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

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Contents

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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.

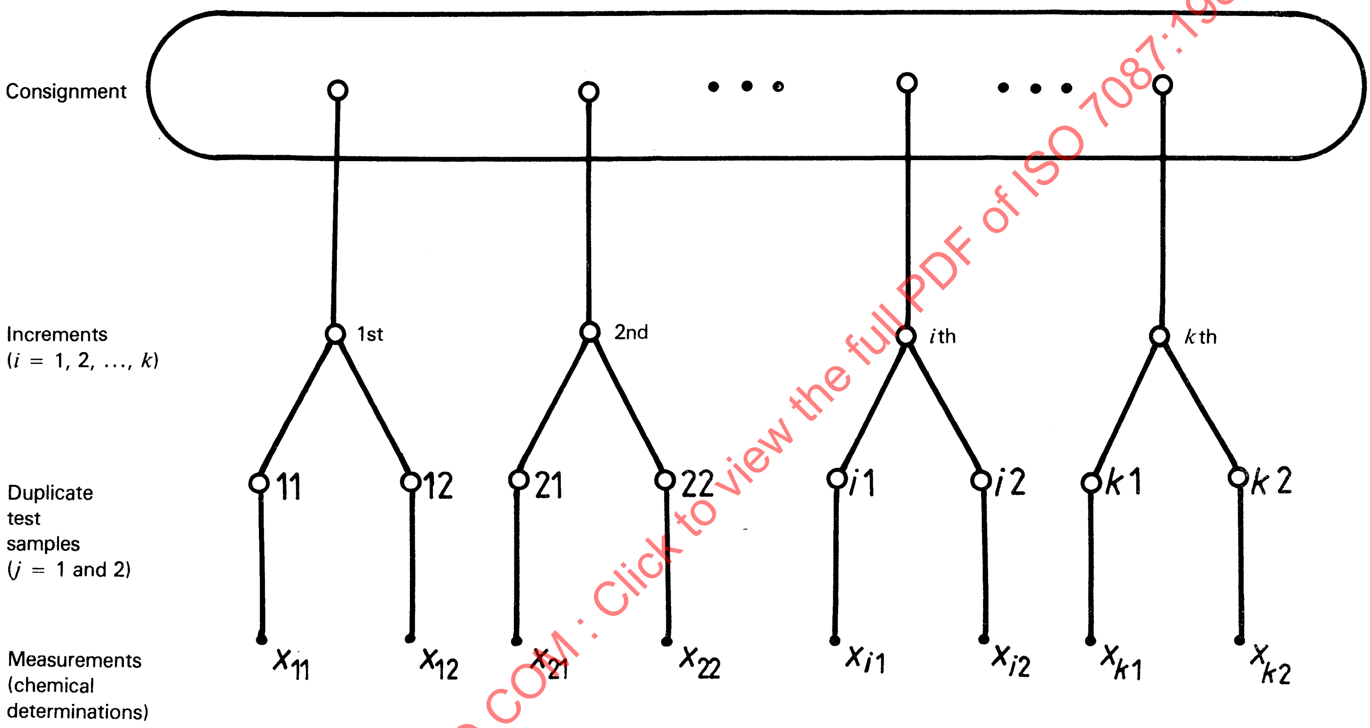

Figure 1 — Flow scheme for crushable ferroalloys (Type I)

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 $\bar{x}_i$	Ranges R_i
	$j = 1$	$j = 2$		
1	x_{11}	x_{12}	$\bar{x}_{1.} = (x_{11} + x_{12})/2$	$R_1 = \|x_{11} - x_{12}\|$
2	x_{21}	x_{22}	$\bar{x}_{2.} = (x_{21} + x_{22})/2$	$R_2 = \|x_{21} - x_{22}\|$
3	x_{31}	x_{32}	$\bar{x}_{3.} = (x_{31} + x_{32})/2$	$R_3 = \|x_{31} - x_{32}\|$
4	x_{41}	x_{42}	$\bar{x}_{4.} = (x_{41} + x_{42})/2$	$R_4 = \|x_{41} - x_{42}\|$
5	x_{51}	x_{52}	$\bar{x}_{5.} = (x_{51} + x_{52})/2$	$R_5 = \|x_{51} - x_{52}\|$
6	x_{61}	x_{62}	$\bar{x}_{6.} = (x_{61} + x_{62})/2$	$R_6 = \|x_{61} - x_{62}\|$
7	x_{71}	x_{72}	$\bar{x}_{7.} = (x_{71} + x_{72})/2$	$R_7 = \|x_{71} - x_{72}\|$
8	x_{81}	x_{82}	$\bar{x}_{8.} = (x_{81} + x_{82})/2$	$R_8 = \|x_{81} - x_{82}\|$
9	x_{91}	x_{92}	$\bar{x}_{9.} = (x_{91} + x_{92})/2$	$R_9 = \|x_{91} - x_{92}\|$
10	x_{101}	x_{102}	$\bar{x}_{10.} = (x_{101} + x_{102})/2$	$R_{10} = \|x_{101} - x_{102}\|$

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

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

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

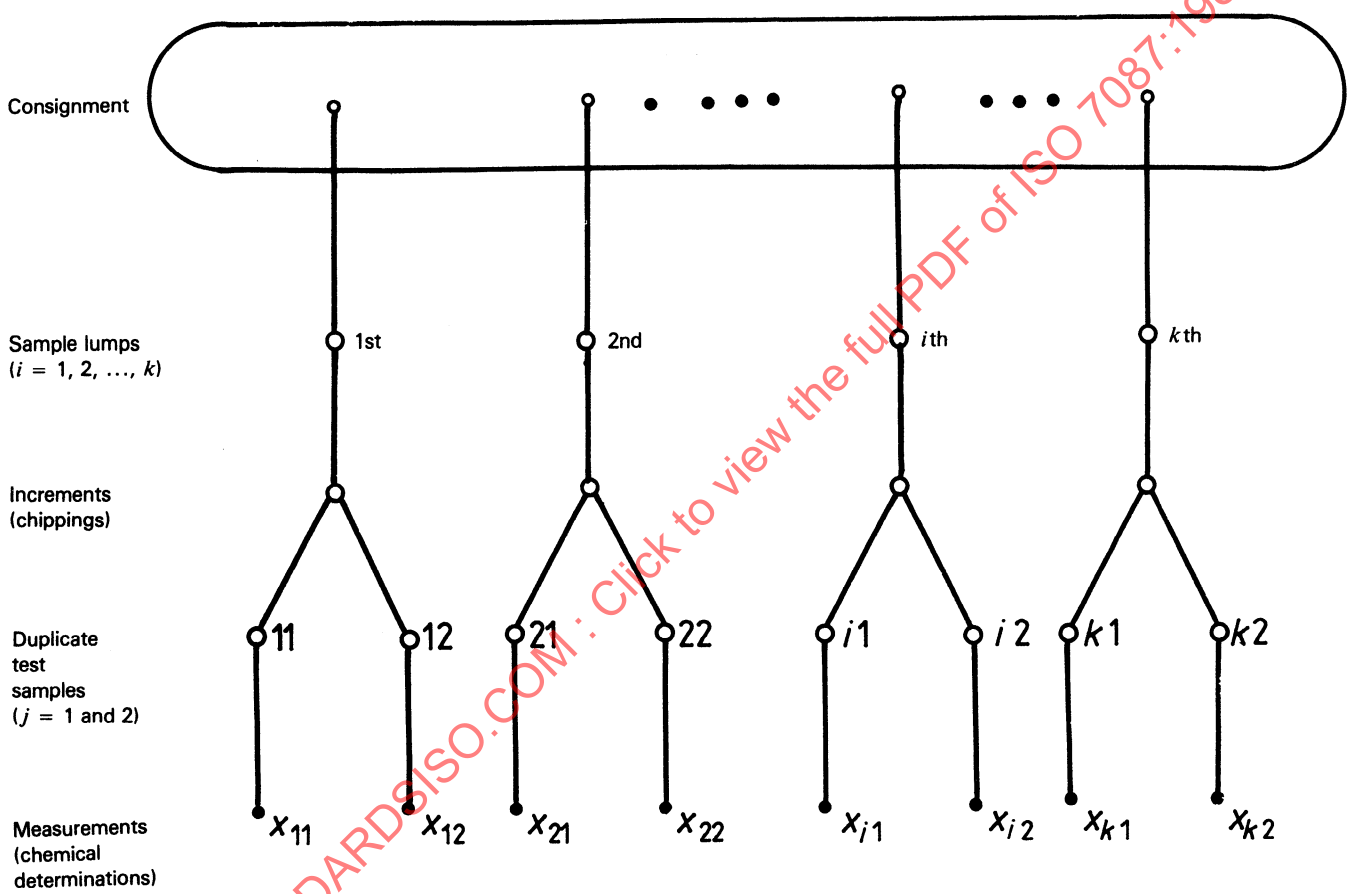

Figure 2 — Flow scheme for uncrushable ferroalloys

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 lumps

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}	$R_{CD10} = \|C_{102} - D_{10}\|$	$\overline{x}_1$

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

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

$\overline{R}$

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

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

$$d_2 = 1{,}128$$

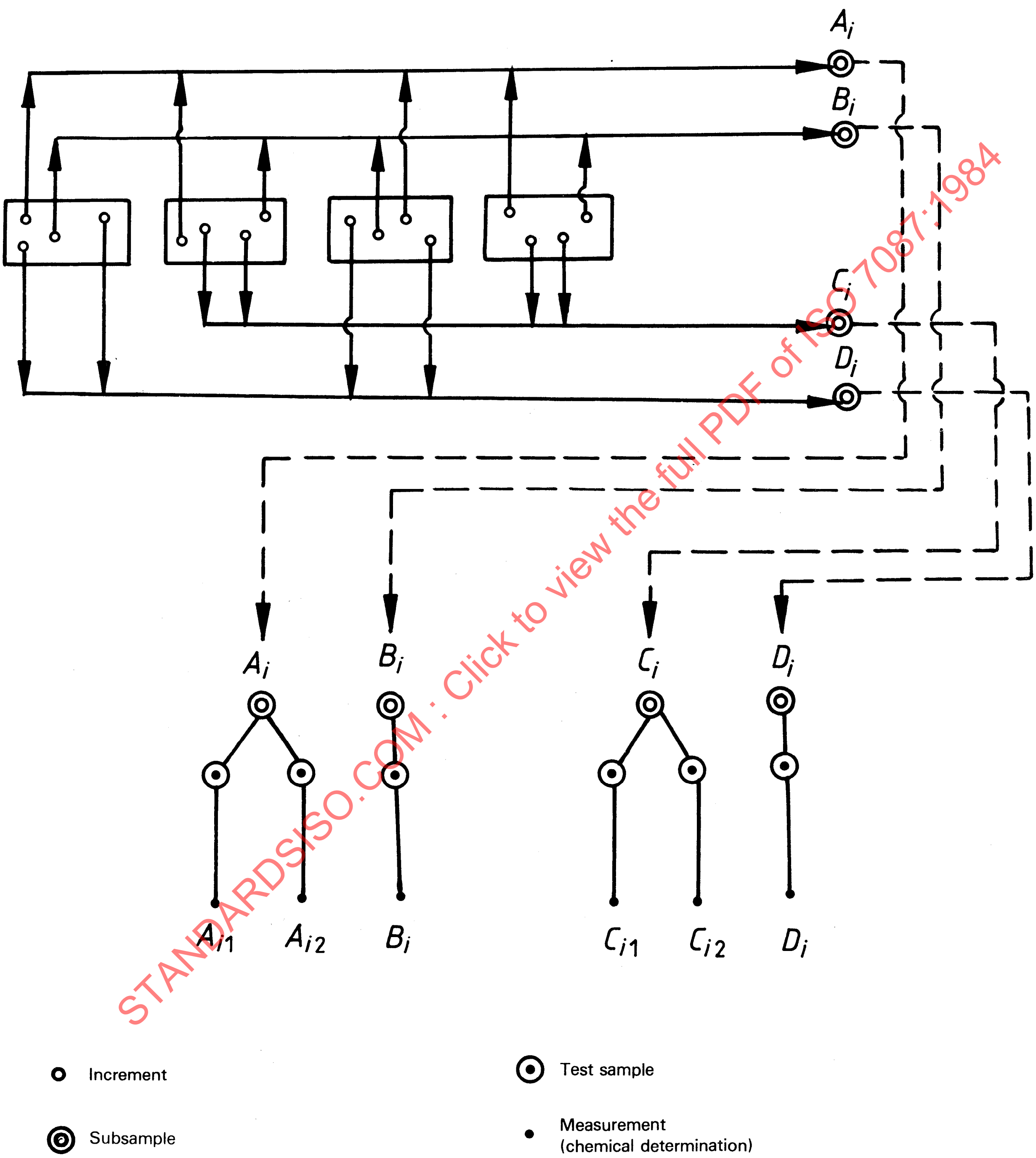

Figure 3 — Flow scheme for ferroalloys in packed units (Type II)
(Example for $m = 4$)

5 Methods for analysis of experimental data

5.1 Selection of the method

When the experiment is conducted according to 4.2 for ferroalloys in bulk, whether crushable (4.2.1) or uncrushable (4.2.2), the method of data analysis for the random method (5.2) shall be applied. When it is conducted according to clause 4.3 for ferroalloys in packed units, the method for data analysis of two-stage sampling (5.3) shall be applied.

NOTE — During the process of data analysis, if the calculated value of the variance is turned out to be a negative one, it should be regarded as being zero ($\sigma^2 = 0$) provided that no causative defects in the experimental operations can be observed.

5.2 Method for data analysis for random sampling

The procedure for evaluation of the quality variation between increments shall be as given below.

5.2.1 Calculate the range of paired measurements

$$R_i = |x_{i1} - x_{i2}| \qquad \ldots (1)$$

where x_{i1}, x_{i2} are the measurements of each of the duplicate test samples of the ith increment.

5.2.2 Calculate the mean range

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

where k is the number of ranges of experiments.

5.2.3 Calculate the estimated value of combined variance of division and measurement

$$\hat{\sigma}^2_{DM} = \left(\frac{\bar{R}}{d_2}\right)^2 \qquad \ldots (3)$$

where d_2 is the factor for obtaining the standard deviation from the range;
d_2 = 1,128 for paired measurements.

5.2.4 Calculate the estimated value of the unbiased variance :

$$V = \frac{1}{k-1} S \qquad \ldots (4)$$

$$S = \sum_{i=1}^{k}\left(\bar{x}^2_{i.}\right) - \frac{1}{k}\left(\sum_{i=1}^{k}\bar{x}_{i.}\right)^2 \qquad \ldots (5)$$

where

S is the sum of squares expressed by a working formula;

$\bar{x}_{i.}$ is the arithmetic mean of paired measurements of the ith increment.

5.2.5 Calculate the estimated value of the quality variation between increments, using the results based on the data from a single experiment obtained from formulae (3), (4) and (5) :

$$\hat{\sigma}^2_i = V - \frac{1}{2}\hat{\sigma}^2_{DM} \qquad \ldots (6)$$

5.3 Method for data analysis for two-stage sampling

The procedure for evaluation of the quality variation between packed units and the quality variation within packed units shall be as given below.

5.3.1 Calculate the range of measurements of the duplicate test samples of subsample A_i and of subsample C_i :

$$R_{Ai} = |A_{i1} - A_{i2}| \qquad \ldots (7)$$

$$R_{Ci} = |C_{i1} - C_{i2}| \qquad \ldots (8)$$

5.3.2 Calculate the arithmetic mean of two different ranges :

$$\bar{R} = \frac{1}{2p}\left(\sum_{i=1}^{p} R_{Ai} + \sum_{i=1}^{p} R_{Ci}\right) \qquad \ldots (9)$$

where p is the number of experiments.

5.3.3 Calculate the estimated value of combined variance of division and measurement :

$$\hat{\sigma}^2_{DM} = \left(\frac{\bar{R}}{d_2}\right)^{2*} \qquad \ldots (10)$$

5.3.4 Calculate the ranges of the binary subsamples :

$$R_{ABi} = |A_{i1} - B_i| \quad \text{or}$$

$$R_{ABi} = |A_{i2} - B_i| \qquad \ldots (11)$$

$$R_{CDi} = |C_{i1} - D_i| \quad \text{or}$$

$$R_{CDi} = |C_{i2} - D_i| \qquad \ldots (12)$$

where

R_{ABi} and R_{CDi} are the ranges of the respective paired measurements of the binary subsamples A, B; C, D of the ith experiment;

A_i, B_i and C_i, D_i are the respective measurements of the test samples of the two binary subsamples of the ith experiment.

* For other methods of calculation, see the annex.

5.3.5 Calculate the estimated values of the quality variation between packed units and the quality variation within packed units as follows :

$$\hat{\sigma}_b^2 = m\left(\frac{\overline{R}_{CD}^2 - \overline{R}_{AB}^2}{2d_2^2}\right) \quad \ldots (13)$$

$$\hat{\sigma}_w^2 = m\left\{\left(\frac{\overline{R}_{AB}}{d_2}\right)^2 - \hat{\sigma}_{DM}^2\right\}^* \quad \ldots (14)$$

where

$\overline{R}_{AB}$ and $\overline{R}_{CD}$ are the respective means of k ranges of R_{ABi} and R_{CDi};

m is the number of packed units selected at the first stage of two-stage sampling.

6 Expression of experimental results

6.1 Random sampling

The arithmetic mean of 10 or more estimated values of the standard deviation between increments shall be calculated by the following formula :

$$\hat{\sigma}_i = \sqrt{\frac{1}{h}\sum_{j=1}^{h}\hat{\sigma}_{ij}^2} \quad \ldots (15)$$

where

$\hat{\sigma}_{ij}^2$ is the value obtained by formula (6) for the jth experiment;

h is the number of values of $\hat{\sigma}_{ij}^2$.

6.2 Two-stage sampling

The respective estimated values of the standard deviation between packed units and the standard deviation within packed units shall be the positive square roots of the values obtained by the respective formulae (13) and (14).

7 Estimation of precision of sampling

7.1 Method for random sampling

7.1.1 Theoretical background

a) The formula used to determine the number of increments, n, taken from a consignment for random sampling has been based on the statistical theory of random sampling with a parameter σ_i.

b) When n increments of a gross sample are taken, the two-sigma precision of sampling is given by

$$\beta_S = 2\sigma_S = 2\sqrt{\frac{\sigma_i^2}{n}} \quad \ldots (16)$$

7.1.2 Estimation of precision of sampling

Calculate the estimated value of β_S (denoted by $\hat{\beta}_S$) by the following formula :

$$\hat{\beta}_S = 2\sqrt{\frac{\hat{\sigma}_i^2}{n}} \quad \ldots (16')$$

where

$\hat{\sigma}_i^2$ is the value obtained by formula (15);

n is the number of increments of the sample taken from a consignment of which β_S is under study.

7.2 Method for two-stage sampling

7.2.1 Theoretical background

a) The practice for selecting the packed units and taking the increments from the selected packed units has been based on the statistical theory of multi-sampling.

b) When m packed units are selected at the first stage from a consignment consisting of M packed units and $\overline{n}$ increments are taken from each of the selected packed units at the second stage, the two-sigma precision of sampling is given by

$$\beta_S = 2\sqrt{\left(\frac{M-m}{M-1}\right)\sigma_b^2/m + \sigma_w^2/m\overline{n}} \quad \ldots (17)$$

c) When $m/M \leqslant 0{,}1$, the multiplier $(M-m)/(M-1)$ is regarded as being unity, then formula (17) is reduced to

$$\beta_S = 2\sqrt{\sigma_b^2/m + \sigma_w^2/m\overline{n}} \quad \ldots (18)$$

d) When $m = M$, the first term in the square root of formula (17) becomes zero, then it is reduced to

$$\beta_S = 2\sqrt{\sigma_w^2/m\overline{n}} \quad \ldots (19)$$

* For other methods of calculation, see the annex.

7.2.2 Estimation of precision of sampling

The procedure of calculation shall be as follows :

a) Calculate $\hat{\beta}_S$ by the following formula :

$$\hat{\beta}_S = 2\sqrt{\left(\frac{M - m}{M - 1}\right)\hat{\sigma}_b^2/m + \hat{\sigma}_w^2/m\bar{n}} \qquad \ldots (20)$$

where

$\hat{\sigma}_b^2, \hat{\sigma}_w^2$ are the respective values obtained by formulae (13) and (14);

M is the number of packed units of the consignment of which β_S is under study;

m is the number of packed units selected;

$\bar{n}$ is the number of increments taken from each of the packed unit selected.

b) When $m/M \leqslant 0{,}1$, the multiplier $(M - m)/(M - 1)$ is regarded as being unity, and $\hat{\beta}_S$ is calculated as follows :

$$\hat{\beta}_S = 2\sqrt{\hat{\sigma}_b^2/m + \hat{\sigma}_w^2/m\bar{n}} \qquad \ldots (21)$$

c) When $m = M$, the first term in the square root of formula (20) becomes zero, then it is reduced to

$$\beta_S = 2\sqrt{\hat{\sigma}_w^2/m\bar{n}} \qquad \ldots (22)$$

7.3 Actions after review of estimated results

In the event that there is an indication that the precision of sampling does not attain the specified value given in the relevant International Standards, in addition to the maintenance of the succession in routine operations, the following actions shall be included :

a) When it is confirmed that there is a recognizable change in the quality variation, consult the authorative organization in charge for a change of increments, for random sampling and change the number of packed units to be selected at the first stage of two-stage sampling.

b) Where practicable, take a greater number of increments than that specified by increasing either n or m.

NOTE — The contribution of this action to the improvement of the precision of sampling is in proportion to $\sqrt{n/n_1}$ where $n_1 > n$.

c) Where practicable, it is recommended that the mass of an increment be increased.

NOTE — Increasing the mass of an increment far more than that required will not effect substantial improvement of the precision of sampling.

Annex

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

A.1 The calculation of the quality variation within packed units ($\hat{\sigma}^2_{\mathrm{w}}$) may be carried out by the method of variance analysis.

Calculate the sum of the squares for quality variation within packed units (S_{w}) :

$$S_{\mathrm{w}} = \frac{1}{2}\sum_{i=1}^{10}(A_{i1} + A_{i2})^2 + \sum_{i=1}^{10} B_i^2 - \frac{1}{3}\sum_{i=1}^{10}(A_{i1} + A_{i2} + B_i)^2 \qquad \ldots (23)$$

Calculate the sum of the squares for the variance of sample preparation and chemical analysis of samples (S_{DM}) :

$$S_{\mathrm{DM}} = \sum_{i=1}^{10} A_{i1}^2 + \sum_{i=1}^{10} A_{i2}^2 - \frac{1}{2}\sum_{i=1}^{10}(A_{i1} + A_{i2})^2 \qquad \ldots (24)$$

The table for variance analysis is as follows :

Factor	Sum of squares	Degrees of freedom	Variance	Variance in terms of components
w (within packed units)	S_{w}	10	$V_{\mathrm{w}} = \frac{S_{\mathrm{w}}}{10}$	$\sigma^2_{\mathrm{DM}} + \frac{4}{3}\sigma^2_{\mathrm{w}}$
DM (sample preparation and analysis)	S_{DM}	10	$V_{\mathrm{DM}} = \frac{S_{\mathrm{DM}}}{10}$	σ^2_{DM}

The estimate of the combined variance of division and measurement is, then, as follows :

$$\hat{\sigma}^2_{\mathrm{DM}} = V_{\mathrm{DM}} \qquad \ldots (25)$$

And the estimation of the quality variation value within packed units is as follows :

$$\hat{\sigma}^2_{\mathrm{w}} = \frac{3}{4}(V_{\mathrm{w}} - V_{\mathrm{DM}}) \qquad \ldots (26)$$

A.2 The calculation of the quality variation value within packed units ($\hat{\sigma}^2_{\mathrm{w}}$) may be carried out by correction of the calculation of the first term on the right hand side of formula (14) (see 5.3.5).

For this purpose, calculate the ranges for binary subsamples (A, B) :

$$\overline{R}'_{AB} = \frac{1}{10}\sum_{i=1}^{10}|A_{i1} - B_i| \qquad \ldots (27)$$

$$\overline{R}''_{AB} = \frac{1}{10}\sum_{i=1}^{10}|A_{i2} - B_i| \qquad \ldots (28)$$