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Ferroalloys -- Experimental methods for checking the bias of sampling and sample preparation

Ferro-alliages -- Méthodes expérimentales de contrôle de l'erreur systématique de l'échantillonnage et de la préparation des échantillons

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Ferroalloys — Experimental methods for checking the bias of sampling and sample preparation

Ferro-alliages — Méthodes expérimentales de contrôle de l'erreur systématique de l'échantillonnage et de la préparation des échantillons

Reference number
ISO 7347 : 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 7347 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 bias of sampling and sample preparation

1 Scope and field of application

This International Standard specifies the experimental methods to be applied for checking the bias of sampling and sample preparation of ferroalloys given in the relevant International Standards.

It should be read in conjunction with ISO 3713, ISO 7087 and ISO 7373.

2 References

ISO 3713, *Ferroalloys — Sampling and sample preparation — General rules*.

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

ISO 7373, *Ferroalloys — Experimental methods for checking the precision of sample division*.

3 General requirements

3.1 Methods for sampling, sample preparation and chemical analysis

Sampling, sample preparation and chemical analysis for the experiment shall be carried out in accordance with the methods given in the relevant International Standards.

3.2 Principle

In the experimental method, the results obtained from the method to be checked (Method B) shall be compared with the results of a specified reference method (Method A) which has been regarded as producing practically unbiased results from technical and empirical viewpoints. The comparison shall be made by use of a statistical method of test for significance of difference at a 5 % level of significance (two-sided test).

3.3 Quality characteristic

The quality characteristic on which checking the bias of sampling is carried out shall be that given in the relevant International Standard on the methods for ferroalloy sampling.

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

3.4 Number of experiments

The experiment shall be repeated at least 10 times (for example on either 10 consignments or 10 parts of the consignments; on either 10 gross samples or 10 sub-samples).

3.5 Selection of methods of data analysis

When individual increments taken by Method A and by Method B are correctly paired, the method of analysis for paired data shall be applied. When pairing of individual increments taken by Method A and by Method B is not conducted, the method of analysis for unpaired data shall be applied.

When the number of experiments is the same, the method of analysis for paired data is higher in sensitivity, in a statistical sense, than that for unpaired data in detecting the significance of difference. In order to apply the method of paired data analysis, it is necessary to design and perform the experiment so that pairing of increments, one from Method A and the other from Method B, is assured technically.

The method of analysis for unpaired data is based on the condition that the number of measurements of Method A and of Method B are the same. Care should be taken to obtain the same number of increments for both of the methods.

NOTE — The prescribed methods for statistical analysis of experimental data may also be applied to the case of checking the difference in the results obtained from different samples of one consignment taken at different places, for example, a loading point and a discharging point (see clause 6).

4 Experimental methods

4.1 Examples of experiment

The reference methods (Method A) are different and vary according to the intended purposes, and it is therefore difficult to establish rigid rules. Examples of reference methods are given below. The methods to be checked (Method B) are given in relation to the respective reference methods.

Example 1: Bias due to mass increment

Method A: A reference sample is a composite of increments of larger mass than the mass specified for routine sampling.

Method B: A sample is a composite of increments of equal or smaller masses than those specified for routine sampling which is considered to introduce no bias into the sample.

NOTE — Segregation of quality of ferroalloys is normally observed along the vertical direction of crushed ingots. Care should therefore be taken in taking the increments of reference samples.

Example 2: Sampling of ferroalloys in bulk

Method A: A reference sample is a composite of increments taken from new surfaces of the ferroalloy exposed during loading or unloading a consignment.

Method B: A sample is a composite of increments taken from stockpiles.

Example 3: Sampling of ferroalloys packed in containers

Method A: A reference sample is a composite of the entire quantity of packed units of the ferroalloy taken as the increments.

Method B: A sample is a composite of increments taken from several points selected at random within packed units of the ferroalloy.

Example 4: Drilled sample

Method A: A reference sample is a composite of increments taken by drilling from the top surface through to the bottom surface of ferroalloy lumps having vertical crushed surfaces.

Method B: A sample is a composite of increments taken by drilling in random order from several points on ferroalloy lumps of which the top and bottom surfaces are not clear.

Example 5: Ladle sampling

Method A: A reference sample is a composite of increments of specified mass and specified number taken from a consignment constituted by the tapped lot method.

Method B: A sample is a composite of ladle samples taken from a corresponding consignment.

Example 6: Mechanical sample dividing apparatus

Method A: A reference sample is the rest of a sample obtained after the divided sample has been taken.

Method B: A sample is a divided sample obtained by a routine method.

4.2 Sampling

4.2.1 Two gross samples shall be taken from the same consignment under study or the same part of the consignment: one of the two is by Method A and the other by Method B. The gross sample taken by Method A is designated as gross sample A and the one taken by Method B as gross sample B.

NOTE — For the purposes of checking the difference in the results obtained from different samples of one consignment (see note in 3.5), either of the two samples is regarded as gross sample A and the other as gross sample B.

4.2.2 Each pair of increments for constituting the gross samples A and B shall be taken from the same point of a ferroalloy consignment.

4.2.3 When pairing of increments is not intended, gross sample A shall be the one obtained by Method A and gross sample B the one obtained by Method B.

4.3 Preparation of samples

4.3.1 For the cases of examples 1 through 5 in 4.1, when checking the bias of sampling, the two gross samples A and B shall be processed separately by the same method for the preparation of the respective test samples A and B.

4.3.2 For the case of example 6, when checking the bias of sample preparation, two different test samples A and B shall be prepared separately from the same gross sample or from the same sub-sample.

4.4 Determination and recording

The quality characteristic shall be determined on the test samples A and B by the same method. The results of chemical determination shall be recorded in a data log such as those shown as examples in tables 1 and 2. Table 1 is applicable to the case of paired increments and table 2 to unpaired increments.

Table 1 — Example of a data log of a *t*-test of paired data.
(For explanation of the symbols, see annex A.)

Designation of experiment:

Type and grade of ferroalloy: (for example, ferrochromium A)

Identification of consignment (or gross sample):

Date of experiment:

Increment No.	Quality characteristic [for example, % (*m*/*m*) Cr]			
	x_{Bi}	x_{Ai}	$d_i = x_{Bi} - x_{Ai}$	d_i^2
1				
2				
.				
.				
.				
k				
Sum				

Calculation for *t*-test on one consignment:

$\bar{d}$ = (plus or minus)

V_d =

t_o =

t (ϕ; 0,025) =

Statement on the result of the *t*-test:

NOTE — The experimental conclusion is derived from the overall result obtained after the repetition of experiments on at least ten consignments (or parts of the consignments) of the same type of ferroalloy.

Table 2 — Example of a data log of a *t*-test of unpaired data
(For explanation of the symbols, see annexes B and C.)

Designation of experiment:

Type and grade of ferroalloy: (for example, ferrochromium A)

Identification of consignment (or gross sample):

Date of experiment:

Consignment No.	Grade of ferroalloy	Quality characteristic [for example, % [(*m*/*m*) Cr]					
		Gross sample B			Gross sample A		
		x_{Bi}	X_{Bi}	X_{Bi}^2	x_{Ai}	X_{Ai}	X_{Ai}^2
1							
2							
.							
.							
.							
n							
Sum		Σx_{Bi}	ΣX_{Bi}	ΣX_{Bi}^2	Σx_{Ai}	ΣX_{Ai}	ΣX_{Ai}^2

Calculation for *t*-test on ten consignments:

$\bar{x}_B$ =

S_B =

$\bar{x}_A$ =

S_A =

t_o =

$t(\phi; 0{,}025)$ =

Statement on the result of the *t*-test: