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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Ferroalloys — Sampling and sample preparation for chemical analysis —

Part 1:
Ferrochromium, ferrosilicochromium, ferrosilicon, ferrosilicomanganese, ferromanganese

Ferro-alliages — Échantillonnage et préparation des échantillons pour analyse chimique —

Partie 1: Ferro-chrome, ferro-silico-chrome, ferro-silicium, ferro-silico-manganèse, ferro-manganèse

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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 4552-1 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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Contents

Ferroalloys — Sampling and sample preparation for chemical analysis —

Part 1 : Ferrochromium, ferrosilicochromium, ferrosilicon, ferrosilicomanganese, ferromanganese

1 Scope and field of application

This part of ISO 4552 specifies the methods for sampling and sample preparation for the determination of the chemical composition of a consignment of ferrochromium, ferrosilicochromium, ferrosilicon, ferrosilicomanganese or ferromanganese.

NOTE — For purposes of sampling, ferrochromium is subdivided into non-crushable and crushable alloys. Low-carbon and certain medium-carbon ferrochromiums are non-crushable alloys; medium-carbon and high-carbon ferrochromiums are crushable alloys.

Part 2 of ISO 4552 specifies the methods for use with ferrotitanium, ferromolybdenum, ferrotungsten, ferroniobium and ferrovanadium.

2 Reference

ISO 3713, *Ferroalloys — Sampling and sample preparation — General rules.*

3 General requirements

3.1 Definitions, general requirements for sampling and sample preparation, tools and equipment

See ISO 3713.

3.2 Quality characteristics for precision requirements

The overall precision of the determination of the chemical composition of a consignment β_{SDM}, precision of sampling β_S, precision of sample preparation β_D and precision of the method of analysis β_M at the 95 % confidence level shall be specified with respect to the quality characteristics shown in table 1.

Table 1 — Quality characteristics for precision requirements

Ferroalloy	Quality characteristic, % (m/m)
Ferrochromium	Chromium content
Ferrosilicochromium	Chromium and silicon contents
Ferrosilicon	Silicon content
Ferrosilicomanganese	Silicon and manganese contents
Ferromanganese	Manganese content

4 Overall precision of the determination of the chemical composition of a consignment

The methods of sampling and sample preparation specified in this part of ISO 4552 allow the determination of the chemical composition of a consignment at the 95 % confidence level with the overall precision shown in table 2, depending on the mass of the consignment sampled.

Table 2 — Overall precision of the determination of the chemical composition of a consignment

Mass of consignment t		Overall precision, β_{SDM}, % (m/m)							
Over	Up to and including	FeCr non-crushable	FeCr crushable	FeSiCr		FeSi	FeSiMn		FeMn
		Cr				Si		Mn	
5 000	10 000	0,68	0,53	0,54	0,56	0,74	0,43	0,44	0,43
2 500	5 000	0,69	0,53	0,56	0,56	0,75	0,43	0,45	0,44
1 000	2 500	0,69	0,54	0,56	0,57	0,75	0,44	0,46	0,44
500	1 000	0,70	0,55	0,57	0,57	0,76	0,44	0,47	0,46
250	500	0,70	0,56	0,59	0,59	0,77	0,45	0,48	0,46
100	250	0,71	0,57	0,60	0,60	0,78	0,46	0,49	0,48
50	100	0,73	0,59	0,61	0,61	0,79	0,48	0,50	0,49
25	50	0,75	0,61	0,64	0,64	0,81	0,50	0,53	0,51
10	25	0,78	0,68	0,76	0,72	0,88	0,55	0,59	0,57
5	10	0,82	0,72	0,78	0,78	0,93	0,58	0,64	0,61
	5	0,87	0,79	0,79	0,86	0,99	0,64	0,71	0,67

5 Sampling

5.1 Mass of increment

5.1.1 The minimum mass of an increment as a function of the nominal top size of a consignment shall correspond to that shown in table 3.

Table 3 — Mass of increment

Nominal top size, mm	Minimum mass of increment, kg				
	FeCr crushable	FeSiCr	FeSi	FeSiMn	FeMn
100 and greater	8,0	5,0	8,0	8,0	8,0
75	—	—	4,0	—	—
50	4,0	3,0	—	4,0	4,0
35	—	—	1,5	—	—
25	1,5	1,0	1,0	1,5	1,5
10 and less	1,0	0,5	0,5	1,0	1,0

5.1.2 For non-crushable ferrochromium, the minimum mass of an increment taken from each lump in the form of drillings of 0,1 to 0,2 mm in thickness shall be not less than 20 g.

5.2 Number of increments and precision of sampling

5.2.1 The minimum number of increments necessary for attaining the planned precision of sampling as a function of the mass of a consignment shall correspond to that given in table 4.

NOTES

1 Other sampling parameters may be adopted by agreement between supplier and consumer. In such a case, the minimum number of increments should be calculated using the formula

$$n = \left(\frac{2\sigma_i}{\beta_s}\right)^2$$

2 If the maximum particle size in a consignment is 100 mm and more, increments should be taken in accordance with ISO 3713.

Table 4 — Minimum number of increments and precision of sampling

Mass of consignment t		Minimum number of increments	Precision of sampling, β_S, % (m/m)						
Over	Up to and including		FeCr crushable	FeSiCr		FeSi	FeSiMn		FeMn
			Cr			Si		Mn	
5 000	10 000	33	0,28	0,28	0,31	0,31	0,23	0,26	0,24
2 500	5 000	30	0,29	0,29	0,33	0,33	0,24	0,27	0,25
1 000	2 500	28	0,30	0,30	0,34	0,34	0,25	0,28	0,26
500	1 000	25	0,32	0,32	0,36	0,36	0,26	0,30	0,28
250	500	23	0,33	0,33	0,38	0,38	0,27	0,31	0,29
100	250	20	0,36	0,36	0,40	0,40	0,29	0,33	0,31
50	100	18	0,38	0,38	0,42	0,42	0,31	0,35	0,33
25	50	15	0,41	0,41	0,46	0,46	0,34	0,39	0,36
10	25	10	0,51	0,51	0,57	0,57	0,41	0,47	0,44
5	10	8	0,57	0,57	0,64	0,64	0,46	0,53	0,49
	5	6	0,65	0,65	0,73	0,73	0,53	0,61	0,57

5.2.2 For non-crushable ferrochromium, the minimum number of lumps necessary for attaining the planned precision of sampling as a function of the mass of a consignment, shall correspond to that given in table 5.

Table 5 — Minimum number of lumps and sampling precision for non-crushable ferrochromium

Mass of consignment t		Minimum number of lumps	Precision of sampling, β_S, % (m/m) Cr
Over	Up to and including		
5 000	10 000	39	0,26
2 500	5 000	36	0,27
1 000	2 500	33	0,28
500	1 000	29	0,30
250	500	27	0,31
100	250	24	0,33
50	100	19	0,37
25	50	16	0,40
10	25	12	0,46
5	10	9	0,53
	5	7	0,60

5.3 Method of increment sampling

5.3.1 When sampling an unpacked consignment, the methods of increment sampling and the intervals between increment sampling shall comply with the requirements of ISO 3713.

5.3.2 When sampling a packed consignment, samples shall be taken in one or two stages.

When sampling in one stage, the number of packed units taken shall correspond to the number of increments or lumps shown in tables 4 and 5. One increment shall be taken from each packed unit sampled.

When sampling in two stages, the number of packed units taken at the first stage and the number of increments taken from one packed unit shall be calculated from equation (8) in ISO 3713. M_p and n_s shall be chosen in such a way that β_S does not exceed the values given in tables 4 and 5 for corresponding masses of consignments.

The methods of selecting the packed units and of selecting increments from these packed units shall comply with the requirements of ISO 3713.

5.3.3 For non-crushable ferrochromium, the methods of lump sampling shall be analogous to those of increment sampling for both packed and unpacked consignments (see 5.3.1).

5.3.4 Increments shall be obtained from lumps of non-crushable ferrochromium by one of the following methods:

a) If the top and bottom surfaces of a lump are easily recognizable, an increment in the form of drillings shall be obtained by milling, shaping or drilling through the whole thickness of the lump from the top surface to the bottom surface as shown in figure 1.

b) If the top and bottom surfaces of a lump are easily recognizable and the thickness of the lump is so large that it is difficult to take an increment by method a), drilling may be carried out perpendicular to the broken surface at four equidistant points. The distance between the outer holes

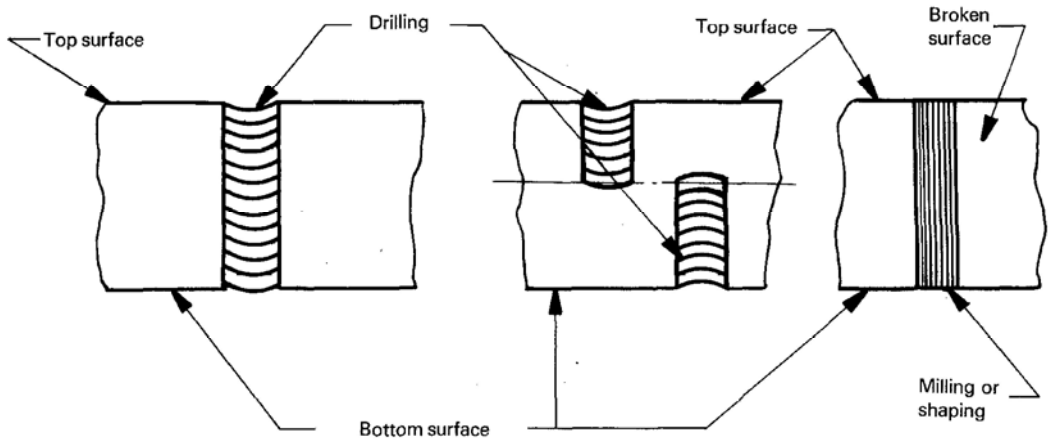

Figure 1 — Procedure of increment sampling of a non-crushable ferrochromium lump

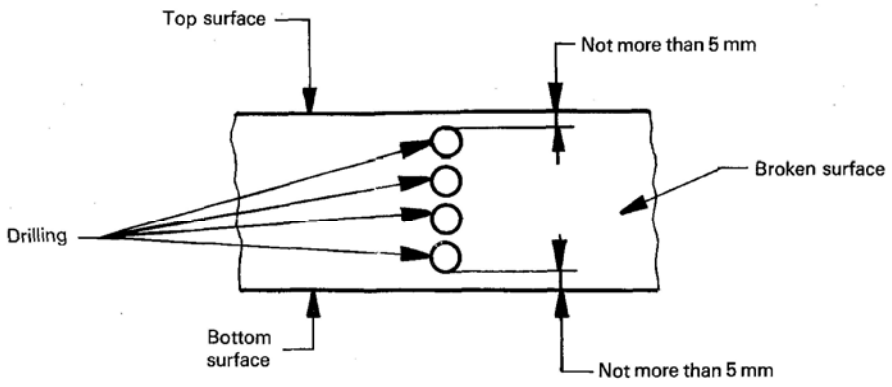

Figure 2 — Procedure of increment sampling of a non-crushable ferrochromium lump by drilling perpendicular to a broken surface

and the top and bottom surfaces of a lump shall be not more than 5 mm as shown in figure 2. Care shall be taken to obtain drillings of uniform masses.

c) If the top and bottom surfaces of a lump are difficult to identify, increment sampling shall be carried out over a whole lump cross-section selected at random by milling or shaping.

The operation of drilling shall be carried out at points selected at random.

5.4 Gross sample

Increments sampled from one consignment shall be combined into a gross sample in accordance with ISO 3713.

6 Sample preparation

6.1 Precision of sample preparation

The methods for sample preparation specified in this part of ISO 4552 give the precision of sample preparation, at the 95 % confidence level, shown in table 6.

Table 6 — Precision of sample preparation

Ferroalloy	Precision of sample preparation, $\pm \beta_D$, % (m/m)		
	Cr	Si	Mn
Ferrochromium			
— crushable	0,4		
— non-crushable	0,6		
Ferrosilicochromium	0,4	0,4	
Ferrosilicon		0,6	
Ferrosilicomanganese		0,3	0,3
Ferromanganese			0,3

6.2 Sample division

6.2.1 The methods of sample division shall comply with the requirements of ISO 3713.

6.2.2 A sample shall be crushed to particles which will completely pass through a sieve with a mesh size of 10 mm × 10 mm. Then a gross sample or a sub-sample shall be divided in accordance with table 7, and increments, if necessary, shall be divided in accordance with the division rules for the increment division method specified in ISO 3713.

An example of gross sample division is given in figure 3.

Table 7 — Rules for division of a gross sample or a sub-sample

Top size of divided sample mm	Minimum mass of divided sample kg
10,0	15,0
5,0	3,0
2,8	1,5
1,0	0,400
0,250	0,200

6.3 Crushing and mixing

The methods for crushing and mixing shall comply with the requirements of ISO 3713.

7 Test sample

7.1 The mass of a test sample for chemical analysis shall be not less than 50 g. The top size of a test sample shall be not greater than 160 µm. The top size of a non crushable ferrochrome sample taken as drillings shall not exceed 1,6 mm.

7.2 The number of test samples, their packing and labelling shall comply with the requirements of ISO 3713.

8 Bibliography

ISO 5445, *Ferrosilicon — Specifications 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 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.*

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

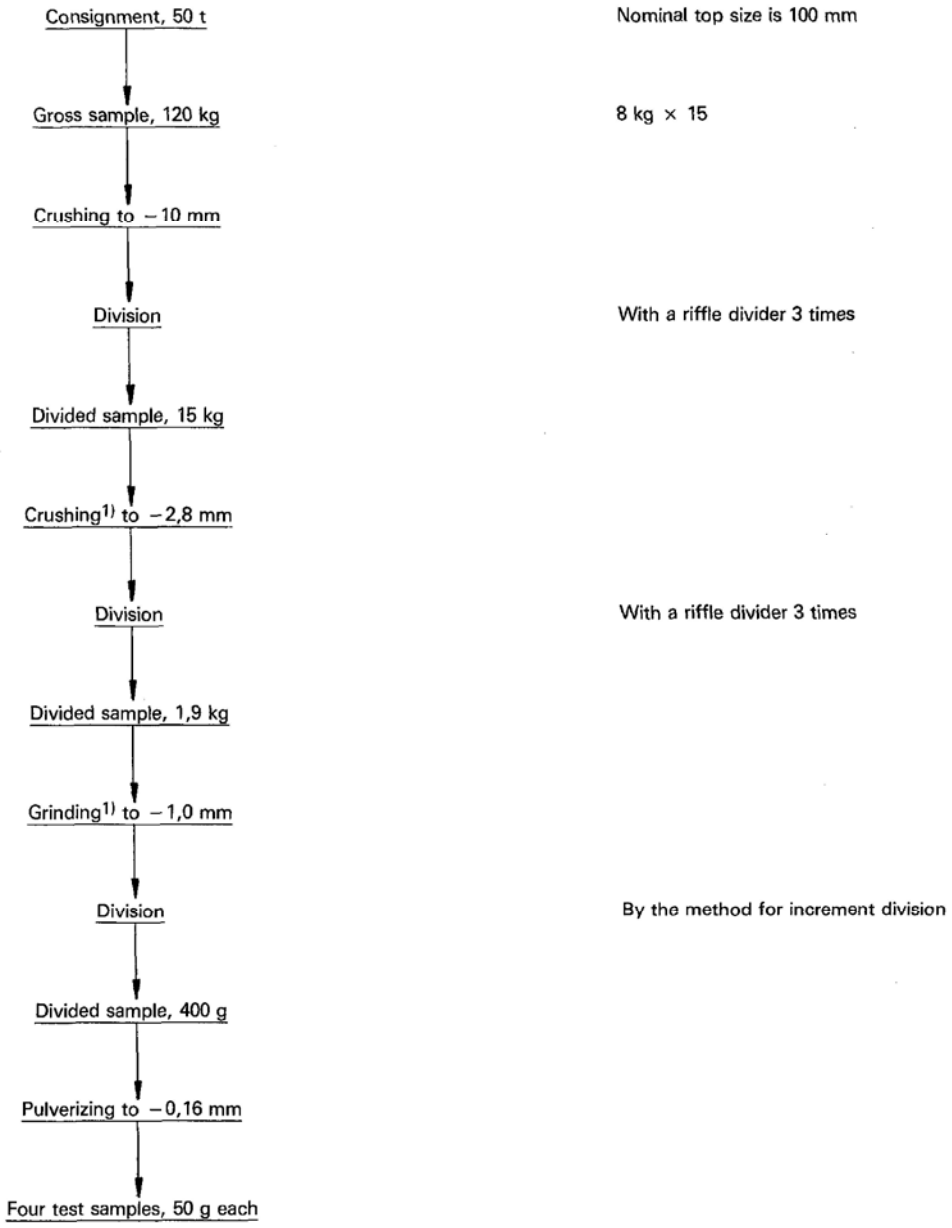

Figure 3 — Diagram for sample preparation of ferrosilicon (example)

1) One of the stages may be omitted.

Annex

Initial data for calculation of sampling parameters

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

A.1 Number of increments taken from one consignment

See tables 4 and 5.

A.1.1 Formula for calculation of the number of increments

The number of increments taken from one consignment was calculated using equation (6) in ISO 3713.

The correction factor $\frac{N-n}{N-1}$ was assumed to be equal to 1.

A.1.2 Heterogeneity of a consignment (standard deviation between increments, σ_i)

The standard deviation between increments σ_i was determined experimentally for consignments constituted by the graded lots method. The values for σ_i are shown in table 8 and correspond to the ranges of the quality characteristics specified in the International Standards on technical conditions for delivery of ferroalloys (see clause 8).

Table 8 — Values of consignment heterogeneity

Ferroalloy	Standard deviation between increments, σ_i, % (m/m)		
	Cr	Si	Mn
Ferrochromium	0,8		
Ferrosilicochromium	0,8	0,9	
Ferrosilicon		0,9	
Ferrosilicomanganese		0,65	0,75
Ferromanganese			0,70

A.1.3 Precision of sampling, β_S

The precision of sampling was assumed to be from about ± 0,25 % for consignments of mass 10 000 t up to about ± 0,7 % for consignments of mass 5 t.

For consignments of intermediate masses, the values of β_S were determined by extrapolation.

A.2 Overall precision of the determination of the chemical composition of a consignment, β_{SDM}

A.2.1 For a single measurement, the overall precision of the determination of the chemical composition of a consignment was calculated using equation (1) in ISO 3713.

A.2.2 Heterogeneity of consignment, σ_i, and number of increments, n

See clause A.1.

A.2.3 Standard deviation of sample preparation, σ_D

The values of σ_D were determined experimentally and are shown in table 9.

Table 9 — Standard deviation of sample preparation

Ferroalloy		Standard deviation of sample preparation, σ_D, % (m/m)		
		Cr	Si	Mn
Ferrochromium	crushable	0,2		
	non-crushable	0,3		
Ferrosilicochromium		0,2	0,2	
Ferrosilicon			0,3	
Ferrosilicomanganese			0,15	0,15
Ferromanganese				0,15

A.2.4 Standard deviation of the method of analysis, σ_M

The values of σ_M for a single measurement were taken from the International Standards on the methods of analysis of ferroalloys and are shown in table 10.

Table 10 — Standard deviation of the method of analysis

International Standard number	Standard deviation of analysis method, σ_M, % (m/m)						
	FeCr	FeSiCr	FeSi		FeSiMn	FeMn	
	Cr		Si			Mn	
ISO 4140	0,1	0,1					
ISO 4158			0,1	0,15	0,1		
ISO 4159						0,1	0,1

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Descriptors : ferroalloys, ferrochromium, ferrosilicochromium, ferrosilicon, ferrosilicomanganese, ferromanganese, chemical analysis, sampling, specimen, preparation.

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