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Certification of the absorbed energy of Charpy V-notch reference test pieces for tests at 20 °C: ERM®-FA013ci

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Joint Research Centre

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Contents

Abstract

This certification report describes the processing and characterisation of ERM®-FA013ci, 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-FA013ci 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-FA013ci 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.

Acknowledgements
The authors would like to acknowledge the support received from colleagues of JRC for the measuring and reviewing of the certification project.

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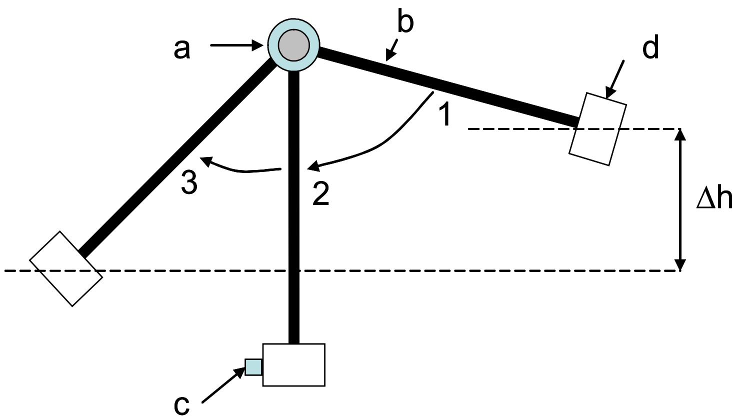
1 Introduction

1.1 Background

1.1.1 The Charpy pendulum impact test

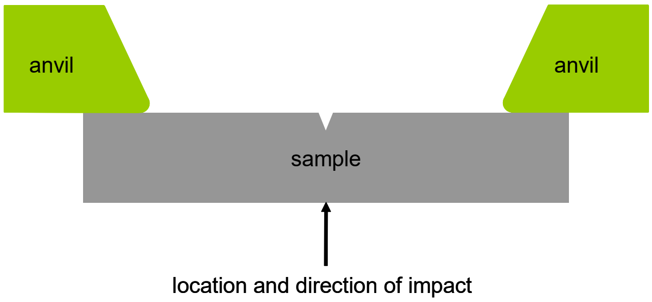
The Charpy pendulum impact test is designed to assess the resistance of a material to shock loading. The test consists of breaking a notched test piece of the test material using a hammer rotating around a fixed horizontal axis and is schematically presented in Figure1.

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 ($\bar{x}_{SB}$) 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 test pieces of the SB, is estimated by comparing the mean KV-value of a number of test pieces of the MB ($\bar{x}_{MB}$), 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]:

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

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 $\frac{KV_{MB}}{\bar{x}_{MB}}$ must be close to 1. ISO 148-3 [12] allows a difference of 5 % ($KV_{MB} \geq 40$ J) or 2 J ($KV_{MB} < 40$ J) between KV_{MB} and $\bar{x}_{MB}$.

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

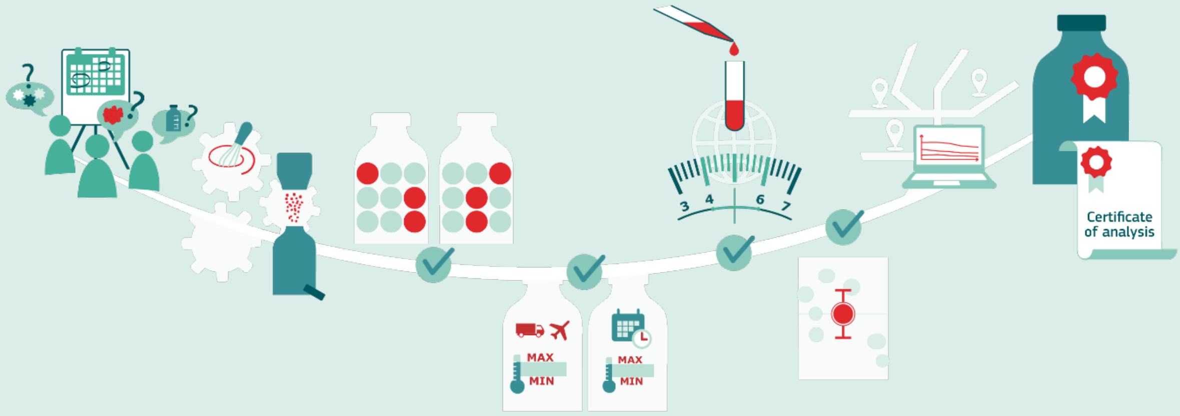
1.2 Choice of the material

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 [13] and pressure equipment [14]. 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 an AISI4340/1.6565 type steel, also an AISI4340/1.6565 was chosen for the SB.

1.3 Outline of the CRM project

Box 1. Reference material production



Reference material (RM) production is defined in ISO 17034 [4] as a project comprising planning and processing of the material, followed by homogeneity and stability testing, characterisation and assigning of one or more property values. Depending on the intended use of the RM a commutability study is carried out.

For certified reference materials (CRMs) a certificate is issued while for RMs a product information sheet is issued by the reference material producer (RMP).

CRMs and RMs are distributed globally and the stability of their assigned values is monitored throughout the life-time of the material.

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.

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

2 Participants

2.1 Project management and data evaluation

European Commission, Joint Research Centre, Directorate F – Health, Consumers and 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 ISO Guide 34:2009 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

Sirris, Zwijnaarde (BE) (measurements under the scope of ISO/IEC 17025:2017 accreditation BELAC No. 232-TEST).

3 Material processing and process control

Box 2. Reference material processing

RM processing covers the raw material conversion into a homogenous and stable material. In the case of Charpy test pieces, this includes cutting of test pieces, heat treatment and creating the V-notch.

3.1 Origin of the starting material

The ERM-FA013ci test pieces were prepared from bars of AISI4340/1.6565 steel produced by Tata Steel International Germany GmbH. One round bar (length 3.00 m) was cut into 36 discs and marked 1-36. All 36 discs were normalised at 900 °C for 150 min.

3.2 Processing

3.2.1 Machining of Charpy test pieces

Machining of the test pieces was performed by IfEP. 12 discs were selected to machine the Charpy tests pieces. These were cut into 1709 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 (Witten, DE) in a vacuum-furnace. 1652 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.

For the heat treatment, 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 assorted in sets of five randomised test pieces and wrapped in anticorrosion paper. These wrapped sets were packed in a sealed plastic bag, and shipped to the JRC (Geel, BE). After arrival, the 1565 test pieces (or 313 sets) of ERM-FA013ci were registered and stored at room temperature, pending distribution.

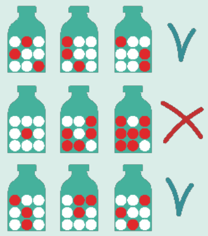
For the purpose of this report, the term 'unit' refers to one set of five Charpy impact test specimens of ERM-FA013ci.

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/-0.03) 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 2019 according to ISO 148-3 [12].
The tests were performed on 17/09/2019. The results are reported in the production report of IfEP [15]. The average *KV* of the 25 test pieces was 19.88 J, which is within the desired energy range (15 J - 25 J). The standard deviation of the test results (s = 0.60 J, *RSD* = 3.0 %) was below the 1.2 J acceptance criterion.
The variation was checked again during the characterisation tests (see Section 6).

4 Homogeneity

Box 3. Homogeneity assessment



A key requirement for any RM produced as a batch of units is equivalence between those units. It is important to know how much the variation between units contributes to the uncertainty of the certified value. Consequently, ISO 17034 [4] requires RMPs to quantify the between-unit variation in homogeneity studies.

The within-unit homogeneity is correlated to the minimum sample size, which is the minimum amount of sample that is, for a given measurand, representative of the whole unit and that should be used in an analysis. Using sample intakes equal to or above the minimum sample size guarantees the assigned value within its stated uncertainty. The concept of within-unit homogeneity does not apply to Charpy test specimens, as no subsampling is possible.

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 17/09/2019 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 = 0.60 J). This leads to $u_h = \frac{s_h}{\sqrt{5}}$ = 0.27 J (1.35 %).

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

Box 4. Stability assessment

Stability testing is necessary to establish the conditions for storage as well as the transport conditions of the RMs to the customers. During transport, especially in summer, temperatures up to 60 °C can be reached, and stability under these conditions must be demonstrated if the RMs are to be transported without any additional cooling.

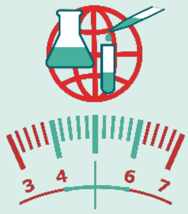
The stability of the absorbed energy of Charpy V-notch certified reference test pieces was first systematically investigated for test pieces of nominally 120 J by Pauwels et al., who did not observe measurable changes of absorbed energy [16]. Additional evidence for the stability of the reference test 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 [17]. 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 [18].

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-FA013ci was characterised on 13/09/2022, the certificate is valid until 09/2032.

6 Characterisation

Box 5. Reference material characterisation



Material characterisation is the process of determining the property value(s) of a RM. While ISO 17034 [4] allows to characterise a RM in various ways. Quality management procedures of the JRC are more stringent and allow characterisation only by either interlaboratory comparison, the use of a primary method confirmed by independent analysis or value transfer using a single measurement method in a single laboratory.

The material characterisation was based on value transfer from the MB to the SB using a single measurement method (ISO 148-1) in a single laboratory. 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-FA013ci (sets 16, 82, 126, 164, 245, 274) were tested under repeatability conditions together with 25 test pieces from MB ERM-FA013ba [3] (sets 245, 253, 254, 257, 258), using a pendulum type Zwick PSW 750, a Charpy pendulum verified in May 2022 according to procedures described in ISO 148-2 [1].

6.2 Measurement procedure used

Tests were performed on 13/09/2022 (laboratory temperature 21 °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.

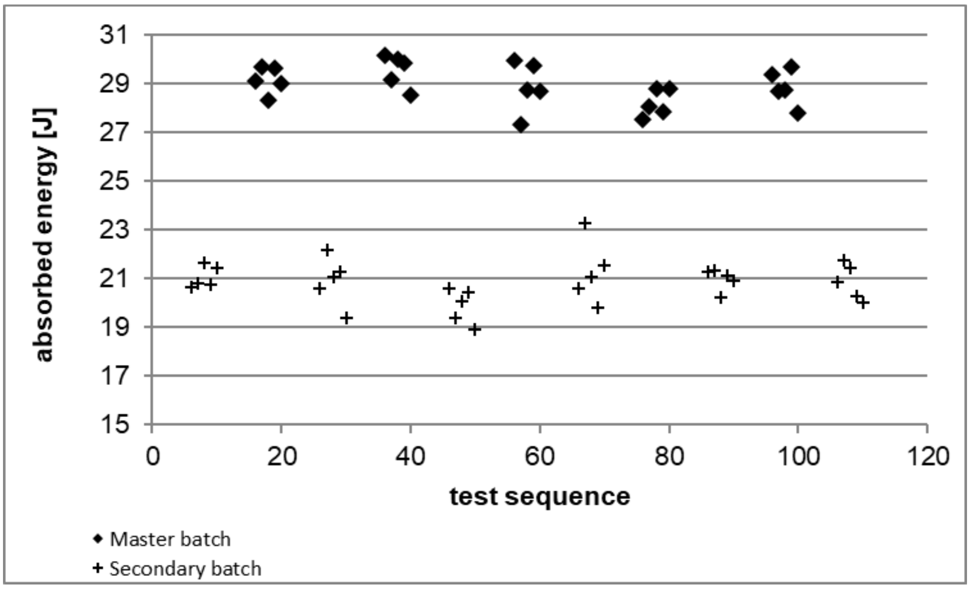
6.3 Evaluation of results

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_{MB} ≥ 40 J) or ±2 J (KV_{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 halves of the test pieces back onto the anvils. This phenomenon has been described by Schmieder et al. [19]. None of the broken ERM-FA013ci 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 not significant on a 95 % significance level.
The accepted data obtained on individual test pieces are shown in Figure 3 and Annex 2.
The sequences of the test pieces showed one outlying individual value for set 274 (23.28 J). This value was retained, as the range of all samples of this set (3.49 J) was still well below the acceptable range for indirect verification (ISO 148-2). In addition, all specimens of set 245 showed consistent low results (average 19.9 J) and the average of the set was flagged as a outlier on a 99 % confidence level using the Grubbs procedure. However, as the whole batch still fulfils the requirements of homogeneity set in ISO 148-3 (measured standard deviation is 0.9 J, allowed standard deviation 2 J) and as the average of this set also agree with the

certified values, this outlying result is not an indication of excessive heterogeneity. The data obtained were therefore still in line with the requirements for reference specimens as specified in ISO 148-3.

Figure 3. Absorbed energy values of 30 test pieces of ERM-FA013ci 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_{MB}, RSD_{SB} [%]
ERM-FA013ba (MB)	25	28.92	0.81	2.80
ERM-FA013ci (SB)	30	20.82	0.88	4.23

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

To calculate KV_{CRM} for ERM-FA013ci one needs KV_{MB} of the MB used, i.e. ERM-FA013ba [3]. Table 2 shows the main MB-data, taken from the certification report 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} = 20.5 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:

$$u_{char} = KV_{CRM}\sqrt{\frac{u_{MB}^2}{KV_{MB}^2} + \frac{s_{SB}^2}{n_{SB}\bar{x}_{SB}^2} + \frac{s_{MB}^2}{n_{MB}\bar{x}_{MB}^2}} \qquad \text{Equation 3}$$

$\bar{X}_{SB}$ 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 (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].

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

Table 3. Uncertainty budget of u_{char} for ERM-FA013ci

	Source of uncertainty	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
$\bar{X}_{SB}$	Comparison of SB and MB in repeatability conditions	20.82	0.16	unimodal	0.77	29
$\bar{X}_{MB}$		28.92	0.16	normal	0.56	24
$u_{char,rel}$ [%]					1.25	52
u_{char} [J]					0.26	

7 Value assignment

Box 6. Assignment of values to a reference material

Based on the outcome of characterisation measurements three types of values can be assigned, namely certified, indicative or additional material information values.

Certified values are values that fulfil the highest standards of accuracy. Procedures at JRC Directorate F require a sufficient number of datasets to assign certified values. Full uncertainty budgets in accordance with ISO 17034 [4] and ISO Guide 35 [5] are required. Certified values of a CRM can be used for calibration and trueness controls.
Indicative values are values where either the uncertainty is deemed too large or too few independent datasets are available to allow certification. Indicative values of an RM can be used for statistical quality control (homogeneity and stability has been assessed) but not for calibration, demonstration of method or laboratory proficiency or method trueness.
Additional material information values are values for which homogeneity and stability has usually not been assessed and insufficient data for characterisation is available. Consequently, an estimate of the reliability of the values is not possible and no uncertainty is given. Additional material information values cannot be used for calibration, demonstration of method or laboratory proficiency or method trueness. They can be used to e.g. anticipate possible interferences in measurement processes.

For this CRM, a certified value was assigned.

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} = 61) 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} = 3.68% or 0.8 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-FA013ci

	Certified value 1) [J]	$u_{char, rel}$ [%]	$u_{h, rel}$ [%]	$u_{CRM, rel}$ [%]	$U_{CRM, rel}$ [%]	U_{CRM} 2) [J]
Absorbed energy	20.5	1.25	1.35	1.84	3.68	0.8

1) Mean *KV* of the five test pieces
2) Expanded (k = 2) and rounded uncertainty; uncertainties are always rounded up [20] 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

Box 7. Metrological traceability

Metrological traceability of measurement results is a key requirement for ensuring the comparability of data. As CRMs are used to make measurement results traceable, metrological traceability of its certified values to a stated reference is essential.

The certified value of a CRM is metrologically traceable if the measurements used for establishing it can be related to a reference through an unbroken chain of calibrations.

This requires that these measurements

• refer to the same property (e.g. Pb) and the same (kind of) quantity (e.g. Pb content),
• result in a number and its uncertainty (e.g. 6 ± 2) expressed in the same measurement unit (e.g. µg/kg).

The concept of traceability rests on several anchor points, namely identity, quantity value and measurement unit. The identity of a measurand can be defined by its structure alone or can be operationally defined, the quantity value of the measurand can refer to the SI, or to other appropriate references.

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.

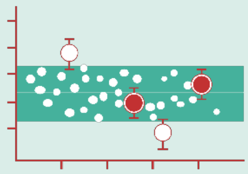
The certified value of ERM-FA013ci is made traceable to the SI-traceable certified value of the MB by testing SB and MB test pieces under repeatability conditions on an impact pendulum verified and calibrated with SI-traceably calibrated tools. Therefore, the certified value of ERM-FA013ci is traceable to the International System of Units (SI) via the corresponding Master Batch ERM-FA013ba of a similar nominal absorbed energy.

8.2 Commutability

Box 8. Commutability

Commutability is a prerequisite for RMs intended to be used for calibration or quality control of different measurement procedures targeting the same measurand. The concept of commutability of a RM is defined by the VIM [21] 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*"



Commutability is a property of an RM indicating how well an RM mimics the characteristics of a typical routine sample in various measurement procedures for a stated measurand.

The same RM may be commutable for some measurement procedures but non-commutable for others. A commutability statement is therefore only valid for the mentioned measurement procedure(s).

The samples of ERM-FA013ci 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 [22].

9 Instructions for use

9.1 Storage conditions

The material should be stored at (18 ± 5) °C in the dark and dry place.

For more information regarding the shelf life of reference materials please see ERM Application Note 7, available on https://crm.jrc.ec.europa.eu/e/132/User-support-Application-Notes.

Note that the European Commission cannot be held responsible for changes that may happen during storage of the material at the customer's premises, especially of opened samples.

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.

9.4 Use of the certified value

Test pieces of ERM-FA013ci 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.

10 Conclusions

ERM-FA013ci is a matrix material certified for absorbed energy when subjected to the Charpy impact test according to ISO 148-1:2016 for strikers with a 2 mm tip radius. This material was produced and certified in accordance with ISO 17034:2016 [4] and ISO Guide 35:2017 [5]. ERM-FA013ci was produced within the scope of ISO 17034 accreditation.
The following value was assigned:

Table 5. Value assigned for ERM-FA013ci

Steel Charpy V-notch test pieces		
	Certified value [2)] [J]	Uncertainty [3)] [J]
Absorbed energy (*KV*) [1)]	20.5	0.8
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-FA013ci 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 61 effective degrees of freedom.		

References

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List of abbreviations and definitions

AISI	American Iron and Steel Institute
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
$\mathrm{RSD}_{\mathrm{MB}}$	Relative standard deviation of the n_{MB} results of the test pieces of the Master Batch tested for the certification of the Secondary Batch
$\mathrm{RSD}_{\mathrm{SB}}$	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
$\bar{x}_{\mathrm{MB}}$	Mean *KV* value of the n_{MB} measurements on test pieces of the Master Batch tested when characterising the Secondary Batch
$\bar{x}_{\mathrm{SB}}$	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

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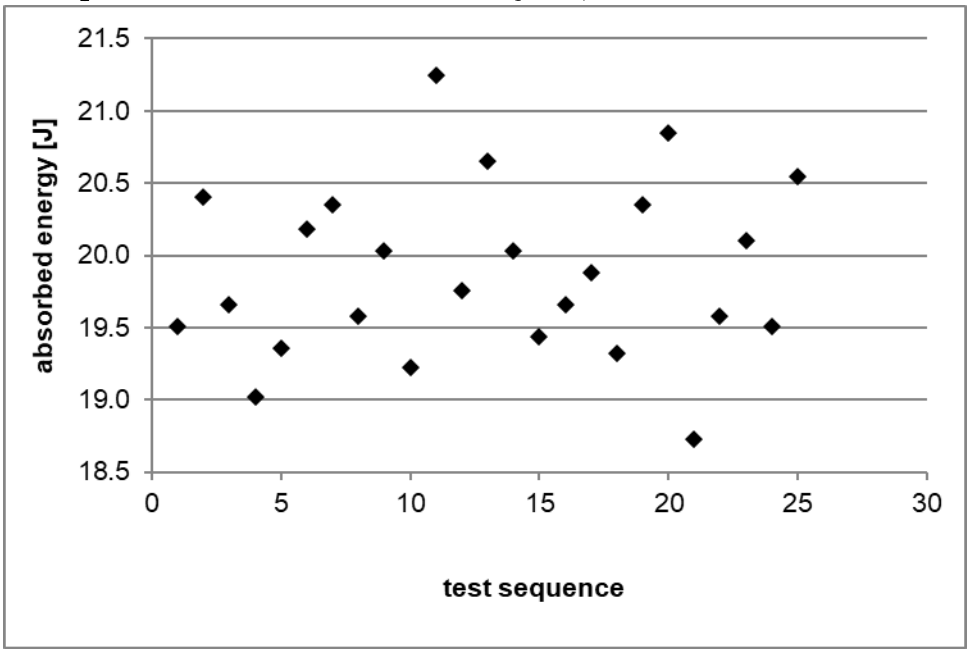
Annexes

Annex 1: Results of tests for the homogeneity assessment of ERM-FA013ci.

Figure 1.1: Results of tests for the homogeneity assessment of ERM FA013ci.



Annex 2: Results of characterisation measurements of ERM-FA013ci and ERM-FA013ba as measured according to ISO 148-1 [2] at Sirris on 13/09/2022.

Table 2.1. Results of characterisation measurements of ERM FA013ci and ERM FA013ba as measured according to ISO 148-1 [2] at Sirris on 13/09/2022.

Result number	Master Batch ERM-FA013ba *KV* [J]	Secondary Batch ERM-FA013ci *KV* [J]
1	29.10	20.64
2	29.67	20.82
3	28.31	21.63
4	29.62	20.75
5	28.98	21.41
6	30.14	20.60
7	29.14	22.15
8	30.02	21.08
9	29.86	21.27
10	28.50	19.40
11	29.94	20.57
12	27.32	19.40
13	28.71	20.09
14	29.74	20.42
15	28.66	18.89
16	27.55	20.60
17	28.06	23.28
18	28.81	21.08
19	27.82	19.79
20	28.77	21.56
21	29.34	21.26
22	28.69	21.30
23	28.73	20.23
24	29.70	21.11
25	27.78	20.92
26		20.85
27		21.77
28		21.44
29		20.26
30		20.01
Mean [J]	28.92	20.82
Standard deviation [J]	0.81	0.88
RSD [%]	2.80	4.23

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