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Ferroalloys — Sampling and preparation of samples — General rules

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

Reference number
ISO 3713 : 1987 (E)

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 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.

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Annexes

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

$$m_\text{i} = \frac{q_m\, b}{3{,}6\, v} \qquad \ldots (5)$$

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.

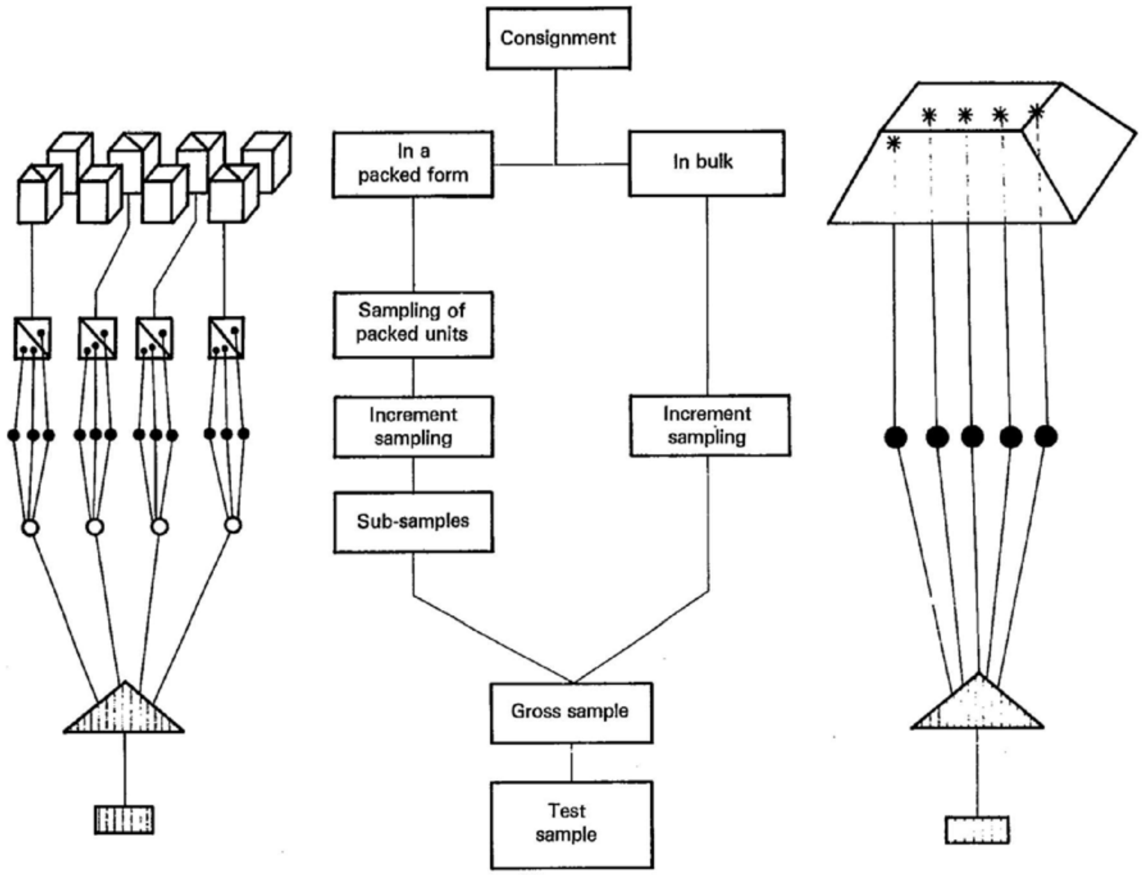

Figure 1 — Succession of sampling

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

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

where N is the number of increments constituting a consignment.

NOTES

1 Equation (6) is derived from the equation

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

2 If $n/N < 0,1$, 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

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

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

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

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

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

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

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

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

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

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

$$\Delta m_i < \frac{m_C}{n} \qquad \ldots (10)$$

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

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

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.

5.4.6 The first increment within the first interval shall be taken randomly.

5.4.7 If the planned number of increments has been taken but displacing a consignment has not finished, sampling shall be continued until the displacement of a consignment is over.

5.4.8 When taking samples from a belt of a stopped conveyor, an increment shall be taken from an established point in the direction of the stream movement. The sampler used shall take the whole of the width and thickness of the stream over a defined length.

NOTE — This length should be sufficient to take an increment of the minimum mass and be not less than three times the nominal top size of a ferroalloy.

5.5 Manual method of increment sampling

5.5.1 In manual sampling, an increment shall be taken in a single motion at one time with a special scoop (see 7.2.1) suitable for taking samples of constant masses.

When it is difficult to take an increment in a single motion, it shall be taken in several motions from one place selected at random. To take increments of constant and almost equal masses, it is necessary to use a container for increment sampling (see 7.2.2).

5.5.2 When sampling a stationary consignment of less than 10 mm in particle size, an increment may be taken with a probe. In this case, the mass of the ferroalloy taken by a probe at a single motion shall be not less than the specified minimum mass of an increment.

5.5.3 When sampling for the determination of the chemical composition of a consignment consisting of lumps of more than 100 mm in particle size, an increment shall be taken by splitting off pieces having, if possible, the bottom and top surfaces of an ingot; the pieces shall be taken from not less than four large lumps selected at random at the point of sampling. An increment of the planned mass shall be constituted of the split off pieces. It is recommended that a special container for increment sampling (see 7.2.2) be used. When using this method, it is necessary to ensure that it does not introduce any bias.

5.5.4 When sampling for the determination of the chemical composition of a consignment consisting of particles of different sizes, the size distribution of an increment shall correspond to that of the consignment which is known from the results of a sieve analysis (see ISO 4551) or from past experience.

5.5.5 In manual sampling of a ferroalloy in a stationary state the points of sampling shall be disposed in a definite order on its surface. A crater shall be made at each point of sampling and an increment shall be taken with a scoop along the walls of the crater upwards in a straight line. The ferroalloy taken shall not over-fill the scoop. When using this method, it is necessary to ensure that it does not introduce any bias.

5.5.6 When loading-unloading a ferroalloy with devices of cyclic action, increments shall be taken by hand from a new surface formed or from a ferroalloy poured out of a grab onto a special surface.

5.5.6.1 The number of grabs out of which samples are taken shall be not less than the planned number of increments.

5.5.6.2 Intervals between the grabs out of which samples are taken shall be calculated using the equation

$$r = \frac{m_C}{m_g n} \qquad \ldots \quad (12)$$

where

r is an interval by the number of grabs;

m_g is the mass, in kilograms, of a ferroalloy displaced with one grab during one cycle.

5.6 Sampling a consignment in a packed form

5.6.1 Sampling a packed consignment shall be carried out in two stages (two-stage sampling). At the first stage, the planned number of packed units, M_p, shall be selected. At the second stage, the planned number of increments, n_s, shall be taken from each packed unit selected (see 5.3.5).

5.6.2 Packed units shall be selected by systematic sampling or by random sampling, using tables of random numbers if the packed units are numbered.

5.6.3 If the number of packed units, M_t, in a consignment is less than the planned number of packed units, M_p, increments shall be taken from all the packed units. The number of increments taken from each packed unit shall be determined by dividing the planned number of increments to be taken from a consignment by the number of the packed units in it. The results of dividing shall be rounded up to the nearest whole number.

5.6.4 The methods of sampling increments from packed units shall comply with 5.5. Before increment sampling, it is recommended that the contents of a packed unit be poured out onto a clean surface.

5.6.5 If the mass of a ferroalloy in a packed unit is insufficient for taking the planned number of increments, the whole mass of the packed unit shall be taken as a sample.

5.7 Methods of combining increments

5.7.1 Increments taken from one consignment shall be combined into one gross sample.

5.7.2 When it is required to obtain a better precision of the determination of the quality characteristics of a consignment than those specified in the relevant International Standards, each increment or sub-sample shall be individually analysed without combining them.

5.7.3 If a consignment is divided into several parts and several increments are taken from each part,

a) the increments of each part shall be combined into sub-samples in proportion to the mass of each part; then each of the sub-samples shall be prepared and analysed individually;

b) the increments of each part shall be combined into sub-samples and, at an appropriate stage of preparation, the latter shall be combined into a gross sample for an analysis, provided that the mass of each part or the number of increments comprising each sub-sample is almost equal.

5.7.4 If a consignment consists of several packed units,

a) the increments of each packed unit shall be combined into sub-samples in proportion to the mass of each packed unit; each sub-sample shall be prepared and analysed individually;

b) the increments of each packed unit shall be combined into sub-samples; at an appropriate stage of preparation, the latter shall be combined into a gross sample for an analysis, provided that the mass of each packed unit or the number of increments comprising each sub-sample is almost equal.

6 Sample preparation

6.1 Succession of sample preparation

6.1.1 Preparation of a test sample for the determination of the chemical composition shall be carried out from an increment, a sub-sample or a gross sample.

6.1.2 The process of sample preparation consists of consecutive operations of crushing, mixing and division of a sample.

6.1.3 The particle size and mass of a test sample shall be as specified in the International Standards on sampling of individual types or groups of ferroalloys.

6.1.4 The succession and operations of sample preparation of non-crushable ferroalloys shall be as specified in the International Standards on sampling of these types of ferroalloys.

6.2 Precision of sample preparation

6.2.1 All the operations of sample preparation shall ensure the planned precision of sample preparation β_D.

6.2.2 The value of the precision of sample preparation shall be as specified in the International Standards on sampling of individual types or groups of ferroalloys.

6.3 Crushing

6.3.1 The equipment for crushing and grinding of a sample shall be selected according to 7.2.4.

6.3.2 Before use, the inner parts of crushing devices shall be thoroughly cleaned. A quantity of the same ferroalloy taken from the consignment to be sampled shall be passed through the crushing device once or several times.

6.3.3 In the course of crushing, it is necessary to see that the sample does not change its quality, for example due to heating during abrasion.

6.3.4 A check sieving and additional grinding of the particles retained on the sieve shall be carried out after each operation of crushing, so that the entire sample passes through the given sieve.

6.3.5 In the course of crushing, losses of the material of a sample (for example, due to spilling, ejection of dust, etc.) shall be avoided.

6.4 Mixing

6.4.1 The sample shall be thoroughly mixed before each operation of division in order to ensure the precision of division and to prevent the introduction of any bias.

6.4.2 Mixing may be carried out either with a mechanical mixer or by hand.

6.4.3 A mechanical mixer shall be selected on the basis of the mass of the sample and the size of its particles. Before use, the inner parts of a mixer shall be thoroughly cleaned.

6.4.4 Mixing by hand may be carried out by one of the following methods:

a) using a shovel or a scoop;

b) the "ring and cone" method;

c) the rolling method.

6.4.5 Mixing with a shovel (or a scoop) consists in transferring the material from one heap to another several times. The material shall be taken at random from different places in the first heap.

6.4.6 When using the "ring and cone" method, a sample shall be placed in the form of a ring on a flat surface; then the material shall be taken evenly by a scoop or a shovel along the external or internal circumference and poured into the centre of the ring in the form of a cone. Each portion of the material shall be placed on the top of the cone. To maintain the axis of the cone, a vertical rod or a guide funnel shall be used. After the material has been completely transferred, the cone shall be destroyed using the rod to form a new ring. The operation of converting a cone into a ring may be the first step in the process.

6.4.7 The rolling method shall be used only for ferroalloys of small particle size. The sample shall be poured on to a piece of material which cannot contaminate it. Then each corner of the material shall be raised in turn so that the sample rolls from one corner to the opposite one and back again to the original position. The cycle of operations shall be repeated not less than 25 times.

6.5 Division

6.5.1 Only samples of less than 10 mm in particle size shall be divided.

6.5.2 Before division, a sample shall be thoroughly mixed not less than three times using one of the methods specified in 6.4.

6.5.3 Devices for sample division shall be thoroughly cleaned. A quantity of the same ferroalloy taken from the consignment to be sampled shall be passed through them one or more times.

6.5.4 The minimum permissible mass, $m_{S,\,min}$, of the sample after division shall be determined on the basis of the top size of the sample using the equation:

$$m_{S,\,min} = \kappa\, d^{\alpha} \qquad \ldots (13)$$

where

d is the top size, in millimetres;

α, κ are dimensionless correction factors.

NOTE — α and κ are values characterizing physical properties of ferroalloys and are determined experimentally for each ferroalloy.

6.5.5 Tables or graphs of sampling division calculated using equation (13) are specified in the International Standards on sampling of individual types or groups of ferroalloys.

6.5.6 A sample shall be divided by the following methods (separately or in conjunction):

a) the increment division method;

b) the coning and quartering method;

c) division with a riffle divider;

d) division with a mechanical divider.

6.5.7 The method of increment division can give the planned precision in spite of a high coefficient of division. It is better, however, to avoid the application of this method if a ferroalloy is inclined to segregation. The procedure of division is as follows:

a) Spread the crushed and thoroughly mixed sample in a uniform layer on a smooth surface in the form of a rectangle. The thickness of the layer shall be approximately equal to three times the top size of the sample.

b) Divide the rectangle into 20 or more parts when dividing a gross sample, into 10 or more parts when dividing a subsample, and into 4 or more parts when dividing an increment.

c) Take a full scoop of the ferroalloy from each part (the place of sampling being selected at random in each part) and form a divided sample. It is recommended that a scoop in accordance with table 1 be used. Insert the scoop to the bottom of the sample layer. To prevent any bias insert a bumper plate vertically in front of the scoop to the bottom of the sample layer.

6.5.8 Division by the coning and quartering method secures division of a sample into two parts. This method is suitable for use after mixing by the "ring and cone" method (see 6.4.6); the procedure is as follows:

a) With the rod, using circular motions from the centre to the edge, flatten the cone formed after the third mixing. The flattened cone shall have the form of a circle and be of uniform thickness. The centre of the circle shall coincide with that of the original cone.

b) Divide the circle into quarters by two lines which intersect at right angles at its centre. If available, use a cross.

c) Reject two diametrically opposite sectors and combine the remainder, crush it once more and, if necessary, repeat the operation beginning from the threefold mixing.

To prevent any bias, alternately opposing parts shall be rejected in each consequent operation.

6.5.9 Division with a riffle divider secures division of the sample into two parts. The size of the riffle divider shall be selected, on the basis of the top size of the sample to be divided, in accordance with 7.2.5. The procedure of division with a riffle divider is as follows:

a) Mix the sample and place it in a special container.

b) Drop the sample uniformly from the container while it is being lightly shaken, so that the ferroalloy falls uniformly along the centre line of the riffles.

c) Select at random one of the two parts obtained.

NOTE — Care shall be taken not to leave any material in the slots of the riffles, and shaking of the container shall be carried out in such a manner as to prevent any bias.

6.5.10 A mechanical divider may be used, provided that it does not introduce any bias and ensures the planned precision.

6.6 Sample preparation for the determination of size composition

In accordance with ISO 4551.

7 Main devices and tools for sampling and sample preparation

7.1 For mechanical sampling, only mechanical devices meeting the following requirements shall be used:

a) A sampling device shall cross the entire stream of ferro-alloy and take a complete cross-section.

b) The capacity of the sampling device shall be sufficient to take one complete increment at one cut.

c) The construction of a sampling device shall be convenient for cleaning and checking.

7.2 The following implements shall be used for manual sampling:

a) scoops;

b) containers for sampling;

c) probes.

7.2.1 A scoop for increment sampling shall be of dimensions corresponding to the increment volumes and shall be sufficiently durable (see figure 2 and table 1). Examples for choosing scoops are given in annex B.

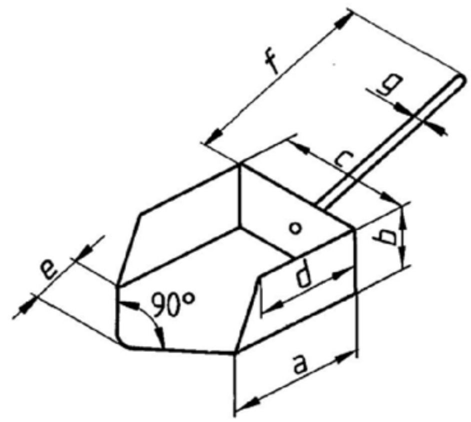

Figure 2 — A scoop for increment sampling

7.2.2 A container for increment sampling shall be selected on the basis of the volume of a sample (see figure 3 and table 2).

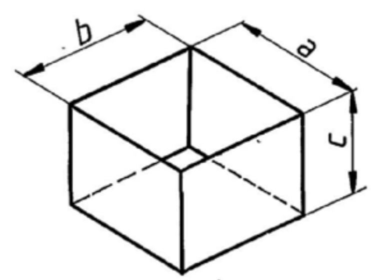

Figure 3 — A container for increment sampling

Table 1 — Recommended dimensions of scoops for increment sampling

Number of scoop	Approximate volume cm³	Dimensions mm						Material thickness mm	a/c	b/c
		a	b	c	d	e*	f g			
1	15	30	15	30	25	12	Select according to circumstances	0,5	1,0	0,50
3	40	40	25	40	30	15		0,5	1,0	0,62
5	75	50	30	50	40	20		1	1,0	0,60
10	125	60	35	60	50	25		1	1,0	0,58
15	200	70	40	70	60	30		2	1,0	0,57
20	300	80	45	80	70	35		2	1,0	0,56
30	400	90	50	90	80	40		2	1,0	0,56
40	790	110	65	110	95	50		2	1,0	0,59
50	1 700	150	75	150	130	65		2	1,0	0,50
75	4 000	200	100	200	170	80		2	1,0	0,50
100	7 000	250	110	250	220	100		2	1,0	0,44

* If a scoop is also used for sample division, $e = 0$, i.e. the front of the scoop is cut off.

Table 2 — Recommended dimensions of containers for increment sampling

Approximate volume cm³	Dimensions mm		
	a	b	c
75	50	50	30
125	60	60	35
200	70	70	40
300	80	80	45
400	90	90	50
790	110	110	65
1 700	150	150	75
4 000	200	200	100
7 000	250	250	120

7.2.3 The diameter of the opening of a probe for increment sampling of a consignment of a ferroalloy of less than 10 mm in particle size shall be not less than three times the nominal top size. The construction of the probe shall ensure sampling from the whole layer of a ferroalloy. Examples of probe constructions are given in figure 4.

7.2.4 The devices for crushing of samples shall be selected in accordance with the planned particle size and the hardness of the ferroalloy.

7.2.5 A divider for sample division shall be selected on the basis of the particle size in accordance with table 3. An example of a riffle divider is shown in annex A.

Table 3 — Types of riffle divider

Nominal top size in sample, d mm	Number of divider	Width of riffle mm
$5 < d \leqslant 10$	20	20
$2,4 < d \leqslant 5$	10	10
$d \leqslant 2,4$	6	6

7.2.6 Devices and tools used for sampling and sample preparation shall ensure the planned precision at all the stages of the procedure.

7.2.7 All the devices and tools used shall be cleaned, checked and adjusted before sampling.

8 Test sample

8.1 Prevention of contamination

8.1.1 In the course of sampling and sample preparation, no loss of any material from the sample or contamination of the sample with foreign matter or with material from the devices and tools shall take place.

8.1.2 Vessels for transportation and storage of samples at all the stages of sampling shall be clean, intact and have hermetic covers.

8.2 Samples for determination of chemical composition of a consignment

8.2.1 Four samples intended for a producer, a customer, an arbitration organization and for reserve shall be prepared from each consignment. The reserved sample shall be kept for not less than 6 months.

8.2.2 Each sample shall be placed in a separate hermetically sealed container.

8.2.3 A sample shall be supplied with two identical labels; one shall be stuck on the vessel and the other, in a protective wrapping preserving the sample from contamination, shall be placed inside it.

8.2.4 The following information shall be written on the label:

a) name of the producer;

b) designation and grade of the ferroalloy;

c) mass of the consignment;

d) date and place of sampling;

e) signature of the sampler;

f) any additional information.

8.3 Samples for determination of size composition

In accordance with ISO 4551.

9 Bibliography

ISO 4552, *Ferroalloys — Sampling and sample preparation for chemical analysis*

— *Part 1: Ferrochromium, ferrosilicochromium, ferrosilicon, ferrosilicomanganese, ferromanganese.*

— *Part 2: Ferrotitanium, ferromolybdenum, ferrotungsten, ferroniobium, ferrovanadium.*

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

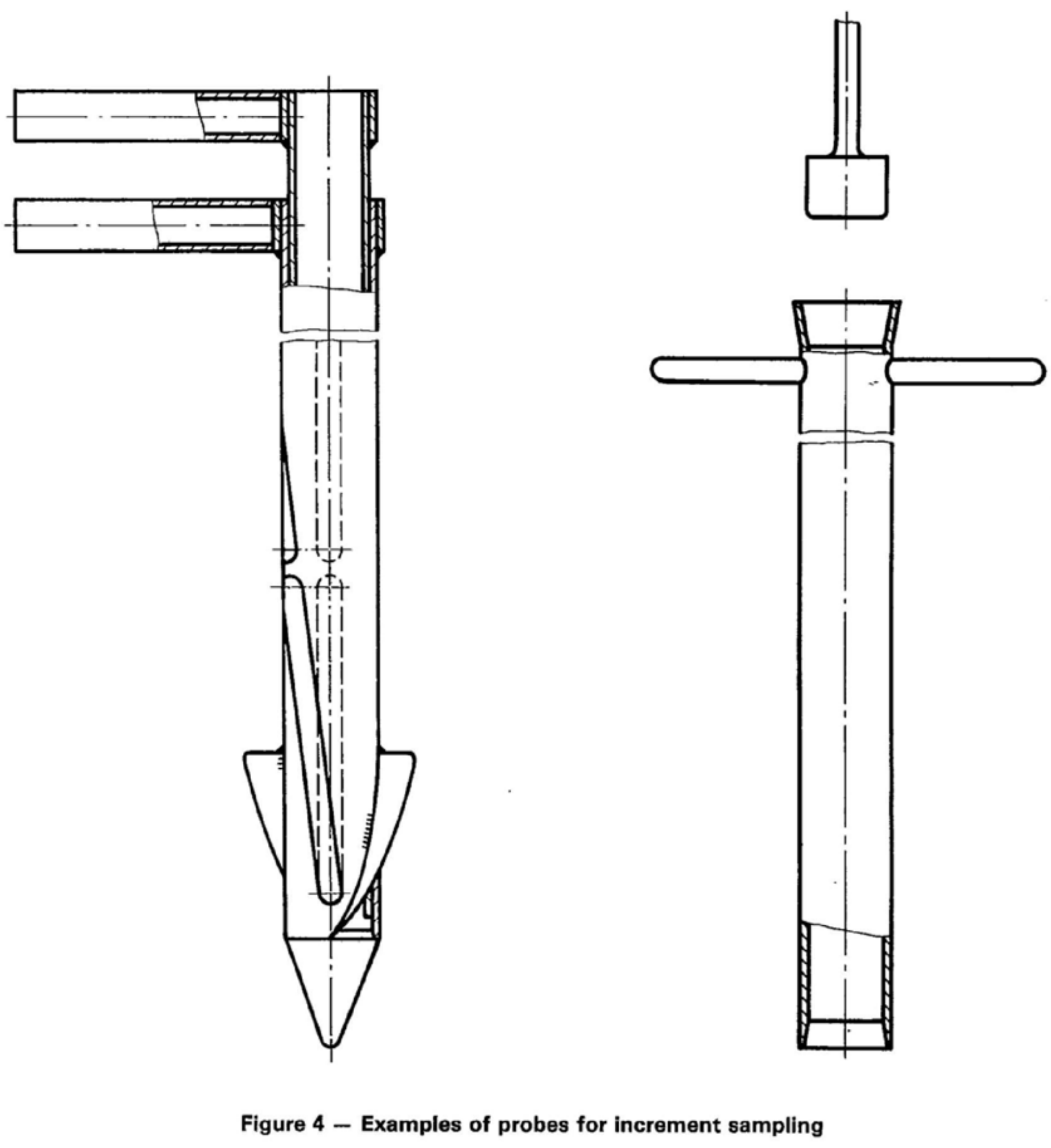

Figure 4 — Examples of probes for increment sampling

Annex A

Riffle divider

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

Table 4 — Dimensions of riffle dividers, mm
(see figure 5)

Dimensions	Type of divider		
	20	10	6
	Number of riffles		
	16	16	16
A	20 ± 1	10 ± 1	6 ± 0,5
B	346	171	112
C	105	55	40
D	210	110	80
E	135	75	60
F	30	20	20
G	210	110	80
H	85	45	30
J	360	184	120
K	140	65	55
M	140	65	55
N	210	110	80
P	105	55	40
Q	35	20	15
R	210	110	80
S	300	150	100
T	200	120	80
U	135	70	45
V	105	50	35
θ	60°		

A.1 *A* is the dimension specified; the others are given as examples.

A.2 The angle between the riffles shall be not more than 60°.

A.3 The number of riffles shall be even and not less than that stated in table 4.

A.4 The sample receiver shall be mounted against the riffles so as to avoid spilling of a sample.

A.5 The internal surface of a divider shall be smooth and free from rust.

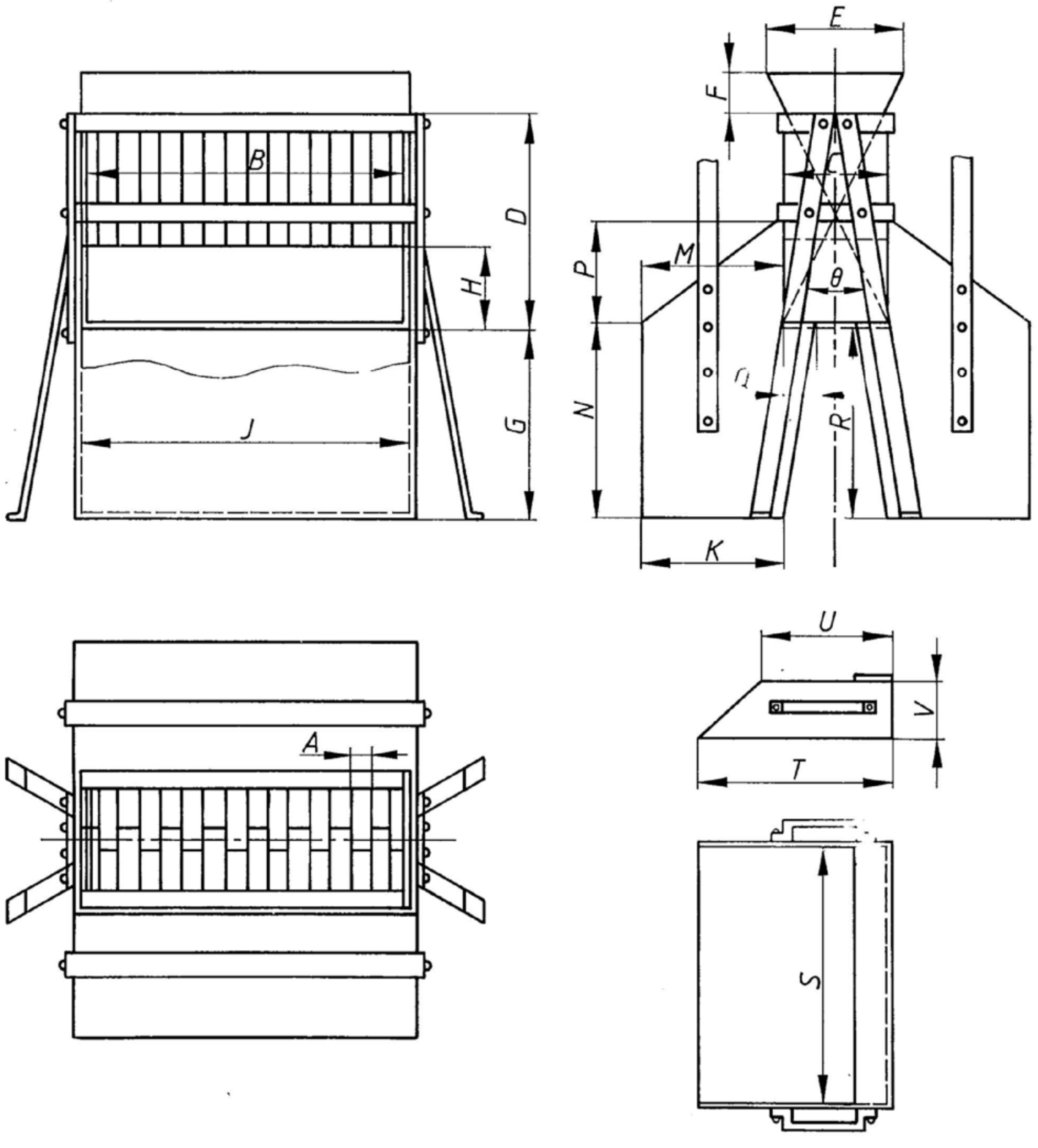

Figure 5 — Riffle divider

Annex B

Example of procedure for selecting a scoop for increment sampling

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

B.1 Determine experimentally, or based on previous experience or on the data provided, the bulk density, ϱ, of a ferroalloy, in kilograms per cubic metre. This will depend on the particle size and ferroalloy grade.

B.2 Calculate the volume, V_i, of an increment, in cubic metres, using the following equation:

$$V_i = \frac{m_i}{\varrho} 10^6$$

where

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

ϱ is the bulk density, in kilograms per cubic metre, of the ferroalloy.

B.3 Using table 1, select a scoop the volume of which is nearest to the volume obtained according to clause B.2.

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Descriptors: ferroalloys, sampling, specimen preparation.

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