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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Ferroalloys — Experimental methods for checking the precision of sample division

Ferro-alliages — Méthodes expérimentales de contrôle de la fidélité de la division des échantillons

Reference number
ISO 7373 : 1987 (E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 7373 was prepared by Technical Committee ISO/TC 132, *Ferroalloys.*

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Ferroalloys — Experimental methods for checking the precision of sample division

1 Scope and field of application

This International Standard specifies the experimental methods for checking the precision of sample division of ferroalloys carried out on the gross sample or sub-sample obtained from a ferroalloy consignment according to the methods prescribed in the relevant International Standard.

The prescribed methods are applicable to the taking of increments of crushable ferroalloys but are not applicable to uncrushable ferroalloys of which increments are taken by boring.

2 References

ISO 4552, *Ferroalloys — Sampling and sample preparation for chemical analysis —*

Part 1: Ferrochromium, ferrosilicochromium, ferrosilicon, ferrosilicomanganese, ferromanganese.

Part 2: Ferrotitanium, ferromolybdenum, ferrotungsten, ferroniobium, ferrovanadium.

ISO 7087, *Ferroalloys — Experimental methods for the evaluation of the quality variation and methods for checking the precision of sampling.*

3 General requirements

3.1 Mass of a gross sample

For the purposes of the experiment, the mass of a consignment of ferroalloys covered by ISO 4552-1 shall be not less than 100 t and that of a consignment of ferroalloys covered by ISO 4552-2 shall be not less than 5 t so that the mass of the gross sample is sufficient to obtain a test sample of the required mass.

3.2 Methods of analysis

The analysis of experimental samples shall be carried out in accordance with the methods prescribed in the relevant International Standards.

3.3 Quality characteristic

The quality characteristic on which checking of the precision of sample division is carried out shall be as given in the relevant International Standards.

Any other element may be selected as a quality characteristic by mutual agreement between the interested parties.

3.4 Number of experiments

The experiment shall be repeated at least 10 times for each type of ferroalloy, either on gross samples or on sub-samples.

NOTE — Experimental samples may be obtained by splitting the gross sample taken for the determination of the consignment quality.

4 Experimental methods

4.1 Selection of experimental methods

4.1.1 For ferroalloys the physical properties of which prohibit the preparation of samples by one or two stages of division and when the proportions of sample to be discarded during sequental preparation stages have to be reclaimed as commodities, for example ferroalloys covered by ISO 4552-2, it is recommended that the experiment be conducted according to the method of division in three or more stages given in 4.2.

4.1.2 When the existing apparatus for crushing is capable of preparing the sample in one or two stages of division and a better estimation of the precision of division is required, it is recommended that the experiment be conducted according to the method of division in one or two stages given in 4.3.

4.2 Method of division in three or more stages

4.2.1 This method shall be applied to ferroalloys covered by ISO 4552-2.

4.2.2 Examples of the whole-through sieve size of the sample to be divided at each of the stages are given in table 1.

Table 1 — Examples of particle size of sample by division stage

Division stage	Whole-through sieve size of sample
Primary	−10 mm or −7,10 mm
Secondary	−5 mm or −2,80 mm
Tertiary	−1,0 mm or −250 µm

4.2.3 An example of a flow diagram for preparation of experimental samples is given in figure 1.

One test sample shall be prepared from each divided sample.

The number of stages of crushing and division shall be the same when preparing each of the binary test samples.

A single chemical determination shall be made on one of the binary test samples and duplicate determinations on the other.

NOTE — The duplicate determinations shall be made on two test portions taken from a test sample at a chemical laboratory.

4.2.4 The sequence of chemical determinations of the experimental test samples shall be random, or else both experimental and usual test samples shall be analysed at the same time in a random order.

4.2.5 The experimental data shall be recorded in a data log such as that given in table 2.

Table 2 — Example of data sheet for sample division experiment

Designation of experiment: .....

Type and grade of ferroalloy (for example, ferromanganese): .....

Method of division (for example, method by 4.2): .....

Date of experiment: .....

Gross sample	Quality characteristic (for example, % Mn)				
	x_{i1}	x_{i21}	x_{i22}	$\lvert x_{i21} - x_{i22} \rvert$	$\lvert x_{i1} - x_{i21} \rvert$ or $\lvert x_{i1} - x_{i22} \rvert$
1					
2					
.					
.					
.					
k					
				$\bar{R}_1$	$\bar{R}_2$

$$\hat{\sigma}_M^2 = \left(\frac{\bar{R}_1}{1{,}128}\right)^2$$

$$\hat{\sigma}_D = \sqrt{\left(\frac{\bar{R}_2}{1{,}128}\right)^2 - \hat{\sigma}_M^2}$$

4.3 Method of division in one or two stages

4.3.1 This method shall be applied to ferroalloys covered by ISO 4552-1.

4.3.2 The recommended whole-through sieve size of the sample to be divided is −2,8 mm or −1,0 mm.

4.3.3 An example of a flow diagram for preparation of experimental samples is given in figure 2.

One test sample shall be prepared from each divided sample.

The number of stages of crushing and division shall be the same when preparing each of the binary test samples.

A single chemical determination shall be made on one of the binary test samples and duplicate determinations on the other.

NOTE — The duplicate determinations shall be made on two test portions taken from a test sample at a chemical laboratory.

4.3.4 The sequence of chemical determinations of the experimental test samples shall be random, or else both experimental and usual test samples shall be analysed at the same time in random order.

4.3.5 The experimental data shall be recorded in a data log such as that given in table 2 as an example.

5 Data analysis

The method of data analysis for the estimation of precision of division is the same for the experiments conducted by both 4.2 and 4.3.

NOTE — During the process of data analysis, if the calculated value within the square root sign turns out to be negative, the standard deviation should be regarded as being zero ($\sigma = 0$), provided that no attributable defects in the experimental operations are observed.

5.1 Precision of method of chemical analysis

Calculate the estimated value of precision of the method of chemical analysis from the following equations:

$$\bar{R}_1 = \frac{1}{k}\sum_{i=1}^{k} \lvert x_{i21} - x_{i22} \rvert \qquad \ldots (1)$$

$$\hat{\sigma}_M = \frac{\bar{R}_1}{d_2} \qquad \ldots (2)$$

where

x_{i21}, x_{i22} are the respective first and second measurements of the ith test sample x_{i2};

k is the number of experiments;

$\bar{R}_1$ is the mean range of duplicate measurements;

d_2 is the factor to obtain the standard deviation from range, for duplicate measurements; d_2 = 1,128;

$\hat{\sigma}_M$ is the estimated value of precision of the method of chemical analysis in terms of the standard deviation.

5.2 Precision of sample division

Calculate the estimated value of the precision of sample division from the following equations:

$$\bar{R}_2 = \frac{1}{k} \sum_{i=1}^{k} |x_{i1} - x_{i21}| \qquad \ldots (3)$$

or

$$\bar{R}_2 = \frac{1}{k} \sum_{i=1}^{k} |x_{i1} - x_{i22}|$$

$$\hat{\sigma}_D = \sqrt{\left(\frac{\bar{R}_2}{d_2}\right)^2 - \hat{\sigma}_M^2} \qquad \ldots (4)^*$$

where

$\bar{R}_2$ is the mean value of the ranges of the single determination and of one of the duplicate determinations of the binary samples;

$\hat{\sigma}_D$ is the estimated value of precision of division in terms of the standard deviation.

6 Review of experimental results

The values of the precision of sample division and/or the precision of the method of chemical analysis obtained shall be compared with the required values or those given in the relevant International Standards.

In the event that the precision of sample division and/or the precision of the method of chemical analysis do not attain the specified value or values given in the relevant International Standards, appropriate action concerning the procedure of sample preparation and/or chemical analysis shall be taken by the respective organizations.

To prevent an uncontrollable situation from arising, it should be recognized that the precision of division is liable to worsen in the following circumstances:

a) when a sample having a large particle size is divided excessively at one time into a divided sample of small mass;

b) when the division is carried out in a large number of stages;

c) when apparatus of inadequate precision is used for sample division;

d) when the established operational instructions for sample preparation are not implemented adequately.

* Other methods of calculation are given in ISO 7087.

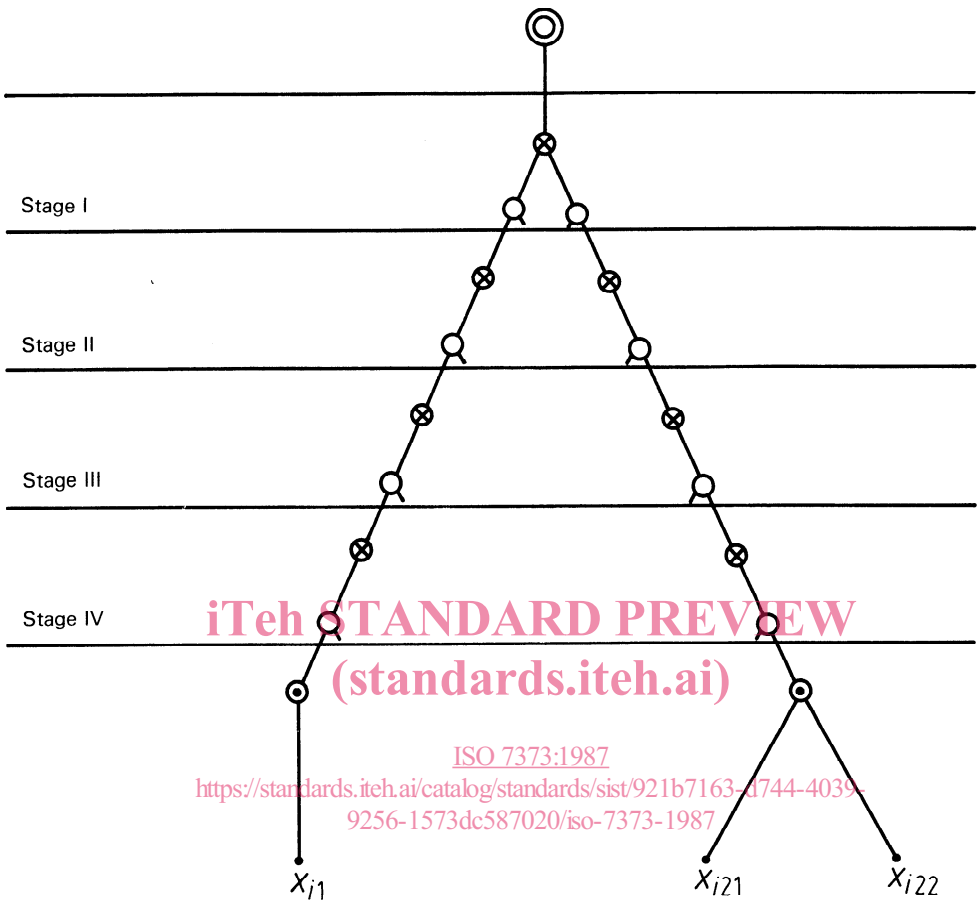
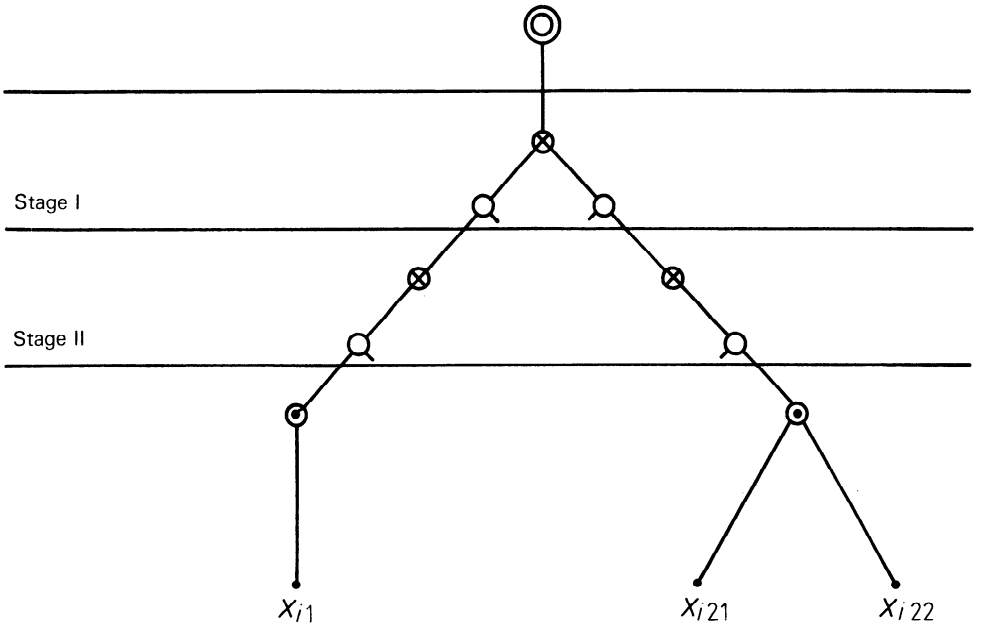


Figure 1 — Example of diagram for sample division in four stages



Legend:

Gross sample or sub-sample

Crushing

Division

Final sample for chemical analysis

Measurement of test portion

Figure 2 — Example of diagram for sample division in two stages

7 Bibliography

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ISO 4159, *Ferromanganese and ferrosilicomanganese — Determination of manganese content — Potentiometric method.*

ISO 4173, *Ferromolybdenum — Determination of molybdenum content — Gravimetric method.*

ISO 5445, *Ferrosilicon — Specifications and conditions of delivery.*

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ISO 5449, *Ferrosilicochromium — Specifications and conditions of delivery.*

ISO 5450, *Ferrotungsten — Specifications and conditions of delivery.*

ISO 5451, *Ferrovanadium — Specifications and conditions of delivery.*

ISO 5452, *Ferromolybdenum — Specifications and conditions of delivery.*

ISO 5453, *Ferroniobium — Specifications and conditions of delivery.*

ISO 5454, *Ferrotitanium — Specifications and conditions of delivery.*