International Standard ISO 7087

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Ferroalloys — Experimental methods for the evaluation of the quality variation and methods for checking the precision of sampling

Ferro-alliages — Méthodes expérimentales d'évaluation de la variation de qualité et méthodes de contrôle de la fidélité de l'échantillonnage

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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.

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 7087 was prepared by Technical Committee ISO/TC 132, *Ferroalloys*.

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Example of two other methods for the calculation of quality variation value (for two-stage sampling) for an experiment of 10 consignments . . . 12

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Ferroalloys — Experimental methods for the evaluation of the quality variation and methods for checking the precision of sampling

1 Scope and field of application

This International Standard specifies experimental methods for the evaluation of quality variation of ferroalloys for the purposes of determining the parameters of random sampling and two-stage sampling given in the relevant International Standards. It also specifies the methods for checking the precision of taking samples by the random method and two-stage method.

2 References

ISO 3713, *Ferroalloys — Sampling and sample preparation — general rules.*[1]

ISO 4552/1, *Ferroalloys — Sampling and sample preparation for chemical analysis — Part 1 : Ferrochromium, ferrosilicochromium, ferrosilicon, ferrosilicomanganese and ferromanganese.*[1]

ISO 4552/2, *Ferroalloys — Sampling and sample preparation for chemical analysis — Part 2 : Ferrotitanium, ferromolybdenum, ferrotungsten, ferroniobium and ferrovanadium.*[1]

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

ISO 5446, *Ferromanganese — Specification and conditions of delivery.*

ISO 5447, *Ferrosilicomanganese — Specification and conditions of delivery.*

ISO 5448, *Ferrochromium — Specification and conditions of delivery.*

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

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

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

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

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

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

ISO 7347, *Ferroalloys — Experimental methods for checking the bias of sampling and sample preparation.*[1]

ISO 7373, *Ferroalloys — Experimental methods for checking the precision of sample division.*[1]

3 General requirements for experiment

3.1 Quality variation

The quality variation is a measure of heterogeneity of the ferroalloy and is expressed in terms of the standard deviation denoted by σ. It shall be the standard deviation between increments (σ_i) for random sampling, and the standard deviations between packed units (σ_b) and within packed units (σ_w) for two-stage sampling.

1) At present at the stage of draft.

NOTES

1 Random sampling is applied to consignments of ferroalloys, whether crushable or uncrushable, delivered in bulk.

2 Two-stage sampling is applied to consignments delivered in packed units.

3.2 Quality characteristic

The quality characteristic for the determination of the quality variation is given in the relevant International Standards on methods for ferroalloy sampling.

The content of any other element may be selected as the quality characteristic by mutual agreement between the parties concerned.

3.3 Evaluation of the quality variation of ferroalloys

The quality variation shall be evaluated for each type of ferroalloy as designated between the parties concerned.

3.4 Consignments for experiment

The value of quality variation of a consignment of a ferroalloy is related to the method of constituting the consignment. Three methods are practised depending upon the process of production and the type of ferroalloy. These are tapped lot method, graded lot method and blended lot method.

For the tapped lot method, the value of the quality variation tends to be small and depends on the degree of crushing and on the thoroughness of mixing of the material. If the experiment is conducted on consignments constituted by this method, it is liable to underestimate the value of the quality variation.

For the graded lot method, the difference between the taps constituting a consignment shall be as given in the relevant International Standards on technical conditions for delivery of ferroalloys. Quality variation of other differences between the taps of a consignment may be determined by agreement between the parties concerned.

Quality variation experiments are preferably carried out on consignments constituted by the graded lot method, in order to obtain the most reliable estimate of quality variation.

3.5 Method for sampling and chemical analysis

Sampling, preparation of samples and chemical analysis for experimental purposes shall be carried out in accordance with the relevant International Standards.

3.6 Number of experiments

The experiment shall be conducted on one consignment. For random sampling, an experiment shall cover either the whole consignment or part of the consignment. For two-stage sampling, it shall cover m packed units out of M packed units of the consignment.

The experiment shall be repeated at least 10 times.

3.7 Order of chemical determinations

The sequence of chemical determinations of a set of experimental test samples shall be in random order.

4 Experimental methods

4.1 Types of experiment

4.1.1 Type I for ferroalloys sampled by the random method

This type of experiment is applied to ferroalloys in bulk.

4.1.2 Type II for ferroalloys sampled by the two-stage method

This type of experiment is applied to ferroalloys in packed units.

4.2 Type I

4.2.1 Method for crushable ferroalloys (see figure 1)

This method is applicable to ferroalloys of which increments of a sample are obtained with a sampling device such as an increment shovel.

The number of increments to be taken from a ferroalloy consignment shall be 10 or more.

Duplicate test samples shall be prepared from each of the increments.

A single chemical determination of the quality characteristic shall be carried out on each of the test samples in random order.

The data for the experiment shall be recorded on a data log such as that given in table 1 as an example.

4.2.2 Method for uncrushable ferroalloys (see figure 2)

This method is applicable to ferroalloys of which increments of a sample are obtained as chippings from each of the selected lumps by means of a drilling machine.

The number of lumps to be taken from a ferroalloy consignment delivered in bulk shall be 10 or more.

Each increment as a mass of chippings shall be taken from each of the selected lumps.

Duplicate test samples shall be prepared from each increment.

A single chemical determination of the quality characteristic shall be carried out on each of the test samples in random order.

The data for the experiment shall be recorded on a data log such as that given in table 1 as an example.

4.3 Type II

This method is applied to consignments of ferroalloys, whether crushable or uncrushable, delivered in packed units (see figure 3).

A total of m packed units shall be selected at the first stage of two-stage sampling.

NOTE — For the sake of convenience in the treatment of the data, it is recommended that the number m be an even number.

At the second stage of two-stage sampling, each of four increments in the form of particles or chippings shall be taken from within each of the selected packed units.

Two different binary subsamples denoted by A, B; C, D, composed each of four increments, shall be constituted as follows : A and B each consist of one increment from each of the four selected packed units; C consists of two increments from each of the even-number packed units; D consists of two increments from each of the two odd-number packed units.

The test samples shall be prepared from the subsamples as follows : two test samples for each of the subsamples A and C; one test sample for each of the subsamples B and D.

A single chemical determination of the quality characteristic shall be carried out on each of the test samples in random order.

The data for an experiment shall be recorded on a data log such as that given in table 2 as an example.

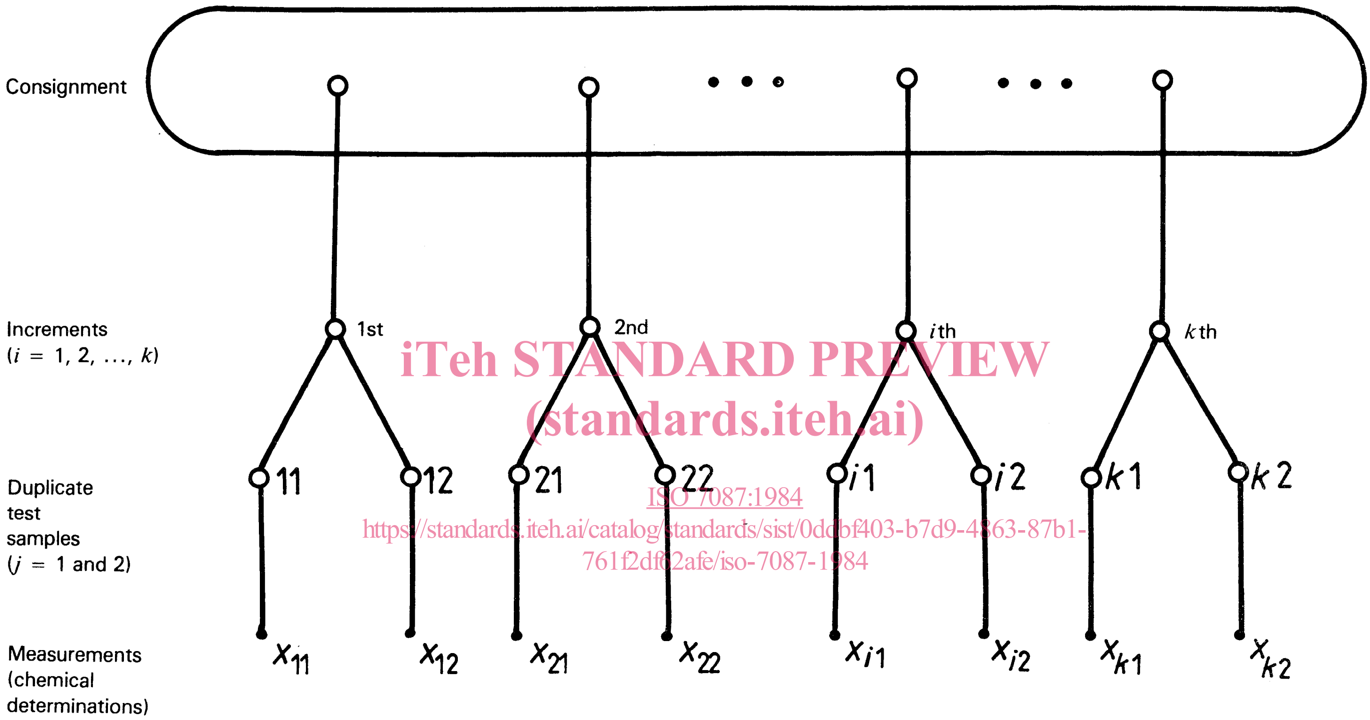

Figure 1 — Flow scheme for crushable ferroalloys (Type I)

Table 1 — Data log for the experiment (Type I) (Example for $k = 10$)

Data sheet No. :

Particulars of consignment :

Type and name of ferroalloy :
Method of constitution of consignment :
Designation and mass of consignment :
Other identification :

Name of company and works :

Particulars of experiments :

Dates of experiment :
Mass and number of increments or lumps :
Quality characteristics
(e.g., % Mn for ferromanganese) :

Increment number	Duplicate measurements x_{ij} (e.g. % Mn)		Arithmetic mean $\overline{x}_i$	Ranges R_i
	$j = 1$	$j = 2$		
1	x_{11}	x_{12}	$\overline{x}_{1.} = (x_{11} + x_{12})/2$	$R_1 = \lvert x_{11} - x_{12} \rvert$
2	x_{21}	x_{22}	$\overline{x}_{2.} = (x_{21} + x_{22})/2$	$R_2 = \lvert x_{21} - x_{22} \rvert$
3	x_{31}	x_{32}	$\overline{x}_{3.} = (x_{31} + x_{32})/2$	$R_3 = \lvert x_{31} - x_{32} \rvert$
4	x_{41}	x_{42}	$\overline{x}_{4.} = (x_{41} + x_{42})/2$	$R_4 = \lvert x_{41} - x_{42} \rvert$
5	x_{51}	x_{52}	$\overline{x}_{5.} = (x_{51} + x_{52})/2$	$R_5 = \lvert x_{51} - x_{52} \rvert$
6	x_{61}	x_{62}	$\overline{x}_{6.} = (x_{61} + x_{62})/2$	$R_6 = \lvert x_{61} - x_{62} \rvert$
7	x_{71}	x_{72}	$\overline{x}_{7.} = (x_{71} + x_{72})/2$	$R_7 = \lvert x_{71} - x_{72} \rvert$
8	x_{81}	x_{82}	$\overline{x}_{8.} = (x_{81} + x_{82})/2$	$R_8 = \lvert x_{81} - x_{82} \rvert$
9	x_{91}	x_{92}	$\overline{x}_{9.} = (x_{91} + x_{92})/2$	$R_9 = \lvert x_{91} - x_{92} \rvert$
10	x_{101}	x_{102}	$\overline{x}_{10.} = (x_{101} + x_{102})/2$	$R_{10} = \lvert x_{101} - x_{102} \rvert$

$k = 10$ $\quad V = S/(10 - 1)$ $\quad \hat{\sigma}_i = \sqrt{V - \sigma^2_{DM}/2}$

$\hat{\sigma}_{DM} = \overline{R}/d_2;\quad d_2 = 1{,}128$

$$S = \left(\overline{x}^2_{1.} + \overline{x}^2_{2.} + \ldots + \overline{x}^2_{10.}\right) - \frac{1}{10}\left(\overline{x}_{1.} + \overline{x}_{2.} + \ldots + \overline{x}_{10.}\right)^2$$

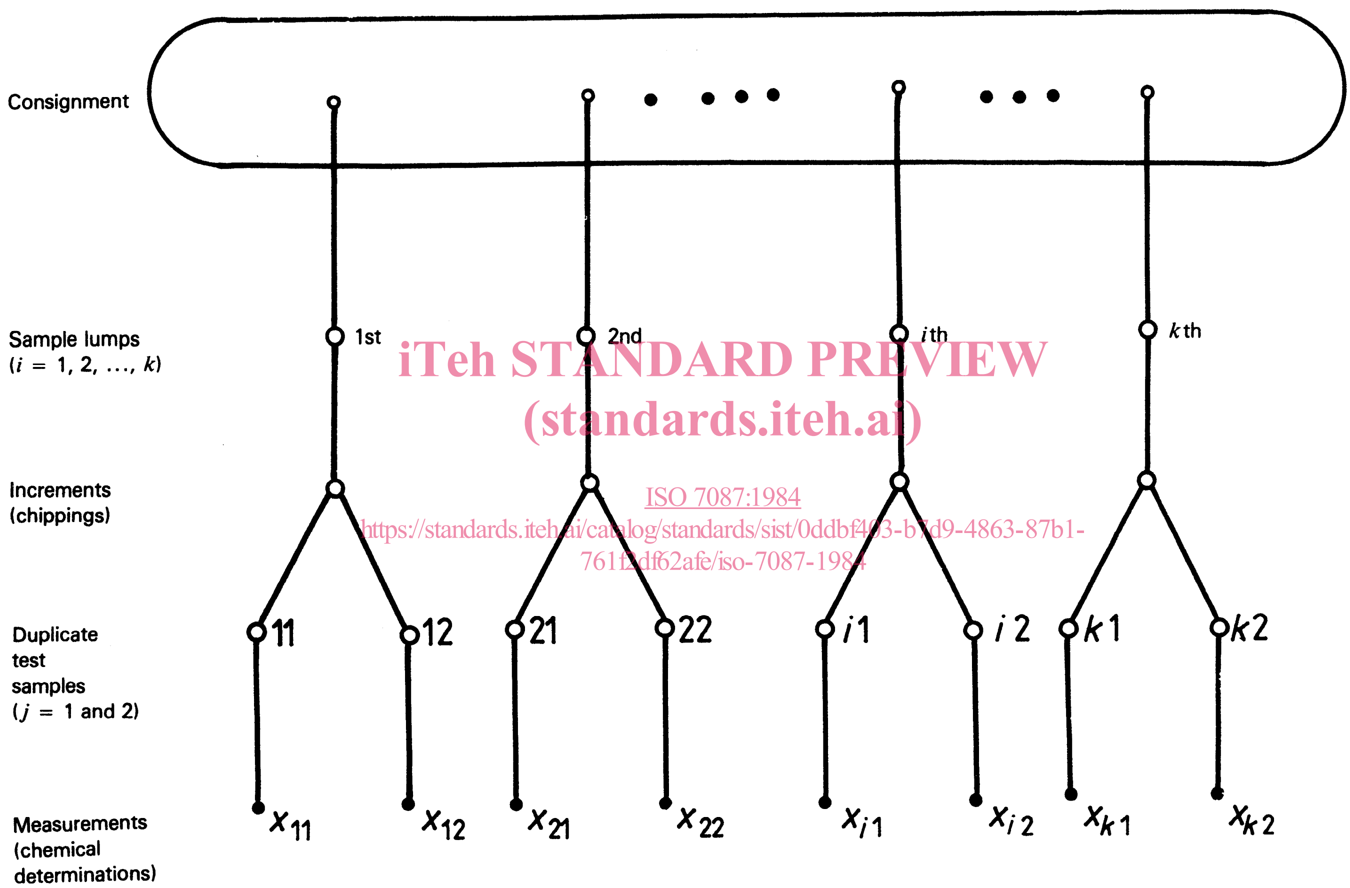

Figure 2 — Flow scheme for uncrushable ferroalloys

Table 2 — Data log for the experiment (Type II) (Example for p = 10)

Data sheet No. :
Particulars of consignment :
Type and name of ferroalloy :
Method of constitution of consignment :
Other identification :

Name of company and works :
Particulars of experiments :
Dates of experiment :
Mass and number of increments or lump
Quality characteristics
(e.g., % Mn for ferromanganese) :

Number of experiment	Measurements of AB pairs				Measurements of CD pairs				
	A_{ij}	R_{Aj}	B_i	R_{ABi}	C_{ij}	R_{Ci}	D_i	R_{CDi}	$\overline{x}_1$
1	A_{11}, A_{12}	$R_{A1} = \|A_{11} - A_{12}\|$	B_1	$R_{AB1} = \|A_{11} - B_1\|$	C_{11}, C_{12}	$R_{C1} = \|C_{11} - C_{12}\|$	D_1	$R_{CD1} = \|C_{12} - D_1\|$	$\overline{x}_1$
2	A_{21}, A_{22}	$R_{A2} = \|A_{21} - A_{22}\|$	B_2	$R_{AB2} = \|A_{21} - B_2\|$	C_{21}, C_{22}	$R_{C2} = \|C_{21} - C_{22}\|$	D_2	$R_{CD2} = \|C_{22} - D_2\|$	$\overline{x}_2$
3	A_{31}, A_{32}	$R_{A3} = \|A_{31} - A_{32}\|$	B_3	$R_{AB3} = \|A_{31} - B_3\|$	C_{31}, C_{32}	$R_{C3} = \|C_{31} - C_{32}\|$	D_3	$R_{CD3} = \|C_{32} - D_3\|$	$\overline{x}_3$
4	A_{41}, A_{42}	$R_{A4} = \|A_{41} - A_{42}\|$	B_4	$R_{AB4} = \|A_{41} - B_4\|$	C_{41}, C_{42}	$R_{C4} = \|C_{41} - C_{42}\|$	D_4	$R_{CD4} = \|C_{42} - D_4\|$	$\overline{x}_4$
5	A_{51}, A_{52}	$R_{A5} = \|A_{51} - A_{52}\|$	B_5	$R_{AB5} = \|A_{51} - B_5\|$	C_{51}, C_{52}	$R_{C5} = \|C_{51} - C_{52}\|$	D_5	$R_{CD5} = \|C_{52} - D_5\|$	$\overline{x}_5$
6	A_{61}, A_{62}	$R_{A6} = \|A_{61} - A_{62}\|$	B_6	$R_{AB6} = \|A_{61} - B_6\|$	C_{61}, C_{62}	$R_{C6} = \|C_{61} - C_{62}\|$	D_6	$R_{CD6} = \|C_{62} - D_6\|$	$\overline{x}_6$
7	A_{71}, A_{72}	$R_{A7} = \|A_{71} - A_{72}\|$	B_7	$R_{AB7} = \|A_{71} - B_7\|$	C_{71}, C_{72}	$R_{C7} = \|C_{71} - C_{72}\|$	D_7	$R_{CD7} = \|C_{72} - D_7\|$	$\overline{x}_7$
8	A_{81}, A_{82}	$R_{A8} = \|A_{81} - A_{82}\|$	B_8	$R_{AB8} = \|A_{81} - B_8\|$	C_{81}, C_{82}	$R_{C8} = \|C_{81} - C_{82}\|$	D_8	$R_{CD8} = \|C_{82} - D_8\|$	$\overline{x}_8$
9	A_{91}, A_{92}	$R_{A9} = \|A_{91} - A_{92}\|$	B_9	$R_{AB9} = \|A_{91} - B_9\|$	C_{91}, C_{92}	$R_{C9} = \|C_{91} - C_{92}\|$	D_9	$R_{CD9} = \|C_{92} - D_9\|$	$\overline{x}_9$
10	A_{101}, A_{102}	$R_{A10} = \|A_{101} - A_{102}\|$	B_{10}	$R_{AB10} = \|A_{101} - B_{10}\|$	C_{101}, C_{102}	$R_{C10} = \|C_{101} - C_{102}\|$	D_{10}	$R_{CD10} = \|C_{102} - D_{10}\|$	$\overline{x}_1$

$$\hat{\sigma}_{DM} = \frac{\overline{R}}{d_2}, \quad \overline{R} = \frac{1}{20}\left(\sum_{i=1}^{10} R_{Ai} + \sum_{i=1}^{10} R_{Ci}\right)$$

$$\overline{R}_{AB} = \frac{1}{10}\sum_{i=1}^{10} R_{ABi}$$

$\overline{R}$

$$\hat{\sigma}_w^2 = m\left[\left(\frac{\overline{R}_{AB}}{d_2}\right)^2 - \sigma_{DM}^2\right]$$

$$\hat{\sigma}_b^2 = m\left(\frac{\overline{R}_{CD}^2 - \overline{R}_{AB}^2}{2\,d_2^2}\right)$$

$$d_2 = 1{,}128$$