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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

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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Foreword

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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 :

$\overline{d}$ = (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:

Consign-ment No.	Grade of ferro-alloy	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 :

$\overline{x}_B$ =

S_B =

$\overline{x}_A$ =

S_A =

t_o =

t(ϕ; 0,025) =

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

5 Data analysis

5.1 Test for significance of difference

The test for significance of difference between two experimental results, in other words, the deviation of the results of Method B from the results of reference Method A, shall be carried out by a statistical method called the t-test. For unpaired data, prior to conducting the t-test, the F-test for equality of the two variances of both of the methods shall be carried out.

The analysis of data according to the note in 3.5 shall be made by the procedure given in 5.3.

5.2 Paired data

5.2.1 Calculate the observed value of t, denoted by t_o. The method of calculation is given in annex A.

5.2.2 Compare the absolute value of t_o obtained by experiment with the $t(\phi;$ 0,025) point obtained from table 3 (see annex A):

When $|t_o| < t(\phi; 0{,}025)$, then $\bar{d}$ is not significant.

When $|t_o| \geqslant t(\phi; 0{,}025)$, then $\bar{d}$ is significant.

NOTE — See clause 6 for the conclusions that may be drawn from the outcome of these tests.

5.3 Unpaired data

5.3.1 The equality of the variance of the results of Method A and the variance of the results of Method B shall be tested by a statistical method called the F-test referred to as the variance ratio test. The method of calculation is given in annex B.

5.3.2 Compare the observed F_o value obtained by experiment with the $F(\phi;$ 0,05) point obtained from table 3 (see annex B).

When $F_o < F(\phi; 0{,}05)$, then the test is passed.

When $F_o \geqslant F(\phi; 0{,}05)$, then the test is failed.

5.3.3 If the F-test is passed, apply the t-test. If the F-test is failed, reject the experimental results, and, if necessary, carry out a further experiment.

5.3.4 The method of calculation for the t-test for significance of difference is given in annex C.

5.3.5 Compare the absolute value of t_o obtained by experiment with the $t(\phi;$ 0,025) point obtained from table 3 (see annex A).

When $|t_o| < t(\phi; 0{,}025)$, then $\bar{d}$ is not significant.

When $|t_o| \geqslant t(\phi; 0{,}025)$, then $\bar{d}$ is significant.

NOTE — See clause 6 for the conclusions that may be drawn from the outcome of these tests.

6 Review of experimental results

In the event that the difference between the results obtained from Method B and Method A is insignificant by the t-test, then Method B may be adopted as a routine method, provided that an agreement is reached between the interested parties, where such is required.

The following conditions shall also be taken into account:

a) The absence of bias means that the value obtained from routine operations does not depart from the true value or reference value in this method with a statistically significant difference.

b) If, even though the difference is statistically significant, it is regarded as being negligible from a practical standpoint, Method B may be adopted as a routine method, provided that the interested parties agree, where this is required.

c) When the difference is statistically insignificant but is regarded as being so large as to be significant from a practical standpoint by the interested parties, a further experiment shall be carried out.

Annex A

Method of calculation of the t-test for paired data

(This annex forms an integral part of the Standard.)

A.1 Calculate the difference between paired measurements :

$$d_i = x_{Bi} - x_{Ai} \qquad i = 1, 2, \ldots, k \qquad \ldots (1)$$

where

d_i is the ith difference or deviation of x_{Bi} from x_{Ai};

x_{Bi} is the ith measurement of sample B obtained by method B to be controlled;

x_{Ai} is the ith measurement of sample A obtained by the reference method A;

k is the number of pairs of measurements.

A.2 Calculate the mean value of differences to one decimal place more than that used in the measurements themselves:

$$\bar{d} = \frac{1}{k} \sum_{i=1}^{k} d_i \qquad \ldots (2)$$

where

$\bar{d}$ is the mean value of k differences.

A.3 Calculate the unbiased estimate of variance of the difference:

$$V_d = \frac{1}{\phi} \left\{ \sum_{i=1}^{k} d_i^2 - \left(\sum_{i=1}^{k} d_i \right)^2 / k \right\} \qquad \ldots (3)$$

where

V_d is the unbiased estimate of variance of the differences;

ϕ is the number of degrees of freedom — in this method $\phi = k - 1$.

A.4 Calculate the observed value of t, denoted by t_o, by rounding off to the third decimal place:

$$t_o = \frac{\bar{d}}{\sqrt{\frac{V_d}{k}}} \qquad \ldots (4)$$

A.5 Obtain the statistic t point at a 2,5 % level of significance for ϕ degrees of freedom, denoted by $t(\phi; 0{,}025)$, from table 3.

Table 3 — Point of $t(\phi;$ 0,025) (two-sided)

ϕ	9	10	11	12	13	14
t	2,262	2,228	2,201	2,179	2,160	2,145
ϕ	15	16	17	18	19	20
t	2,131	2,120	2,110	2,101	2,093	2,086