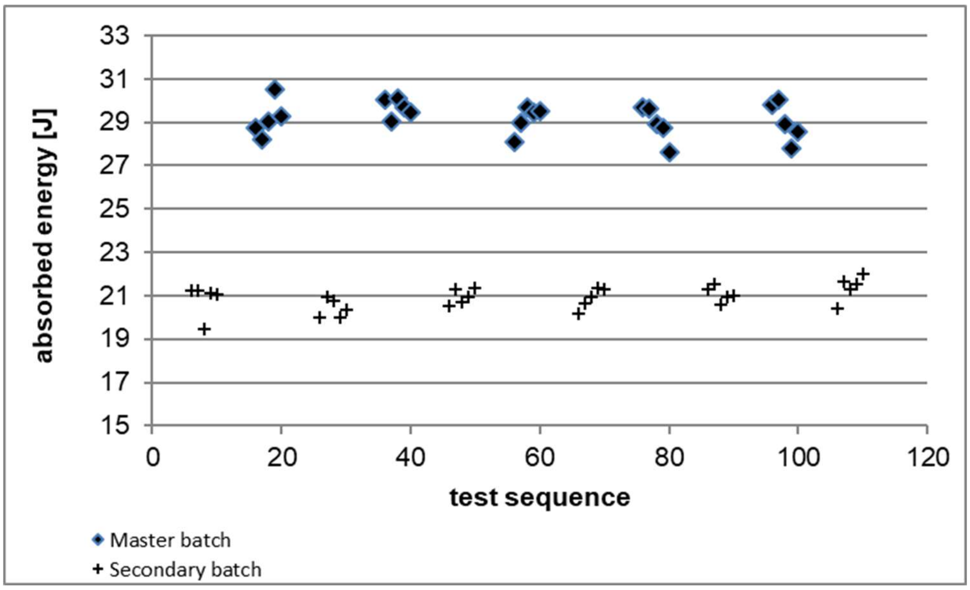

Figure 3: Absorbed energy values of 30 test pieces of ERM-FA013cf and 25 test pieces of ERM-FA013ba; data are displayed in the actual test sequence.

6.4 Statistical evaluation

The results of the measurements are summarised in Table 1.

Table 1: Data from the characterisation measurements

	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-FA013ba (MB)	25	29.17	0.74	2.52
ERM-FA013cf (SB)	30	20.90	0.57	2.71

The SB-results meet the ISO 148-3 [12] acceptance criteria for repeatability for a batch of reference materials ($s_{SB} \leq 2$ J for $KV_{SB} < 40$ J or ≤ 5 % for $KV_{SB} \geq 40$ J).

To calculate KV_{CRM} for ERM-FA013cf one needs KV_{MB} of the MB used, i.e. ERM-FA013ba [3]. Table 2 shows the main MB-data, taken from the Certificate of Analysis of ERM-FA013ba [3].

Table 2: Data from the certification of Master Batch ERM-FA013ba [3]

	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-FA013ba	28.46	0.23	0.81

From the data in Table 1 and Table 2, and using Equation 1, one readily obtains that KV_{CRM} = 20.4 J (rounding in accordance with uncertainty; see Table 4). The uncertainty associated with the characterisation of the SB, u_{char}, is assessed as in Equation 3 [11], which sums the relative uncertainties of the three factors in Equation 1:

$$u_{\text{char}} = KV_{\text{CRM}} \sqrt{\frac{u_{\text{MB}}^2}{KV_{\text{MB}}^2} + \frac{s_{\text{SB}}^2}{n_{\text{SB}} \cdot \overline{X}_{\text{SB}}^2} + \frac{s_{\text{MB}}^2}{n_{\text{MB}} \cdot \overline{X}_{\text{MB}}^2}} \quad \text{Equation 3}$$

$\overline{X}_{\text{SB}}$ and $\overline{X}_{\text{MB}}$ were obtained under repeatability conditions. Therefore, the uncertainty of the ratio $\overline{X}_{\text{SB}} / \overline{X}_{\text{MB}}$ is not affected by the contributions from reproducibility and bias of the pendulum used to compare MB and SB. Table 3 summarises the input quantities of the u_{char} uncertainty budget, their respective statistical properties, and shows how they were combined. The effective number of degrees of freedom (ν_{eff}) for u_{char} is obtained using the Welch-Satterthwaite equation from the combined uncertainty (u_c) and the individual uncertainty contributions (u_i) and their respective degrees of freedom (ν_i) (Equation 4) [9].

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

Table 3: Uncertainty budget of u_{char} for ERM-FA013cf

	Source of uncertainty	Measured value [J]	Standard uncertainty [J]	Probability distribution	Relative uncertainty [%]	Degrees of freedom
KV_{MB}	Certification of MB	28.46	0.23	normal	0.81	14
$\overline{X}_{\text{SB}}$	Comparison of SB and MB in repeatability conditions	20.90	0.10	normal	0.50	29
$\overline{X}_{\text{MB}}$		29.17	0.15	normal	0.50	24
					$u_{\text{char,rel}}$ [%] 1.07	37
					u_{char} [J] 0.219	

7 Value assignment

For this CRM, a certified value was assigned.
Certified values are values that fulfil the highest standards of accuracy. Full uncertainty budgets in accordance with ISO 17034 [4] and ISO Guide 35 [5] were established.

7.1 Certified value, combined and expanded uncertainty

The energy value obtained from the comparison of the SB with the MB as described in 6.3 was assigned as certified value.
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 4).

The relevant number of degrees of freedom calculated using the Welch-Satterthwaite equation (Equation 4, [9]), is sufficiently large (ν_{CRM} = 57) to justify the use of a coverage factor k = 2 to expand the confidence level to about 95 %. The obtained expanded uncertainty provides justification for the SB-MB approach followed: U_{CRM} is sufficiently smaller (U_{CRM} = 3.10 % or 0.7 J) than the verification criterion of 4 J for KV < 40 J or 10 % for KV ≥40 J for industrial pendulums [1] or even 2 J for KV < 40 J or 5 % for KV ≥40 J for reference pendulums [12].

Table 4: Uncertainty budget of KV_{CRM} for ERM-FA013cf

	Certified value [1] [J]	$u_{\text{char, rel}}$ [%]	$u_{\text{h, rel}}$ [%]	$u_{\text{CRM, rel}}$ [%]	$U_{\text{CRM, rel}}$ [%]	U_{CRM} [2] [J]
Absorbed energy	20.4	1.07	1.12	1.55	3.10	0.7

[1] Mean *KV* of the five test pieces

[2] Expanded (k = 2) and rounded uncertainty; uncertainties are always rounded up [18] and in a way that the rounding error corresponds to 3 % to 30 % of the uncertainty

8 Metrological traceability and commutability

8.1 Metrological traceability

Identity

Absorbed energy *KV* is an operationally defined measurand, and can only be obtained by following ISO 148-1:2016 [2], using an ISO-type striker of 2 mm tip radius at the specified test temperature.

Quantity value

The certified value of the MB ERM-FA013ba is traceable to the SI, since it was obtained from an intercomparison of qualified laboratories, thus combining results obtained from tests in accordance with the ISO 148-2 [1] and using instruments verified and calibrated with SI-traceable calibration tools. The uncertainty contribution from these individual calibrations all contribute to the uncertainty of the certified value of the MB.

The certified value of ERM-FA013cf is made traceable to the SI-traceable certified value of the MB by testing SB and MB test pieces under repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools. Therefore, the certified value of ERM-FA013cf is traceable to the International System of Units (SI) via the corresponding Master Batch ERM-FA013ba of a similar nominal absorbed energy.

8.2 Commutability

The concept of commutability of a reference material is defined by the VIM [19] as:
"property of a reference material, demonstrated by the closeness of agreement between the relation among the measurement results for a stated quantity in this material, obtained according to two given measurement procedures, and the relation obtained among the measurement results for other specified materials"

The samples of ERM-FA013cf are made from commercially sourced alloy steel which behaves like any other alloy steel of the same energy level. The material is therefore commutable with other alloy steel samples.

The certified value of this CRM is operationally defined and can only be obtained when tested in accordance with ISO 148-1:2016 [2]. The certified values are not valid when the test pieces are broken with an ASTM-type striker of 8 mm tip radius [6].
If the CRM nevertheless is used with a different method, then the user has to assess the commutability of the reference material. For further guidance, see Application Note 8 [20].

9 Instructions for use

9.1 Intended use

Test pieces of ERM-FA013cf are 'certified reference test pieces' as defined in ISO 148-3 [12]. 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 when testing limited numbers of test pieces, also the degrees of freedom (ν_{CRM}) of the uncertainty, all given on page 1 of the certificate.

9.2 Sample preparation

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

The following cleaning and conditioning procedure is considered to be good practice.

1. Use absorbent cleaning-tissue and technically pure ethanol to clean and degrease each specimen. Pay particular attention to the notch of the sample.
2. Once samples are degreased, only manipulate the samples wearing clean gloves. This is to prevent development of corrosion between the time of cleaning and the actual test.
3. Before testing, bring the specimens to the test temperature (20 ± 2) °C. To ensure thermal equilibrium, move the specimens to the test laboratory at least 3 h before the tests.

9.3 Pendulum impact tests

After cleaning and equilibration, the five test pieces constituting a CRM unit need to be broken with a pendulum impact test machine in accordance with ISO 148-2 [1]. 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, during testing, the anvils must be checked regularly and if debris is found, it must be removed. The uncertainty of the certified value applies to the mean of the five *KV*-values.

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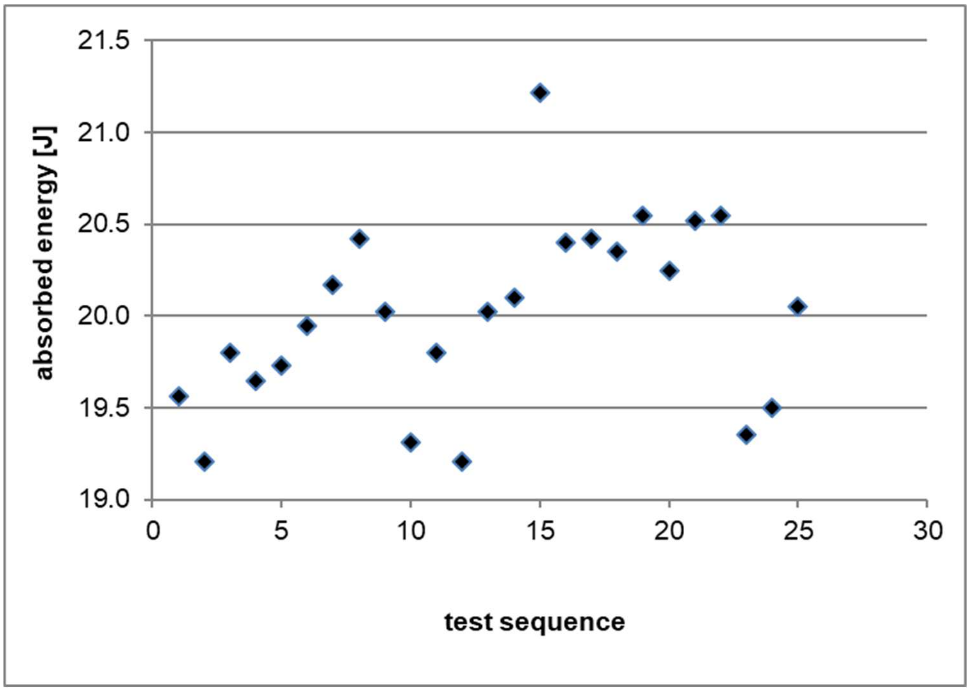
Annex 1

Results of tests for the homogeneity assessment of ERM-FA013cf.



Annex 2

Results of characterisation measurements of ERM-FA013cf and ERM-FA013ba as measured according to ISO 148-1 [2] at JRC Geel 03/03/2022.

	Master Batch ERM-FA013ba	**Secondary Batch ERM-FA013cf**
	***KV* [J]**	***KV* [J]**
1	28.76	21.22
2	28.23	21.22
3	29.02	19.43
4	30.49	21.10
5	29.29	21.04
6	30.02	19.96
7	29.02	20.92
8	30.09	20.74
9	29.69	19.96
10	29.42	20.32
11	28.10	20.50
12	28.95	21.28
13	29.69	20.68
14	29.42	20.92
15	29.49	21.34
16	29.69	20.14
17	29.62	20.62
18	28.89	20.92
19	28.76	21.34
20	27.64	21.28
21	29.82	21.28
22	30.02	21.52
23	28.89	20.56
24	27.77	20.92
25	28.56	20.98
26		20.38
27		21.64
28		21.28
29		21.52
30		22.01
Mean [J]	29.17	20.9
Standard deviation [J]	0.74	0.57
***RSD* [%]**	2.52	2.71

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