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 4552-2 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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Annex

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Ferroalloys — Sampling and sample preparation for chemical analysis —

Part 2:
Ferrotitanium, ferromolybdenum, ferrotungsten, ferroniobium, ferrovanadium

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 ferrotitanium, ferrotungsten, ferromolybdenum, ferroniobium or ferrovanadium.

Part 1 of ISO 4552 specifies the methods for use with ferrochromium, ferrosilicon, ferrosilicomanganese and ferromanganese.

2 References

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

ISO 6467, *Ferrovanadium — Determination of vanadium — Potentiometric method.*

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*)
Ferrotitanium	Titanium content
Ferromolybdenum	Molybdenum content
Ferrotungsten	Tungsten content
Ferroniobium	Niobium content
Ferrovanadium	Vanadium 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 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	FeTi	FeMo	FeW	FeNb	FeV
		Ti	Mo	W	Nb	V
40	64	0,81	—	—	—	0,74
25	40	0,82	—	—	—	0,75
16	25	0,83	0,90	0,90	0,88	0,75
10	16	0,83	0,91	0,91	0,89	0,76
5	10	0,84	0,92	0,92	0,90	0,77
3	5	0,86	0,93	0,93	0,91	0,78
1	3	0,88	0,95	0,95	0,93	0,80
0,5	1	0,90	0,98	0,98	0,95	0,82
	0,5	0,95	1,03	1,03	0,98	0,89

5 Sampling

5.1 Mass of increment

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				
	FeTi	FeMo	FeW	FeNb	FeV
⩾ 50	5,0	5,0	5,0	3,5	1,0
50	3,5	3,5	3,5	2,5	0,5
25	1,5	1,5	1,5	1,0	0,2
⩽ 10	0,5	0,5	0,5	0,2	0,2

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.

NOTE — Other sampling parameters may be adopted by agreement between the interested parties. In such a case, the minimum number of increments should be calculated using the formula

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

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		FeTi	FeMo	FeW	FeNb
			Ti	Mo	W	Nb
40	64	28	0,23	—	—	—
25	40	24	0,25	—	—	—
16	25	20	0,27	0,29	0,29	0,25
10	16	17	0,29	0,32	0,32	0,27
5	10	14	0,32	0,35	0,35	0,29
3	5	11	0,36	0,39	0,39	0,33
1	3	9	0,40	0,43	0,43	0,37
0,5	1	7	0,45	0,49	0,49	0,42
	0,5	5	0,54	0,58	0,58	0,49

Table 5 — Minimum number of increments and sampling precision for ferrovanadium

Mass of consignment, t		Minimum number of increments	Precision of sampling, ± β_S % (m/m)
Over	Up to and including		V
40	64	23	0,19
25	40	20	0,20
16	25	17	0,22
10	16	14	0,24
5	10	11	0,27
3	5	9	0,30
1	3	7	0,34
0,5	1	5	0,40
	0,5	3	0,52

5.2.2 For ferrovanadium, 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 5.

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, the number of packed units taken shall correspond to that of the increments shown in tables 4 and 5. One increment shall be taken from each packed unit sampled.

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

5.4 Gross sample

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

If more precise determination of the quality characteristics of a consignment is required, each increment or each sub-sample may be prepared and analysed separately.

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 ± β_D, % (m/m)
Ferrotitanium	0,5 Ti
Ferromolybdenum	0,6 Mo
Ferrotungsten	0,6 W
Ferroniobium	0,6 Nb
Ferrovanadium	0,4 V

6.2 Sample division

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

6.2.2 A gross sample or an increment shall be crushed to particles which will pass through a sieve with a mesh size of 10 mm × 10 mm, and shall then be divided in accordance with table 7.

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

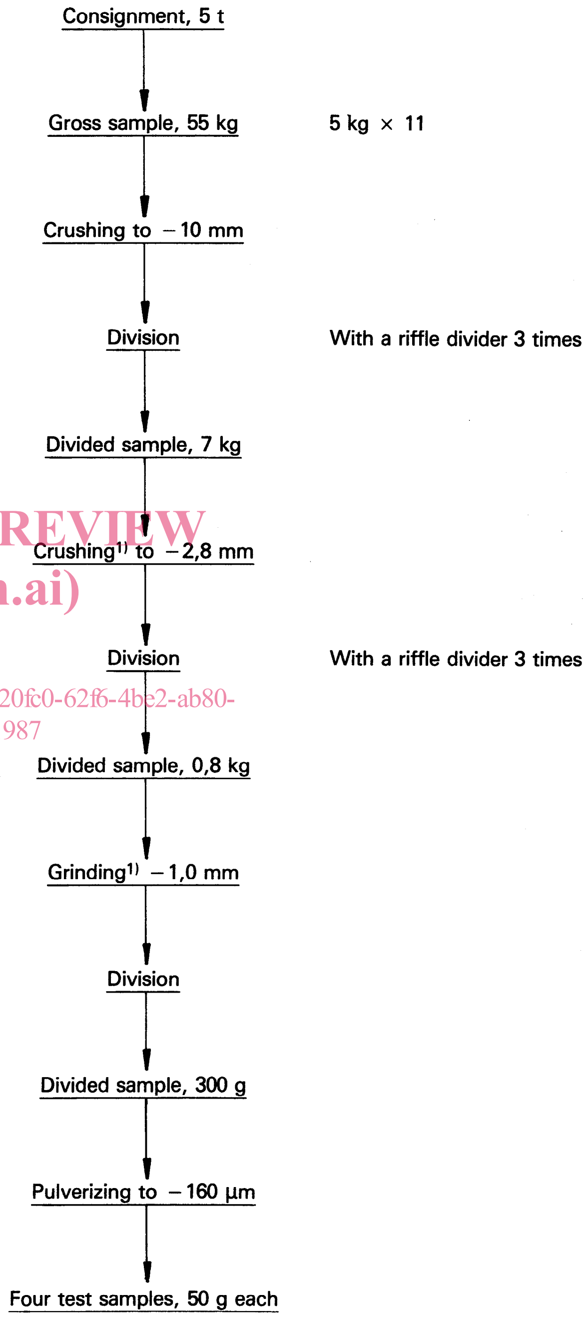

Figure — Diagram for gross sample preparation of ferrotitanium (example)

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[1] One of the stages may be omitted.

Table 7 — Rules for sample division of a gross sample or a subsample

Nominal top size in sample, mm	Minimum mass of divided sample, kg	
	FeTi, FeNb	FeMo, FeW, FeV
10	7,0	13,0
5	1,2	2,0
2,8	0,7	1,0
1,0	0,3	0,4
0,5	0,2	0,2

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 nominal top size in a test sample shall be not greater than 160 µm for all the ferroalloys except ferrovanadium, the nominal top size of which shall be not greater than 250 µm (see ISO 6467).

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

8 Bibliography

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

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

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

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

ISO 5454, *Ferrotitanium — Specifications 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.*

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. The values for σ_i adopted for the calculation of the increment number are shown in table 8.

A.1.3 Precision of sampling, β_S

The precision of sampling was assumed to be from about ± 0,20 % for consignments of mass 64 t to about ± 0,55 % for consignments of mass 0,5 t, which ensures a maximum value of the overall precision β_{SDM} of not more than ± 1,0 %.

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

A.2.1 Formula for calculation

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.

Table 8 — Values of consignment heterogeneity

Member Body	Standard deviation between increments, σ_i, % (m/m)							
	FeMo HC	FeMo LC	FeW	FeTi (L Ti)	FeTi (H Ti)	FeNb	FeV* (H V)	FeV* (L V)
	Mo		W	Ti		Nb	V	
Japan	0,65	0,45	0,56	0,465		0,353	0,261	—
USSR	—	0,70	0,67	0,345	0,67	0,63	—	0,57
σ_i	0,65	0,59	0,62	—	0,576	0,51	0,44	
	0,62							
Adopted for calculations	0,65		0,65	0,60		0,55	0,45	

* FeV40 melted by using converter slag of high vanadium content.