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**Metallic materials — Charpy  
pendulum impact test —**

**Part 3:  
Preparation and characterization of  
Charpy V-notch test pieces for indirect  
verification of pendulum impact  
machines**

*Matériaux métalliques — Essai de flexion par choc sur éprouvette  
Charpy —*

*Partie 3: Préparation et caractérisation des éprouvettes Charpy  
à entaille en V pour la vérification indirecte des machines d'essai  
mouton-pendule*





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ISO copyright office  
Ch. de Blandonnet 8 • CP 401  
CH-1214 Vernier, Geneva, Switzerland  
Tel. +41 22 749 01 11  
Fax +41 22 749 09 47  
copyright@iso.org  
www.iso.org

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see [www.iso.org/directives](http://www.iso.org/directives)).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see [www.iso.org/patents](http://www.iso.org/patents)).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: [www.iso.org/iso/foreword.html](http://www.iso.org/iso/foreword.html).

The committee responsible for this document is ISO/TC 164, *Mechanical testing of metals*, Subcommittee SC 4, *Toughness testing — Fracture (F), Pendulum (P), Tear (T)*.

This third edition cancels and replaces the second edition (ISO 148-3:2008), which has been technically revised.

ISO 148 consists of the following parts, under the general title *Metallic materials — Charpy pendulum impact test*:

- *Part 1: Test method*
- *Part 2: Verification of testing machines*
- *Part 3: Preparation and characterization of Charpy V-notch test pieces for indirect verification of pendulum impact machines*

## Introduction

The suitability of a pendulum impact testing machine for acceptance testing of metallic materials has usually been based on a calibration of its scale and verification of compliance with specified dimensions, such as the shape and spacing of the anvils supporting the test piece. The scale calibration is commonly verified by measuring the mass of the pendulum and its elevation at various scale readings. This procedure for evaluation of machines had the distinct advantage of requiring only measurements of quantities that could be traced to national standards. The objective nature of these traceable measurements minimized the necessity for arbitration regarding the suitability of the machines for material acceptance tests.

However, sometimes two machines that had been evaluated by the direct-verification procedures described above, and which met all dimensional requirements, were found to give significantly different impact values when testing test pieces of the same material.

This difference was commercially important when values obtained using one machine met the material specification, while the values obtained using the other machine did not. To avoid such disagreements, some purchasers of materials added the requirement that all pendulum impact testing machines used for acceptance testing of material sold to them should be indirectly verified by testing reference test pieces supplied by them. A machine was considered acceptable only if the values obtained using the machine agreed, within specified limits, with the value furnished with the reference test pieces.

Successful experience in the use of reference test pieces led to the requirement in ISO 148-2 that indirect verification should be performed using reference test pieces in addition to direct verification. Other standards and codes also require indirect verification using reference test pieces; for example, EN 10045-2<sup>[1]</sup> (now obsolete) and ASTM E23<sup>[2]</sup> require the use of reference test pieces. The purpose of this part of ISO 148 is to specify the requirements, preparation and methods for qualifying test pieces used for the indirect verification of pendulum impact testing machines.



# Metallic materials — Charpy pendulum impact test —

## Part 3:

# Preparation and characterization of Charpy V-notch test pieces for indirect verification of pendulum impact machines

## 1 Scope

This part of ISO 148 specifies the requirements, preparation and methods for qualifying test pieces used for the indirect verification of pendulum impact testing machines in accordance with ISO 148-2.

It specifies notched test pieces with nominal dimensions identical to those specified in ISO 148-1; however, the tolerances are more stringent.

NOTE 1 The chemical composition or heat treatment, or both, are varied according to the energy level desired.

NOTE 2 Reference test pieces are qualified on reference pendulum impact testing machines which are also described in this part of ISO 148.

## 2 Normative references

The following referenced documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 148-1, *Metallic materials — Charpy pendulum impact test — Part 1: Test method*

ISO 148-2, *Metallic materials — Charpy pendulum impact test — Part 2: Verification of testing machines*

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

### 3.1 Definitions pertaining to the machine

#### 3.1.1

##### **industrial machine**

pendulum impact testing machine used for industrial, general or most research-laboratory testing of metallic materials

Note 1 to entry: These machines are not used to establish reference values.

#### 3.1.2

##### **reference machine**

pendulum impact testing machine used to determine certified values for batches of reference test pieces

## 3.2 Definitions pertaining to energy

### 3.2.1

#### total absorbed energy

$K_T$   
total absorbed energy required to break a test piece with a pendulum impact testing machine, which is not corrected for any losses of energy

Note 1 to entry: It is equal to the difference in the potential energy from the starting position of the pendulum to the end of the first half swing during which the test piece is broken.

### 3.2.2

#### absorbed energy

$K$   
energy required to break a test piece with a pendulum impact testing machine, after correction for energy losses

Note 1 to entry: The letter V or U is used to indicate the notch geometry, i.e.  $KV$  or  $KU$ . The number 2 or 8 is used as a subscript to indicate the radius of the striking edge of the striker, for example  $KV_2$ .

### 3.2.3

#### reference absorbed energy

$K_R$   
certified value of absorbed energy assigned to the test pieces used to verify the performance of pendulum impact testing machines

## 3.3 Definitions related to groups of test pieces

### 3.3.1

#### batch

definite quantity of reference test pieces manufactured under identical conditions of production, with a common certified absorbed energy

### 3.3.2

#### set

group of test pieces chosen at random from a batch

#### 3.3.2.1

##### characterization set

set of test pieces taken from a batch and used to determine the reference energy of the batch

#### 3.3.2.2

##### reference set

set of test pieces used to verify a pendulum impact testing machine

## 3.4 Definitions pertaining to test pieces

### 3.4.1

#### width

$W$   
distance between the notched face and the opposite face

Note 1 to entry: In previous versions of the ISO 148 series (prior to 2016), the distance between the notched face and the opposite face was specified as "height". Changing this dimension to "width" makes ISO 148-1 consistent with the terminology used in other ISO fracture standards.



### 3.4.2 thickness

*B*

dimension perpendicular to the width and parallel to the notch

Note 1 to entry: In previous versions of the ISO 148 series (prior to 2016), the dimension perpendicular to the width that is parallel to the notch was specified as “width”. Changing this dimension to “thickness” makes ISO 148-1 consistent with the terminology used in other ISO fracture standards.

### 3.4.3 length

*L*

largest dimension perpendicular to the notch

### 3.4.4 reference test piece

impact test piece used to verify the suitability of a pendulum impact testing machine by comparing the indicated absorbed energy measured by that machine to the reference absorbed energy associated with the test pieces

### 3.4.5 certified reference test piece

impact test piece accompanied by a certificate providing the certified absorbed energy value,  $K_R$ , and its uncertainty at a stated level of confidence

Note 1 to entry: The certified reference value is the value determined by a certified national or international body, or by an organization accredited for the production of certified Charpy reference test pieces in accordance with ISO Guide 34<sup>[3]</sup>, following the procedures described in this part of ISO 148.

## 4 Symbols and abbreviated terms

**Table 1 — Symbols/abbreviated terms and their designations and units**

| Symbol/<br>abbreviated term | Unit | Designation   |
|-----------------------------|------|---|
| CRM                         | —    | certified reference material  |
| GUM                         | —    | guide to the expression of uncertainty in measurement   |
| <i>k</i>                    | —    | coverage factor   |
| <i>K</i>                    | J    | absorbed energy   |
| $K_T$                       | J    | total absorbed energy   |
| $K_R$                       | J    | reference absorbed energy of a set of Charpy reference test pieces  |
| $K_V$                       | J    | absorbed energy as measured in accordance with ISO 148-1 on a V-notched sample  |
| $KV_{char}$                 | J    | $KV$ value as determined for a batch of V-notched Charpy reference materials in a batch certification characterization exercise |
| $KV_{PB}$                   | J    | certified $K_V$ value of a PB reference material  |
| $KV_R$                      | J    | certified $K_V$ value of a Charpy reference material  |
| $KV_{SB}$                   | J    | certified $K_V$ value of an SB reference material   |
| $n_{hom}$                   | —    | number of samples tested for the homogeneity assessment   |
| $n_{PB}$                    | —    | number of PB specimens used to compare SB with PB   |
| $n_{SB}$                    | —    | number of SB specimens used to compare SB with PB   |
| $n_V$                       | —    | number of reference samples tested for the indirect verification of a pendulum impact testing machine                           |
| <i>p</i>                    | —    | number of laboratories/instruments participating in a laboratory comparison   |

**Table 1** (continued)

| Symbol/<br>abbreviated term | Unit | Designation   |
|-----------------------------|------|---|
| PB                          | —    | primary batch   |
| REMCO                       | —    | ISO Committee on Reference Materials  |
| RM                          | —    | reference material  |
| SB                          | —    | secondary batch   |
| $s_p$                       | J    | standard deviation of the mean $K_V$ values obtained at $p$ laboratories  |
| $s_{PB}$                    | J    | standard deviation of results obtained on $n_{PB}$ PB samples when comparing them with $n_{SB}$ SB samples                            |
| $s_{RM}$                    | J    | standard deviation of the $K_V$ values obtained on $n_{hom}$ samples in the homogeneity assessment of the batch of reference material |
| $u_{char}$                  | J    | standard uncertainty of $KV_{char}$   |
| $u_{char,PB}$               | J    | standard uncertainty of $KV_{char}$ for a PB  |
| $u_{char,SB}$               | J    | standard uncertainty of $KV_{char}$ for an SB   |
| $u_{hom}$                   | J    | standard uncertainty of the homogeneity assessment of the reference material  |
| $u_{lts}$                   | J    | standard uncertainty of the long-term-stability assessment of the reference material  |
| $u_{RM}$                    | J    | standard uncertainty of the certified value of a reference material used for indirect verification                                    |
| $U_{RM}$                    | J    | expanded uncertainty of the certified value of a reference material at a confidence level of about 95 %                               |
| $u_{sts}$                   | J    | standard uncertainty of the short-term-stability assessment of a reference material   |
| $u_{\bar{X}_{PB}}$          | J    | standard uncertainty of $\bar{X}_{PB}$  |
| $u_{\bar{X}_{SB}}$          | J    | standard uncertainty of $\bar{X}_{SB}$  |
| $\bar{X}_{PB}$              | J    | mean of $n_{PB}$ specimens used to compare SB with PB   |
| $\bar{X}_{SB}$              | J    | mean of $n_{SB}$ specimens used to compare SB with PB   |
| $\delta KV_{hom}$           | J    | part of the error of the measured $KV$ value due to batch heterogeneity   |
| $\delta KV_{lts}$           | J    | part of the error of the measured $KV$ value due to long-term instability   |
| $\delta KV_{sts}$           | J    | part of the error of the measured $KV$ value due to short-term instability  |
| $\nu_{char}$                | —    | degrees of freedom corresponding to $u_{char}$  |
| $\nu_{hom}$                 | —    | degrees of freedom corresponding to $u_{hom}$   |
| $\nu_{RM}$                  | —    | degrees of freedom corresponding to $u_{RM}$  |

## 5 Reference testing machine

### 5.1 Characteristics

#### 5.1.1 General

The characteristics of reference machines used to determine the reference energy of reference test pieces shall comply with the requirements of ISO 148-2 except as modified below.

### 5.1.2 Geometrical characteristics (see [Table 2](#) and [Figures 1](#) and [2](#))

The following geometrical characteristics apply:

- a) the radius of the anvils shall be  $\left(1^{+0,10}_{0,00}\right)$  mm ;
- b) the distance between the anvils shall be  $\left(40^{+0,10}_{0,00}\right)$  mm ;
- c) the striking edge shall be within  $\pm 0,25$  mm of the plane of symmetry of the anvils.

**Table 2 — Geometrical characteristics**

| Reference number <sup>a</sup>   | Designation  | Value | Tolerance        | Units |
|---|--|-------|------------------|-------|
| 1   | Length of test piece   | 55,00 | +0,00 *<br>-0,30 | mm    |
| 2   | Half-length of test piece                                      | 27,5  | ±0,2 *           | mm    |
| 3   | Width of test piece  | 10,00 | ±0,06            | mm    |
| 4   | Thickness of test piece  | 10,00 | ±0,07 *          | mm    |
| 5   | Ligament length  | 8,00  | ±0,06            | mm    |
| 6   | Angle of notch   | 45,0  | ±1,0 *           | °     |
| 7   | Radius at base of notch  | 0,250 | ±0,025           | mm    |
| 8   | Angle between adjacent faces                                   | 90,00 | ±0,15 *          | °     |
| 9   | Angle between plane of symmetry of notch and longitudinal axis | 90    | ±2               | °     |
| 10  | Radius of anvils   | 1,00  | +0,10 *<br>-0,00 | mm    |
| 11  | Angle of taper of anvils                                       | 11    | ±1,0             | °     |
| 12  | Distance between anvils  | 40,00 | +0,10 *<br>-0,00 | mm    |
| 13  | Distance of striking edge from plane of symmetry of anvils     | —     | ±0,25 *          | mm    |
| 14  | Angle of striker   | 30    | ±1               | °     |
| 15A   | Radius of striking edge of 2 mm striker                        | 2,00  | +0,20 *<br>-0,00 | mm    |
| 15B   | Radius of striking edge of 8 mm striker                        | 8,00  | ±0,05            | mm    |
| 15C   | Radius of shoulder of 8 mm striker                             | 0,25  | +0,50<br>-0,05   | mm    |
| 15D   | Width of striking edge of 8 mm striker                         | 4,00  | ±0,20            | mm    |
| NOTE 1 Tolerances followed by an asterisk * are tighter than those in ISO 148-1 or ISO 148-2. |  |       |                  |       |
| NOTE 2 See <a href="#">Figures 1</a> and <a href="#">2</a> .                                  |  |       |                  |       |
| <sup>a</sup> See <a href="#">Figure 1</a> .   |  |       |                  |       |

### 5.1.3 Capacity

The capacity of a reference machine (nominal initial potential energy) shall be appropriate for the specimens to be tested and certified with it. Certified energies shall not exceed 80 % of the machine capacity.

### 5.1.4 Hardness

The portions of the striker and the anvils (see [Figure 1](#)) that contact the specimen and apply or react to the impacting force shall have a minimum hardness of 56 HRC.

**5.1.5 Vibration**

Ensure that the reference machine is not subjected to external vibrations induced by other equipment in close proximity, such as forging hammers, presses, moving vehicles. The machine shall also be free of excessive vibrations during an impact test.

NOTE Such vibrations can be detected by placing a small container of water at any convenient location on the machine framework; the absence of ripples on the water surface during an impact test indicates that this requirement has been met. Excessive vibration in a machine firmly fastened to the floor indicates the need for a separate foundation and/or the use of vibration isolators.

**5.1.6 Energy-indicating mechanism**

The resolution shall be at least 1/400 of the nominal energy.

**5.2 Verification of reference testing machine**

Direct verification shall be carried out in accordance with ISO 148-2 and with the additional requirements of 5.1.

Indirect verification shall be carried out using certified reference test piece. The repeatability and the bias shall be as specified in Table 3.

All equipment used for inspection and verification of a reference testing machine shall be calibrated and shall have a certified traceability to the SI System (the international system of units). The body performing the verification shall maintain calibration records for all inspection, measurement and test equipment.

The dates and details of all inspections and repairs shall be documented and maintained for each reference machine by the owner of the machine.

**Table 3 — Repeatability and bias of reference pendulum impact testing machines**

| Absorbed energy<br>$K$ | Repeatability       | Allowed bias       |
|------------------------|---------------------|--------------------|
| <40 J                  | $\leq 2$ J          | $\pm 2$ J          |
| $\geq 40$ J            | $\leq 5$ % of $K_R$ | $\pm 5$ % of $K_R$ |

Repeatability is the standard deviation of the  $K_R$  values measured on a least 10 reference test pieces.

Bias is given by  $\bar{K} - K_R$   
 where  

$$\bar{K} = \frac{K_1 + K_2 + K_3 + \dots + K_n}{n}$$
 where  $n \geq 10$ .

**6 Reference test pieces**

**6.1 General**

Guidelines for the preparation, certification, and use of (certified) reference materials have been drawn up by ISO REMCO, the ISO Committee on reference materials (see References [3] to [7]). The procedures described below provide more details, specific to the case of Charpy reference test pieces.

**6.2 Material**

All the test pieces from a batch shall come from a single ingot or melt.

All test pieces shall be made of steel. The composition of the test pieces is not specified. Batches with different energy levels may have different compositions.

All test pieces from a batch shall receive the same heat treatment.

For each batch, the level for the reference absorbed energy is characterized by using one of the following ranges:

- Low: <30 J
- Medium: ≥30 J to 110 J
- High: ≥110 J to 200 J
- Ultra-high: ≥200 J

### 6.3 Dimensions

The reference test pieces shall meet the dimensional requirements given in [Table 2](#).

NOTE These dimensions are identical with those in ISO 148-1, except that some of the tolerances are tighter.

The radius at the base of the notch shall be tangential to the notch angle.

The surface finish,  $R_a$ , shall not exceed 1,6  $\mu\text{m}$  on the notched surface and 3,2  $\mu\text{m}$  on the other surfaces.

### 6.4 Marking

All test pieces shall be permanently marked so that each test piece can be distinguished from all the others.

The test piece may be marked on any face not in contact with supports, anvils or striker and at a position such that plastic deformation and surface discontinuities caused by marking do not affect the absorbed energy measured in the test.

### 6.5 Qualification of a batch of reference test pieces

**6.5.1** Any group of test pieces meeting the requirements of [6.2](#), [6.3](#) and [6.4](#) may be used as the batch from which reference test pieces are randomly selected.

**6.5.2** To determine the reference energy of a batch, draw one or more sets of at least 25 test pieces at random from the batch and test them on one or more reference machine(s). Take the reference absorbed energy of the batch as the grand average of the values obtained for the 25 or more test pieces, or as the average of the mean values obtained on the different reference machines.

NOTE The certified values can be determined using other methods, providing the method used conforms to ISO Guide 34 and ISO Guide 35[Z].

**6.5.3** Also calculate the standard deviation. The standard deviation shall be as specified in [Table 4](#).

**Table 4 — Maximum allowable standard deviations permitted for Charpy reference test pieces**

| Energy<br>$K_R$ | Standard deviation |
|-----------------|--------------------|
| <40 J           | ≤2,0 J             |
| ≥40 J           | ≤5 % of $K_R$      |

**6.5.4** The report on the impact tests of the reference test pieces shall include the following information:

- a) striker geometry;
- b) temperature at which the tests were performed;
- c) all details necessary for the identification of each test piece;
- d) energy value,  $K$ , of each test piece, corrected for air resistance and friction, with the striker radius and specimen type indicated ( $KV_2$ ,  $KV_8$ ,  $KU_2$ , or  $KU_8$ );
- e) value of the reference absorbed energy and the associated standard deviation;
- f) uncertainty associated with the reference absorbed energy value measured for the set.

NOTE Information on calculating uncertainty is given in [Annex A](#).

## **6.6 Reference test piece sets**

After the characterization set(s) to be tested by the reference machine(s) have been drawn from the batch, draw the remaining test pieces in sets of five. These are the test piece sets. Each test piece set shall remain intact with no substitution permitted.

## **7 Certificates for reference test pieces**

Each set of reference test pieces shall be accompanied by a certificate which provides the following information:

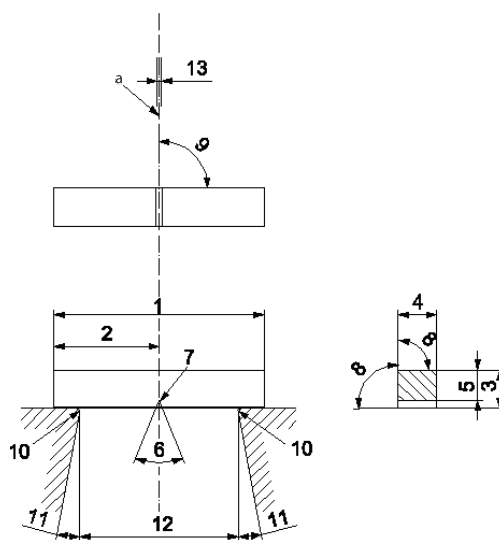
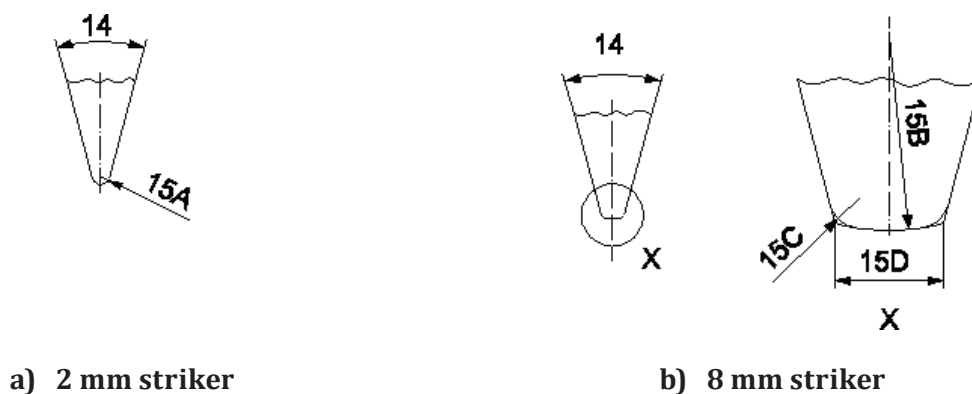
- a) reference to this part of ISO 148, i.e. ISO 148-3;
- b) name, trademark or reference number of the producer;
- c) reference absorbed energy value of the set and its uncertainty at the stated level of confidence;
- d) striker geometry;
- e) temperature at which the reference specimen should be tested;
- f) necessary information for appropriate use of the reference test pieces;
- g) name and general description of the material;
- h) producer's code for the batch;
- i) intended use (making reference to ISO 148-2);
- j) description of the (metrologically valid) procedure used to determine the certified value;
- k) statement on the metrological traceability of the certified value;
- l) storage conditions and shelf-life (period of validity).

## **8 Notes for using sets of reference test pieces**

**8.1** Indirect verification of an industrial machine shall be performed in accordance with ISO 148-2 using the reference test pieces, the striker and the temperature specified by the producer of the test pieces.

**8.2** All the reference test pieces in each set shall be used for a single, indirect verification of the pendulum impact testing machine, testing the test pieces in random order and including all the results in the average. Substitution or replacement of individual test pieces by test pieces from another reference set is not permitted.

Dimensions in millimetres

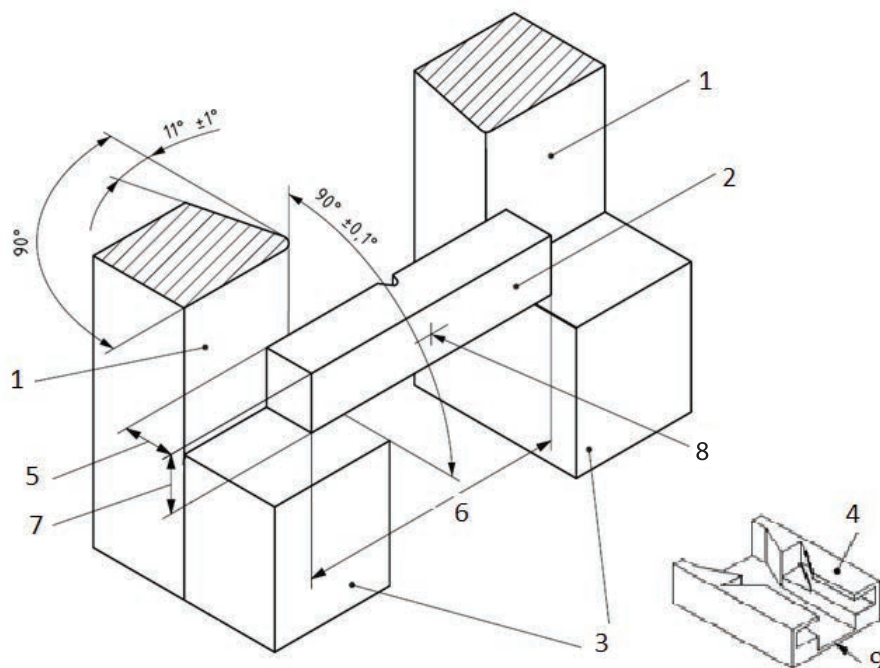


c) Overview

NOTE See [Table 2](#) for geometrical characteristics.

a Line of strike.

Figure 1 — Dimensions of test pieces, anvils and strikers



**Key**

- 1 anvil
- 2 standardized test piece
- 3 test piece supports
- 4 shroud
- 5 width of test piece,  $W$
- 6 length of test piece,  $L$
- 7 thickness of test piece,  $B$
- 8 centre of strike
- 9 direction of pendulum swing

**Figure 2 — Configuration of test-piece supports and anvils in a reference pendulum impact testing machine**



## Annex A (informative)

### Uncertainty of the certified *KV* value of Charpy reference materials

#### A.1 Background

When performing an indirect verification of a pendulum impact testing machine, one compares the reference *KV* value of the reference test pieces with values measured on the pendulum impact testing machine under verification. To determine the measurement uncertainty of this indirect verification exercise and, later, the measurement uncertainty of Charpy measurements on the verified pendulum impact testing machine, one needs the uncertainty of the reference value. Therefore, this uncertainty should be assessed and provided by the reference material (RM) producer.

The ISO Committee on reference materials (REMCO) has drawn up a series of documents on reference materials production and use, which are released as ISO Guides (see References [3] to [7]). Approaches to tackling the uncertainty aspect of RM production are described in generic terms in ISO Guide 34 and in more technical-statistical detail in ISO Guide 35. This Annex provides an ISO-Guides-compliant practical framework for the calculation of the uncertainty of the certified absorbed energy value of a Charpy RM. The text is based on current approaches followed by national metrology institutes (NMIs) active in the Charpy field. The approaches presented here can be used as a guideline by potential new Charpy RM producers, as well as by the users of Charpy RMs who require more insight into the uncertainty stated by the RM producer on the RM certificate.

#### A.2 The GUM-compliant uncertainty budget

ISO Guide 35 provides a basic, GUM[8]-compliant, model for the certification of batches of certified reference materials (CRMs). In Charpy terms, the model can be expressed as follows:

$$KV_R = KV_{\text{char}} + \delta KV_{\text{hom}} + \delta KV_{\text{its}} + \delta KV_{\text{sts}} \quad (\text{A.1})$$

where

- $KV_{\text{char}}$  is the *KV* value obtained from the characterization of the batch (comparing results from different machines);
- $KV_{\text{hom}}$  is an error term due to variation between samples (comparing results in repeatability conditions on a single pendulum);
- $KV_{\text{its}}, KV_{\text{sts}}$  are error terms due to the long-term and short-term instability of the RM (comparing results of samples exposed to different ageing periods).

Homogeneity and stability studies are most often designed in such a way that the values of the corresponding error terms are zero. However, the uncertainties of the error terms are not (always) zero. Assuming independence of the variables, the uncertainty of the certified value of the Charpy RM, therefore, can be expressed as:

$$u_{\text{RM}} = \sqrt{u_{\text{char}}^2 + u_{\text{hom}}^2 + u_{\text{its}}^2 + u_{\text{sts}}^2} \quad (\text{A.2})$$

The better the within-instrument repeatability and the between-instrument reproducibility, the smaller  $u_{\text{char}}$  will be. The better the between-sample homogeneity, the smaller  $u_{\text{hom}}$  will be. Sometimes, the material homogeneity is very good, and  $u_{\text{hom}}$  is dominated by within-instrument repeatability. This

is not the case for typical Charpy RMs. The better the stability of the RM microstructure, under the appropriate transport and storage conditions, the smaller  $u_{sts}$  and  $u_{lts}$  will be.

### A.3 $KV_R$ , the certified $KV$ of a batch of Charpy RMs

Charpy RMs are produced batch-wise. The  $KV$  values of samples from a single batch vary from sample to sample. Yet, the whole batch will be assigned a single certified  $KV$  value. Obviously, this could be best estimated by testing all samples. However, since the impact test is destructive, there would be no samples left for distribution as reference materials. Instead, a representative selection of samples is taken from the batch and tested. An average value will become the certified value,  $KV_R$ . This can be the average of all samples tested, or the average of the mean values of a number of subgroups of the samples tested.

### A.4 $u_{char}$ , the uncertainty of the average $KV$ of a batch of Charpy RMs

#### A.4.1 Differences between pendulum impact testing machines

Even if one would break all samples of a batch to determine the average  $KV$  of the batch, still the question remains whether the average value obtained under the particular test conditions is affected by inaccuracies in the tests performed. To reduce this uncertainty, RM producers generally try to measure the property to be certified in different independent ways. For properties such as the chemical composition of an RM, one can often use different methods. However, in the case of pendulum impact tests, the only way to measure the “method-defined”  $KV$  value is to do Charpy pendulum impact tests in accordance with the applicable standard procedure (ISO 148-1), to which the certified values will be metrologically traceable.

To reduce the effect of machine-specific bias from the standard procedure on the certified reference values, one often performs pendulum impact tests on several pendulum impact testing machines. The larger the number of pendulum impact testing machines used to assess the average of a single batch of samples, the more likely it is that the average of the values obtained is true and unbiased. Of course, this is only true at the condition that individual participating pendulums are good quality instruments. This is the approach of both inter- and intralaboratory comparisons, currently followed in Charpy reference material certification, and recommended in ISO Guide 35.<sup>[Z]</sup>

#### A.4.2 Intercomparison among $p$ pendulum impact testing machines ( $p \geq 6$ )

When a sufficient number of machines participate in a comparison, the standard uncertainty of the average value is calculated as:

$$u_{char} = \frac{s_p}{\sqrt{p}} \tag{A.3}$$

where

- $u_{char}$  is the uncertainty from the characterization of the batch;
- $p$  is the number of laboratories or instruments participating in the intercomparison;
- $s_p$  is the standard deviation of the laboratory mean values.

This approach assumes that the individual laboratory mean values are normally distributed, and that the instruments or laboratories participating are a representative sample from the population of Charpy pendulum impact testing machines that meet the dimensions and performance criteria specified in ISO 148-2. The number of degrees of freedom,  $\nu_{char}$ , associated with this way of calculating  $u_{char}$  is  $p-1$ . ISO Guide 35 recommends a minimum number of six laboratories or instruments for this approach (ISO Guide 35:2006, 9.4.2.3.1).

### A.4.3 Intercomparison among $p$ pendulum impact testing machines ( $p < 6$ )

When the number of instruments participating to the comparison is limited, the value  $s_p$  is not a reliable estimate of the standard deviation of the mean values between instruments. To assess  $u_{\text{char}}$ , other methods should be used. These methods combine the systematic differences observed between the instruments participating in the intercomparison (between-instrument uncertainty) and the measurement uncertainty assessed for the individual instruments (within-instrument uncertainty). An example is the so-called “BOB” or Type-B-on-bias-approach (see Reference [9]).

To have better control over the quality of the impact pendulums participating in the certification exercise, some CRM producers prefer to limit the number of impact pendulums to those in their own laboratory (intralaboratory). This approach offers the benefit of better defining the range for acceptable machine performance. However, it can be argued that it affects the independence of the averaged values. This is why the interlaboratory comparison is generally preferred in ISO Guide 35.

### A.5 Uncertainty due to material instability

The stability of the certified value of a CRM is typically threatened by two possible effects: degradation of the material during transport from producer to user (short-term stability), and degradation of the material during storage between the moment of production and the distribution to the CRM user (long-term stability). In the case of the steels currently used for the production of Charpy CRMs, neither short- nor long-term stability has presented problems (see References [10], [11] and [12]). However, this should remain a subject of investigation, especially when selecting new types of steel for the production of Charpy CRMs. Until then, the values for  $u_{\text{ITS}}$  and  $u_{\text{STS}}$  are considered negligibly small.

### A.6 Uncertainty due to sample-to-sample variation — Homogeneity of the batch

Due to the heterogeneity of the steel microstructure, and the nature of the impact fracture process, samples from the same batch often have measurably different  $KV$  values. This implies that the average value of the set of verification test pieces tested by the CRM user is not exactly the same as the average of the RM batch from which the set was drawn.

For a single sample, the standard uncertainty,  $u_{\text{hom}}$ , associated with this homogeneity issue equals the standard deviation of the batch,  $s_{\text{RM}}$ . To assess this standard deviation, tests are performed on a representative number of samples  $n_{\text{hom}}$ , selected from the batch. The tests are performed in repeatability conditions, excluding or at least minimizing the contributions to the standard deviation coming from machine, operator or other factors.

NOTE The value of  $s_{\text{RM}}$  can also be deduced from the results of the interlaboratory comparison (see A.4). In this case, the within-laboratory and the between-laboratory variance of the results are separated using ANOVA (analysis of variance). The within-laboratory variance is related to  $s_{\text{RM}}$ .

Experience has shown that it is difficult to obtain large batches of Charpy reference test pieces with a standard deviation smaller than 3 %. At least, this is the case for the hardenable kind of steels needed for the production of samples at different energy levels with a minimum hardness, so as to truly put the pendulum to a test during verification. To reduce this relatively large contribution to the overall uncertainty of the certified value, it is common practice that the CRM user tests a set of samples of the batch, rather than a single sample, to verify a pendulum. (Actually, ISO 148-2 prescribes the use of at least five test pieces.) The chances that the average of a set of test pieces equals the average of the whole batch increases with  $n_V$ , the number of test pieces used in the indirect verification, reducing the corresponding uncertainty contribution according to [Formula \(A.4\)](#):

$$u_{\text{hom}} = \frac{s_{\text{RM}}}{\sqrt{n_V}} \quad (\text{A.4})$$

The number of degrees of freedom of this uncertainty contribution,  $\nu_{\text{hom}}$ , equals  $(n_{\text{hom}} - 1)$ .

## A.7 Combined and expanded uncertainty of the certified value, and how to report them

If the uncertainty contributions from material instability can be neglected, the combined standard uncertainty,  $u_{RM}$ , is calculated from the remaining standard uncertainty contributions,  $u_{char}$  and  $u_{hom}$ , as follows:

$$u_{RM} = \sqrt{u_{char}^2 + u_{hom}^2} \quad (A.5)$$

Usually, the uncertainty of a certified value on the certificate is specified for a confidence level of about 95 %. Therefore, the standard combined uncertainty,  $u_{RM}$ , has to be expanded using an appropriate coverage factor,  $k$ . The coverage factor to be used depends on the number of degrees of freedom associated with the combined uncertainty, which can be computed using the Welch-Satterthwaite approximation (see Reference [8]). For a typical case (see example in A.8), the number of effective degrees of freedom is larger than 20 and a coverage factor of  $k = 2$  can be used. If the number of degrees of freedom,  $\nu_{RM}$ , is smaller, the coverage factor can be calculated as:

$$k = t_{95}(\nu_{RM}) \quad (A.6)$$

with  $t$  values taken from the non-standard GUM table (see Reference [8]). The certified value,  $KV_{RM}$ , of reference test pieces always has to be reported together with the corresponding expanded uncertainty,  $U_{RM}$ , and the coverage factor and/or confidence level (see Reference [5]). For the case of Charpy reference test pieces, the user will benefit from the following additional information (see ISO 148-2, Annex A):

- 1)  $\nu_{char}$ , the number of degrees of freedom of  $u_{char}$ , or the number  $p$  of laboratories/instruments participating in the laboratory comparison;
- 2) standard deviation,  $s_{RM}$ , of the homogeneity test results, as a measure for the reference material inhomogeneity, as well as  $n_{hom}$ , the number of samples used to determine the homogeneity;
- 3) value of  $u_{char}$ , which is required for transferring the certified value from one batch of Charpy RMs to another batch (see A.9).

## A.8 Example

An RM producer has processed a batch of Charpy test pieces. To assess the homogeneity of the batch, one laboratory is chosen to test 25 test pieces in repeatability conditions. Table A.1 shows the results.

First, the data are screened for statistical outliers (as described, for example, in ISO 5725-2[13]). Grubbs' test reveals that the result of sample 22 is a statistical outlier at the 95 % confidence level. An inspection of the sample reveals no abnormal anvil or striker traces, indicating that the sample was correctly positioned during the test. Also, no trivial error was detected when inspecting the test report. Since there is no technical explanation indicating that the result is an outlier due to reasons external to the sample, the result cannot be excluded from the homogeneity analysis. If one had detected a technical explanation in the sample itself (such as a significant microstructural flaw on the fracture surface), the result could not have been eliminated either, since this flaw is related to the material homogeneity, which is the object of the homogeneity assessment.

When comparing the obtained value,  $s_{RM}$ , (3,57 J) with the average value of  $KV$  (124,74 J), it is confirmed that the batch meets the criterion imposed in Table 4 on batches of Charpy reference materials ( $s_{RM} \leq 5$  % of the average value). Based on the intended use of the samples, the CRM producer chooses

to distribute the samples in sets of 5, and calculates the corresponding value for the uncertainty contribution using [Formula \(A.4\)](#):

$$u_{\text{hom}} = \frac{s_{\text{RM}}}{\sqrt{n_V}} = \frac{3,57}{\sqrt{5}} = 1,60 \quad (\text{A.7})$$

Then, 11 accredited test laboratories receive 10 test pieces each, randomly selected from the full batch.

NOTE The RM producer is free to choose the number of test pieces per test laboratory. Larger numbers will reduce the uncertainty of the certified value.

The results of the interlaboratory comparison are examined by the RM producer. Grubbs' test does not reveal any statistical outlier among the laboratory mean values. [Table A.2](#) summarizes the results and the RM relevant parameters.

**Table A.1 — Results of homogeneity tests**

| Sample                | KV<br>J | Sample                   | KV<br>J | Sample                | KV<br>J |
|-----------------------|---------|--------------------------|---------|-----------------------|---------|
| 1                     | 127,7   | 10                       | 127,9   | 18                    | 127,3   |
| 2                     | 122,2   | 11                       | 120,7   | 19                    | 123,8   |
| 3                     | 123,5   | 12                       | 127,5   | 20                    | 126     |
| 4                     | 125,6   | 13                       | 122,1   | 21                    | 128,7   |
| 5                     | 122,5   | 14                       | 126,3   | 22                    | 114,5   |
| 6                     | 122,9   | 15                       | 128,9   | 23                    | 121,3   |
| 7                     | 126,7   | 16                       | 125,4   | 24                    | 128,1   |
| 8                     | 123,2   | 17                       | 119,1   | 25                    | 127     |
| 9                     | 129,5   | —                        | —       | —                     | —       |
| Average KV = 124,74 J |         | s <sub>RM</sub> = 3,57 J |         | n <sub>hom</sub> = 25 |         |

**Table A.2 — Results of interlaboratory comparison**

| Laboratory                | KV<br>J                 | Laboratory | KV<br>J                    |                        |
|---------------------------|-------------------------|------------|----------------------------|------------------------|
| 1                         | 122,2                   | 7          | 126,8                      |                        |
| 2                         | 120,9                   | 8          | 125,1                      |                        |
| 3                         | 125,5                   | 9          | 123,7                      |                        |
| 4                         | 122,0                   | 10         | 124,0                      |                        |
| 5                         | 123,8                   | 11         | 124,9                      |                        |
| 6                         | 122,8                   | —          | —                          |                        |
| KV <sub>R</sub> = 123,8 J | s <sub>p</sub> = 1,73 J | P = 11     | u <sub>char</sub> = 0,52 J | v <sub>char</sub> = 10 |

The certified value (123,8 J) is obtained as the rounded average of the laboratory mean values. A standard uncertainty, u<sub>char</sub>, of 0,52 J is calculated from the standard deviation of the laboratory mean values, using [Formula \(A.3\)](#).

[Table A.3](#) shows the standard uncertainty contributions, u<sub>char</sub> and u<sub>hom</sub>, the combined standard uncertainty, u<sub>RM</sub> and v<sub>RM</sub>, and the number of degrees of freedom calculated from the Welch-Satterthwaite equation. Since v<sub>RM</sub> is larger than 20, a coverage factor of k = 2 can be used to calculate the expanded uncertainty, U<sub>RM</sub>, for a confidence level of about 95 %. Its value is rounded up to one decimal, in agreement with the precision of the certified value.

**Table A.3 — Certification-related uncertainty contributions and associated degrees of freedom**

|  |                 |                            |                           |
|--|-----------------|----------------------------|---------------------------|
| $u_{char} = 0,52 \text{ J}$  | $v_{char} = 10$ | $u_{hom} = 1,60 \text{ J}$ | $v_{hom} = 24$            |
| Standard uncertainty of the certified value calculated from $u_{char}$ and $u_{hom}$ using <a href="#">Formula (A.5)</a> |                 |                            | $u_{RM} = 1,68 \text{ J}$ |
| Degrees of freedom calculated using the Welch-Satterthwaite approximation  |                 |                            | $v_{RM} = 28$             |
| Expanded uncertainty of the certified value  |                 |                            | $U_{RM} = 3,4 \text{ J}$  |

To report the certification results, the certified value and its uncertainty, [Table A.4](#) can be used.

**Table A.4 — Summary table of the certified  $KV_R$  with expanded uncertainty,  $U_{RM}$**

| $p$ | $s_{RM}^a$<br>J | $n_{hom}$ | $KV_R$<br>J | $u_{char}$<br>J | $U_{RM}^b$<br>J |
|-----|-----------------|-----------|-------------|-----------------|-----------------|
| 11  | 3,57            | 25        | 123,8       | 0,52            | 3,4             |

<sup>a</sup> This standard deviation is a conservative estimate of the test material heterogeneity. (Its value also contains a contribution from the instrument repeatability, which cannot be assessed separately.)

<sup>b</sup> The expanded uncertainty, calculated according to the procedure described in this annex, corresponds to a confidence level of about 95 %.

## A.9 Additional uncertainty contributions when transferring the certified value from a primary batch to a secondary batch

### A.9.1 Aim

Charpy RMs of a particular batch can be used to determine the certified value of another batch of Charpy test pieces. The assignment of the certified value of the new batch to be certified is based on a comparison of  $KV$  values of previously certified samples and new samples to be certified. In the following, the originally certified batch is called the “Primary Batch (PB)”, which has a certified value  $KV_{PB}$ . The new batch to be certified is called the “Secondary Batch (SB)”. The comparison necessarily results in an additional uncertainty contribution in comparison with the uncertainty of the PB.

### A.9.2 Practice — Repeatability, bias and commutability requirements

The additional uncertainty contribution from the transfer of the certified value from PB to SB can be kept reasonably small if the comparison occurs under strict repeatability conditions. In the case of Charpy pendulum impact tests, this means ideally that SB and PB samples are tested in one single series of tests on one single pendulum, with a mixed order for testing SB and PB samples. The following values are the results of these tests:

$\bar{X}_{PB}$ , mean of  $n_{PB}$  specimens used to compare SB with PB, standard deviation =  $s_{PB}$

$\bar{X}_{SB}$ , mean of  $n_{SB}$  specimens used to compare SB with PB, standard deviation =  $s_{SB}$

This approach is reliable only if the pendulum used to compare PB and SB performs well. In other words, the ratio  $KV_{PB} / \bar{X}_{PB}$  should be close to 1; the allowed difference is 20 % ( $KV_{PB} \geq 40 \text{ J}$ ) or 2 J ( $KV_{PB} < 40 \text{ J}$ ), corresponding to the level of bias allowed for reference pendulums specified in [Table 3](#).

In addition, the batches SB and PB should be comparable to ensure that the pendulum responds in a similar manner to samples from both batches. In practice, this means that the same base material should be used, and the values of  $\bar{X}_{PB}$  and  $\bar{X}_{SB}$  should be close (within  $\pm 20 \%$ ). In terms of CRM production, this is a commutability requirement, the basis for which is discussed in ISO Guide 33.



The certified value of the secondary batch,  $KV_{SB}$ , is obtained by correcting  $\bar{X}_{SB}$  by a kind of punctual calibration of the pendulum on which the SB and PB were compared, using the ratio  $KV_{PB} / \bar{X}_{PB}$ :

$$KV_{SB} = \frac{KV_{PB}}{\bar{X}_{PB}} \cdot \bar{X}_{SB} \quad (\text{A.8})$$

where  $KV_{PB}$  is the certified  $KV$  value of the primary batch.

### A.9.3 The resulting uncertainty

To calculate the uncertainty of  $KV_{SB}$ , one needs to combine the uncertainties of the factors  $KV_{PB}$ ,  $\bar{X}_{SB}$  and  $\bar{X}_{PB}$  from [Formula \(A.8\)](#).

- a) The value of  $KV_{PB}$  links the SB with the results of the PB. In terms of uncertainty, only  $u_{\text{char,PB}}$  is relevant, and not  $u_{\text{hom}}$ , because [Formula \(A.7\)](#) makes the link to the average  $KV$  value of the primary batch, not to a set of five samples from the PB.
- b) The values  $\bar{X}_{SB}$  and  $\bar{X}_{PB}$  are estimates of the average of SB and PB on the particular pendulum in the particular repeatability conditions, used for the comparison. The standard uncertainty contributions from both factors are

$$u_{\bar{X}_{SB}} = \frac{s_{SB}}{\sqrt{n_{SB}}} \quad (\text{A.9})$$

$$u_{\bar{X}_{PB}} = \frac{s_{PB}}{\sqrt{n_{PB}}} \quad (\text{A.10})$$

NOTE The resolution of the pendulum used contributes to the uncertainties of  $\bar{X}_{SB}$  and  $\bar{X}_{PB}$  as well. However, the pendulums used for this type of comparison are chosen to have a good resolution (0,1 J or better), in which case the resolution does not significantly contribute to  $u_{\bar{X}_{SB}}$  and  $u_{\bar{X}_{PB}}$ .

Since the partial derivatives of [Formula \(A.8\)](#) are all equal to 1 or -1, the uncertainty of the characterization of the SB is obtained by combining the contributions  $u_{\text{char,PB}}$ ,  $u_{\bar{X}_{SB}}$  and  $u_{\bar{X}_{PB}}$ . Determine  $u_{\text{char,SB}}$  as follows (see H.6.2 of Reference [8]):

$$u_{\text{char,SB}} = \sqrt{u_{\text{char,PB}}^2 + u_{\bar{X}_{SB}}^2 + u_{\bar{X}_{PB}}^2} \quad (\text{A.11})$$

This uncertainty contribution can be combined with the uncertainty contributions of homogeneity and/or stability, as described in [A.8](#).

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