SLOVENSKI STANDARD
SIST ISO 3713:2000

01-april-2000

Ferozlitine - Vzorļenje in priprava vzorcev - Splońna pravila

Ferroalloys -- Sampling and preparation of samples -- General rules

Ferro-alliages -- Échantillonnage et préparation des échantillons -- Règles générales

Ta slovenski standard je istoveten z: **ISO 3713:1987**

ICS:

77.100 Železove zlitine Ferroalloys

SIST ISO 3713:2000 **en**

INTERNATIONAL STANDARD

ISO 3713

First edition
1987-12-15

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Ferroalloys — Sampling and preparation of samples — General rules

Ferro-alliages — Échantillonnage et préparation des échantillons — Règles générales

Reference number
ISO 3713 : 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 3713 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.

Printed in Switzerland

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Ferroalloys — Sampling and preparation of samples — General rules

1 Scope and field of application

This International Standard gives general rules for sampling and sample preparation of all types of ferroalloys.

The methods given in this International Standard are applicable to increment sampling of consignments supplied both in bulk and in a packed form during loading or unloading, and to sampling of consignments in stationary stockpiles.

This International Standard specifies the methods of both manual and mechanical sampling.

It should be read in conjunction with the relevant International Standards for individual types of ferroalloys.

2 References

ISO 4551, *Ferroalloys — Sampling and sieve analysis.*

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

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

3 Definitions

For the purpose of this International Standard, the following definitions apply.

3.1 lot: A quantity of a ferroalloy produced and processed under conditions which are presumed uniform.

3.2 consignment: A quantity of a ferroalloy delivered at one time. A consignment may consist of one or more lots or parts of a lot.

3.3 packed unit: A part of a consignment definitely separated and placed into a box, a barrel, a container, etc.

3.4 increment: A quantity of a ferroalloy obtained by a sampling device at one time from a consignment supplied in bulk or in a packed form; also a quantity taken by the increment division method.

3.5 sub-sample: A quantity of a ferroalloy consisting of several increments taken from a part of a consignment; also a composite of several increments after having been individually crushed and/or divided as necessary.

3.6 gross sample: The quantity of a ferroalloy consisting of all the increments taken from a consignment; also the composite of all the increments or all the sub-samples having been individually crushed and/or divided as necessary.

3.7 divided sample: A sample obtained by the method of division.

3.8 test sample: Any sample for the determination of the size distribution or chemical composition which is prepared from each increment, from each sub-sample or from a gross sample in accordance with the specified method for the type of sample.

3.9 representative quality characteristic: The content of an element or elements, or the size composition, the quality variation of which determines the parameters of sampling of a given ferroalloy and which is liable to payment in accordance with technical requirements for a given ferroalloy.

3.10 division: A process of decreasing the mass of a sample according to the prescribed rules for the purposes of obtaining the required mass of a test sample.

3.11 precision: The greatest permissible error of the estimation of the average value of a representative quality characteristic expressed as twice the standard deviation (as a percentage) of this characteristic.

3.12 random sampling: A method of increment sampling in which each part of a ferroalloy sampled has equal probability of being taken.

3.13 systematic sampling: A practical method of random sampling in which increments are taken at specified regular intervals in terms of mass, time or space; the first increment is taken at a randomly selected first interval.

3.14 two-stage sampling: A practical method of random sampling by two stages. Selection of primary units of sampling (for example, packed units or parts of a consignment) is carried out at the first stage.

3.15 nominal top size: The upper level of the particle ranges specified in International Standards for technical requirements and conditions for delivery of individual types of ferroalloys.

3.16 top size: Particle size expressed by the aperture size of a sieve on which not more than 1 % of a sample is retained.

4 General

4.1 Heterogeneity of consignment

4.1.1 A consignment of a ferroalloy as the subject of sampling is characterized by heterogeneity expressed by the standard deviation σ_i of a representative quality characteristic between increments.

4.1.2 Heterogeneity (quality variation) of a consignment shall be determined experimentally for each type of ferroalloy, type of sampling and method of constituting a consignment in accordance with ISO 7087.

4.1.3 The method of constituting a consignment is specified in the relevant International Standards on technical requirements for ferroalloy delivery.

4.2 Overall precision

4.2.1 The overall precision ($\beta_{SDM} = 2\sigma_{SDM}$) of the estimate of the representative quality characteristic of a consignment consists of the aggregate of the sampling precision ($\beta_S = 2\sigma_S$), the sample preparation precision ($\beta_D = 2\sigma_D$) and the measurement precision ($\beta_M = 2\sigma_M$).

4.2.2 The true value of the representative quality characteristic of a consignment at the confidence level of 95 % shall be within the interval ($\bar{x} \pm \beta_{SDM}$), where $\bar{x}$ is the arithmetic mean of paired measurements.

4.2.3 The representative quality characteristic against which the precision is established is specified in the International Standards on sampling individual types or groups of ferroalloys.

4.2.4 If a sample is taken from a consignment by random sampling and if this sample is prepared and analysed by standard methods, the overall precision of the determination of the representative quality characteristic of a consignment, β_{SDM}, may be expressed by one of the following methods.

When determining a chemical composition:

a) If a gross sample is constituted and a duplicate analysis is made,

$$\beta_{SDM} = 2\sqrt{\frac{\sigma_i^2}{n} + \sigma_D^2 + \frac{\sigma_M^2}{2}} \qquad \ldots (1)$$

where

σ_i is the measure of the heterogeneity or the standard deviation of the quality characteristic between increments taken from a consignment at random;

n is the minimum number of increments taken from a consignment;

σ_D is the standard deviation of the sample preparation;

σ_M is the standard deviation of the analysis method of the quality characteristic.

b) If K sub-samples, consisting on average of n/K increments, are constituted and a single analysis is made on each sub-sample,

$$\beta_{SDM} = \frac{1}{\sqrt{K}} 2\sqrt{\frac{\sigma_i^2}{n/K} + \sigma_D^2 + \sigma_M^2} \qquad \ldots (2)$$

where K is the number of sub-samples taken from a consignment.

c) If each increment is subjected to a single analysis,

$$\beta_{SDM} = \frac{1}{\sqrt{n}} 2\sqrt{\sigma_i^2 + \sigma_D^2 + \sigma_M^2} \qquad \ldots (3)$$

When determining a size composition, if a gross sample is constituted, divided and subjected to a single sieving,

$$\beta_{SDM} = 2\sqrt{\frac{\sigma_i^2}{n} + \sigma_{DM}^2} \qquad \ldots (4)$$

where σ_{DM} is the combined standard deviation of division and sieving.

4.2.5 The value of the overall precision of the determination of the average quality of a consignment is specified in the relevant International Standards on sampling of individual types or groups of ferroalloys. It satisfies the requirements of ferroalloy consumers and, at the same time, ensures sampling acceptable from an economic point of view.

4.3 Sampling and sample preparation

4.3.1 Sampling and sample preparation shall be carried out in accordance with the International Standards on sampling individual types or groups of ferroalloys.

Other methods of sampling and sample preparation may be used by agreement between the interested parties, provided that their precision is in accordance with that given in the respective International Standard.

The evaluation of the precision of the sampling method shall be made experimentally in accordance with ISO 7087.

4.3.2 It is necessary to ensure that the methods of sampling do not introduce any bias. The evaluation of the bias shall be carried out experimentally in accordance with ISO 7347.

4.3.3 Sampling a ferroalloy consignment shall be carried out in the following order (see figure 1):

a) identify a consignment or a part of a consignment to be sampled;

b) determine the nominal top size in accordance with the order for a definite type of a ferroalloy;

c) determine the mass of an increment;

d) determine the number of increments;

e) determine the place and method of increment sampling;

f) constitute a gross sample or sub-samples;

g) crush and divide the gross sample, sub-samples or increments to a test sample in a definite order when determining chemical composition.

5 Sampling

5.1 Types of sampling

5.1.1 Depending on the condition of a consignment, sampling may be of the following types:

a) sampling from a consignment supplied in bulk;

b) sampling from a consignment supplied in a packed form.

5.1.2 Depending on the methods used, sampling shall be divided into the following types:

a) mechanical sampling;

b) manual sampling.

5.1.3 Increment sampling shall be carried out during loading-unloading or any other displacement of a consignment.

For consignments, parts of a consignment or packed units of small masses, increment sampling may be carried out from a ferroalloy in a stationary state. In such a case, it is necessary to ensure beforehand that the whole of the ferroalloy is available for taking increments.

5.1.4 When sampling non-crushable ferroalloys, increments shall be taken from lumps by drilling, milling or shaping. The detailed descriptions of these methods are given in the International Standards on sampling these types of ferroalloys.

5.2 Mass of an increment

5.2.1 The minimum mass of an increment is specified in the International Standards on sampling individual types or groups of ferroalloys.

5.2.2 In manual sampling, the minimum mass of an increment shall be established on the basis of the nominal top size in a consignment to avoid any bias.

5.2.3 When sampling a stream of a ferroalloy with a mechanical sampler, the mass of an increment may be calculated from the equation

$$m_i = \frac{q_m\, b}{3{,}6\, v} \qquad \ldots \quad (5)$$

where

m_i is the mass, in kilograms, of an increment;

q_m is the average flow rate, in kilograms per second;

b is the width, in metres, of a sample cutter;

v is the rate, in metres per second, of the sample cutter movement.

5.2.4 The masses of the increments taken from a consignment shall be almost equal.

NOTE — The words "almost equal" mean that the coefficient of the variation of the increment masses taken from one consignment shall be under 20 %.

5.2.5 When it is difficult to take increments of equal masses, for instance, from a moving stream, a gross sample or sub-samples shall be constituted from divided samples of almost equal masses.

5.2.6 When sampling by time, the masses of increments taken from a consignment shall be proportional to the flow rate.

5.2.7 When each increment is individually analysed, the masses of increments may be unequal.

5.3 Number of increments

5.3.1 The minimum number of increments to be taken from a consignment shall depend on the planned precision of sampling β_S and the consignment heterogeneity σ_i.

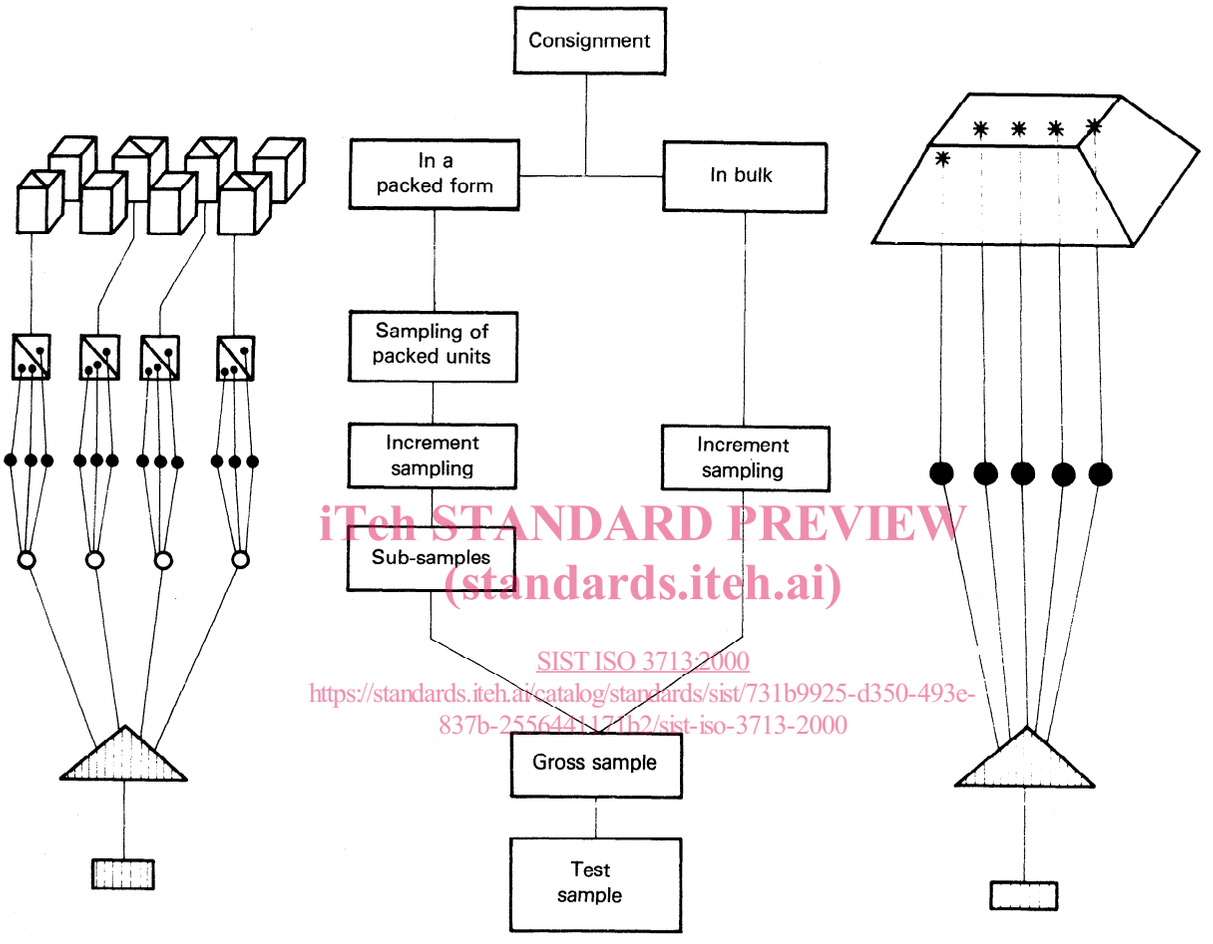

Figure 1 — Succession of sampling

5.3.2 The precision of sampling β_S shall be determined according to the method of constituting a consignment and its mass, and is also established in the International Standards on sampling of individual types or groups of ferroalloys.

5.3.3 For a consignment supplied in bulk, the minimum number of increments shall be established using the equation

$$n = \left(\frac{2\sigma_i}{\beta_S}\right)^2 \frac{N - n}{N - 1} \quad \ldots \quad (6)$$

where N is the number of increments constituting a consignment.

NOTES

1 Equation (6) is derived from the equation

$$\left(\frac{\beta_S}{2}\right)^2 = \frac{\sigma_i^2}{n} \frac{N - n}{N - 1} \quad \ldots \quad (7)$$

2 If $n/N \leqslant 0{,}1$, then $\frac{N - n}{N - 1}$ will be assumed equal to 1.

5.3.4 For a consignment supplied in a packed form, the minimum number of packed units to be selected at the first stage of two-stage sampling shall be calculated using the equation

$$M_p = \frac{M_t \sigma_b^2 + (M_t - 1) \times \sigma_b \times \sigma_w}{(M_t - 1) \times (\beta_S/2)^2 + \sigma_b^2} \quad \ldots \quad (8)$$

where

M_p is the number of packed units to be taken from a consignment at the first stage of sampling (primary sampling units);

M_t is the number of packed units in a consignment;

σ_b is the standard deviation between the packed units of a consignment;

σ_w is the standard deviation between the increments within a packed unit.

When the values of σ_w and σ_b are known, the minimum number of increments to be taken from each selected unit shall be calculated using the equation

$$n_s = \frac{\sigma_w}{\sigma_b} \quad \ldots \quad (9)$$

where n_s is the number of increments to be taken from a packed unit selected (secondary sampling units).

NOTES

1 Equation (8) is derived from the equation

$$\left(\frac{\beta_S}{2}\right)^2 = \frac{M_t - M_p}{M_t - 1} \times \frac{\sigma_b^2}{M_p} + \frac{\sigma_w^2}{M_p n_s} \quad \ldots \quad (8a)$$

2 If $\frac{M_p}{M_t} \leqslant 0{,}1$, then $\frac{M_t - M_p}{M_t - 1}$ will be assumed equal to 1.

$$\left(\frac{\beta_S}{2}\right)^2 = \frac{\sigma_b^2}{M_p} + \frac{\sigma_w^2}{M_p n_s} \quad \ldots \quad (8b)$$

3 If $M_t = M_p$, calculate β_S^2 as follows:

$$\left(\frac{\beta_S}{2}\right)^2 = \frac{\sigma_w^2}{M_p n_s} = \frac{\sigma_w^2}{n} \quad \ldots \quad (8c)$$

5.3.5 In International Standards on sampling of individual types or groups of ferroalloys, the minimum number of packed units and/or increments is given in a tabular form or graphs obtained using equations (6) or (8).

5.4 Mechanical method of increment sampling

5.4.1 When loading-unloading wagons, ships, bunkers for storage, etc., with continuous transport means, sampling shall be carried out from a falling stream with mechanical samplers at equal intervals by mass or time.

5.4.2 The number of cuts made when taking a gross sample with mechanical samplers shall be not less than the number of the planned increments.

5.4.3 Intervals between taking increments shall be equal for the whole consignment and calculated by time or mass of a ferroalloy in accordance with the mass of a consignment and the number of increments.

5.4.4 Intervals by mass shall be calculated using the equation

$$\Delta m_i \leqslant \frac{m_C}{n} \quad \ldots \quad (10)$$

where

Δm_i is an interval by mass, in kilograms, between increment sampling;

m_C is the mass, in kilograms, of a consignment.

NOTE — If systematic sampling of the whole consignment gives a bias, it is necessary to divide the consignment into a number of parts equal to the given number of increments and take increments randomly from each part.

5.4.5 Time-intervals shall be calculated using the equation

$$\Delta t = \frac{60\, m_C}{q_m n} \quad \ldots \quad (11)$$

where

Δt is the time-interval, in minutes, between taking increments;

q_m is as defined in 5.2.3.

NOTE — Intervals between taking increments should be calculated by time only in the case when the stream of a ferroalloy is constant in time.