SLOVENSKI STANDARD
SIST ISO 10835:1998

01-maj-1998

Direktno reducirano ģelezo ï Vzorļ enje in priprava vzorca ï Metode za roļ no vzorļ enje paletirane in kosovne rude

Direct reduced iron -- Sampling and sample preparation -- Manual methods for reduced pellets and lump ores

Minerais de fer préréduits -- Échantillonnage et préparation des échantillons -- Méthodes manuelles pour granulés et morceaux de minerai réduits

Ta slovenski standard je istoveten z: ISO 10835:1995

ICS:

73.060.10	Železove rude	Iron ores

SIST ISO 10835:1998 **en**

INTERNATIONAL STANDARD

ISO 10835

First edition
1995-09-15

Direct reduced iron — Sampling and sample preparation — Manual methods for reduced pellets and lump ores

Minerais de fer préréduits — Échantillonnage et préparation des échantillons — Méthodes manuelles pour granulés et morceaux de minerai réduits

Reference number
ISO 10835:1995(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. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 10835 was prepared by Technical Committee ISO/TC 102, *Iron ores*, Subcommittee SC 1, *Sampling*.

Annexes A and B form an integral part of this International Standard. Annexes C, D and E are for information only.

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Direct reduced iron — Sampling and sample preparation — Manual methods for reduced pellets and lump ores

1 Scope

This International Standard specifies manual methods of increment sampling and sample preparation of direct reduced iron (DRI) to obtain samples for size analysis, moisture determination and chemical analysis.

The methods specified are applicable to the sampling and preparation of samples of reduced pellets and reduced lump ores (called "DRI" in this International Standard).

The methods for sampling are applicable to the taking of samples of DRI from conveyors, railway wagons or containers (including trucks) and stockpiles, during the loading or discharging of a lot in cases where manual sampling can be carried out safely and with due regard to the health of the operator.

NOTES

1 Sampling and sample preparation of DRI feedstock should follow ISO 3081, ISO 3082 and ISO 3083.

2 The theory and basic principles given in this International Standard are similar to those given in ISO 3081 and ISO 3083.

CAUTION — DRI may react with water and air to produce hydrogen and heat. The heat produced may cause ignition. Therefore due consideration shall be given to the safety of operators by respecting applicable regulations or international codes.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 3082:1987, *Iron ores — Increment sampling and sample preparation — Mechanical method.*

ISO 3085:—[1], *Iron ores — Experimental methods for checking the precision of sampling.*

ISO 3086:1986, *Iron ores — Experimental methods for checking the bias of sampling.*

ISO 11323:—[2], *Iron ores — Vocabulary.*

3 Definitions

For the purposes of this International Standard, the definitions given in ISO 11323 apply.

4 General procedures for manual sampling

Sampling shall be carried out while a lot is being transferred.

The general sampling procedure shall be as follows:

a) identify the lot to be sampled;

b) ascertain the nominal top size;

c) determine the mass of increment considering the nominal top size;

d) in the case of systematic or stratified random sampling, determine the minimum number of increments to be taken from the lot, allocate the wagons or containers to be selected from the entire lot and determine the number of increments to be taken from the wagons or containers selected;

1) To be published. (Revision of ISO 3085:1986)

2) To be published.

e) in the case of systematic sampling or stratified random sampling, determine the intervals for taking increments or, in the case of two-stage sampling on a mass basis, the interval for selecting the wagons or containers;

f) determine the point of sampling and the method of taking increments;

g) take increments having almost uniform mass during the whole period of handling the lot.

Sample containers for DRI shall be suitable for storing and transporting the material in very well protected conditions. Samples shall be stored in airtight containers and shall not be left unprotected from the atmosphere at any stage.

5 Fundamentals of sampling

5.1 Overall precision

This International Standard is designed to attain a level of overall precision, β_{SPM}, of 1,5 % at a probability level of 95 %, with respect to the mean values of the metallic iron content of a lot.

The overall precision, β_{SPM}, is a measure of the combined precision of sampling, sample preparation and measurement, and is twice the overall precision in terms of the standard deviation, σ_{SPM}, expressed as an absolute percentage; i.e.,

$$\beta_{SPM} = 2\sigma_{SPM} = \sqrt{\beta_S^2 + \beta_P^2 + \beta_M^2}$$

where

β_S is the precision of sampling;

β_P is the precision of sample preparation;

β_M is the precision of measurement.

5.2 Minimum mass of increment

5.2.1 The mass of each increment shall be as specified in table 1 according to the nominal top size of the DRI sampled.

Table 1 — Minimum mass of increment

Nominal top size mm		Minimum mass of increment kg
Over	Up to and including	
50		12
22,4	50	4
	22,4	0,8

5.2.2 Increments shall be taken in such a manner as to ensure that they are of almost uniform mass. "Almost uniform mass" means that the variation in mass should be less than 20 % in terms of the coefficient of variation. The coefficient of variation (CV), expressed as a percentage, is defined as the ratio of the standard deviation, σ, relative to the mean value, m, of the mass of increments times 100; i.e.,

$$CV = \left(\frac{\sigma}{m}\right) \times 100 < 20\ \%$$

5.3 Number of increments

The minimum number of increments to be taken from a lot shall be the number, n_1, specified in table 2 according to the mass of the lot irrespective of the method of sampling.

Table 2 — Minimum number of increments required, n_1

Mass of lot t		Number of increments n_1
Over	Up to and including	
30 000		35
15 000	30 000	30
5 000	15 000	25
2 000	5 000	20
1 000	2 000	15
500	1 000	10
	500	8

5.4 Method of taking increments

5.4.1 Each increment shall be taken at one time by a single motion of a sampling device from a point selected at random (with equal probability). However, if this is difficult, it may be taken by several motions of the sampling device. The latter shall be proven to have no bias before being applied.

5.4.2 The increments should be taken in such a manner as to ensure that they are of "almost uniform mass" as described in 5.2.2.

In exceptional cases, where increments of almost uniform mass cannot be taken, each increment shall be prepared individually and the quality characteristics of each increment shall be determined. Alternatively, at an appropriate stage of the sample preparation, the divided increments of almost uniform mass may be combined into partial samples or a gross sample.

5.4.3 When the calculated mass of a sample is less than that required for preparing the required test samples, the mass of increment and/or the number of

increments to be taken shall be increased to satisfy the minimum mass required for testing.

6 Apparatus for manual sampling

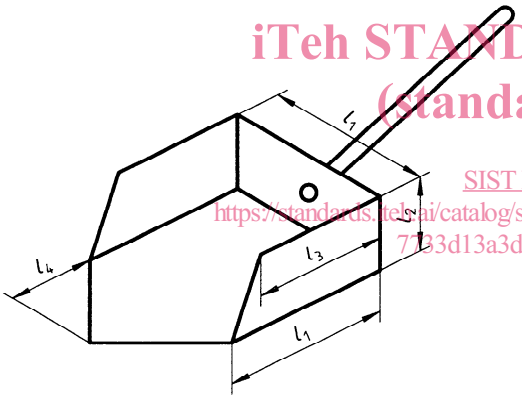
Sampling devices capable of taking the specified mass of increment without any significant bias shall be used.

A shovel for taking increments from a lot should be of the type and dimensions specified in table 3 and figure 1.

Table 3 — Dimensions of increment shovel

Dimensions in millimetres

Nominal top size	Shovel No.	Dimensions of increment shovel			
		l_1	l_2	l_3	l_4
50	50	150	75	130	65
22,4	22,4	80	45	70	35



NOTE — The shovel may have a triangular edge if this proves convenient for insertion of the shovel into the DRI.

Figure 1 — Example of increment shovel

NOTE 3 Other sampling devices, including mechanically assisted devices, may be used to take increments. These devices should have a minimum opening equivalent to l_1 in table 3 and in the case of nominal top sizes over 50 mm, at least three times the nominal top size. The volume of the device in the effective collection area should be sufficient to hold at least twice the minimum mass of increment given in table 1.

7 Methods of manual sampling

7.1 Sampling from conveyors

7.1.1 When the increment is taken from a stopped conveyor belt, a section of adequate length in the direction of the stream and of the full width and thickness of the DRI stream should be taken from the specified position.

The "adequate length" shall be sufficient to ensure that the minimum mass of increment as specified in table 1 can be taken, and shall be more than three times the nominal top size and greater than the width of the smallest increment shovel, 80 mm.

When taking the increment from the conveyor, a sampling frame may be used for convenience.

7.1.2 When the increment is taken from a moving conveyor, the full width and thickness of the DRI stream shall be taken from the falling stream.

7.1.3 The interval for taking increments shall be uniform, on a mass basis, throughout the whole lot and shall not be changed during the handling of the lot.

7.1.3.1 The mass interval, Δm, in tonnes, between taking increments shall be calculated from the following formula.

$$\Delta m = \frac{m_1}{n_1}$$

where

m_1 is the mass, in tonnes, of the lot;

n_1 is the number of increments determined in 5.3.

7.1.3.2 The mass interval for taking increments shall be less than the calculated mass interval, Δm, in 7.1.3.1, to ensure that the number of increments exceeds the minimum specified in 5.3.

7.1.3.3 If the flow rate of the DRI stream is almost uniform, the mass interval may be converted into an equivalent time interval.

7.1.4 The first increment shall be taken after a randomly selected mass has been handled within the first mass interval after the start of the handling operation.

7.1.5 The increments shall be taken subsequently at a fixed mass interval until the handling of the lot has been completed.

7.2 Sampling from wagons or containers

7.2.1 Method of taking increments

7.2.1.1 The increments shall be taken at random from the new surface of DRI exposed during the loading or the unloading of the wagons or containers.

7.2.1.2 When it is suspected that there is some bias between strata (between the top and bottom, the front and the rear, or the left and the right) in the DRI in the wagon or container, it is advisable to take increments from each stratum in each of the wagons or containers selected.

7.2.1.3 There is a danger of introducing bias when the sampling is conducted with a sampling probe or an auger from the top surface of DRI in wagons or containers, or accordingly, this shall be used only after is has been ascertained, by check experiments, that the bias is not significant.

7.2.2 Sampling from all wagons or containers (stratified sampling)

The number of increments, n_3, to be taken from each wagon or container of the lot shall be calculated from the equation

$$n_3 = \frac{n_1}{n_4}$$

where

n_1 is the number of increments in table 2 according to the mass of the lot;

n_4 is the number of wagons or containers in the lot.

The result obtained shall be rounded up to the next higher whole number.

7.2.3 Sampling from selected wagons or containers (two-stage sampling)

The minimum number of wagons or containers to be selected, n_2, shall be identical to the minimum number of increments, n_1, specified in table 2. One increment shall be taken from each wagon or container selected. The interval between selecting wagons or containers, n_5, shall be calculated from the formula

$$n_5 = \frac{n_4}{n_2}$$

where n_2 is the minimum number of wagons or containers to be selected.

The result obtained shall be rounded down to the next lower whole number to ensure adequate precision.

7.3 Sampling from bunker discharge

The sampling of DRI from bunker discharge shall be conducted in accordance with the method specified in 7.1 after the DRI has been transferred to a conveyor.

7.4 Sampling from stockpiles

Sampling shall not be conducted directly from stockpiles which are not being formed and reclaimed. If this were to be done, the precision of sampling would not be in accordance with this International Standard and some significant bias may be introduced.

The sampling of DRI from stockpiles shall be performed from conveyors either by stopped-belt sampling or from a transfer point in accordance with the method specified in 7.1 while the stockpile is being formed or reclaimed.

8 General procedures for sample preparation

The increments taken in accordance with the method specified in clause 7 shall be prepared according to the following general procedures:

a) determine whether test samples are to be prepared from each increment, from each partial sample or from the gross sample according to the requirements for the determination of each quality characteristics;

b) determine whether the sample is for split use or for multiple use;

c) select the method and type of sample division at each stage;

d) establish the flow of sample preparation including the processes of division, comminution and mixing;

e) prepare the test sample.

The preparation of samples of DRI shall be conducted with extreme care to minimize the chance of reoxidation due to dampness, overheating or both. All equipment should be thoroughly cleaned to remove remnants of deleterious material and it is desirable to flush the equipment just prior to use with a small quantity of the same DRI.

9 Fundamentals of sample preparation

9.1 Precision of sample preparation

The precision of sample preparation, β_P, shall be within 0,3 % in metallic iron content with a 95 % probability. However, if sample preparation is carried out first on individual increments or partial samples at an appropriate stage of the sample preparation and then these divided increments or partial samples are combined into a gross sample, the precision of sample preparation can be further improved (see C.2 and C.3).

The overall precision, β_{SPM}, for the cases where division and measurement are carried out on the gross sample, on each of the partial samples or on each of the increments may be calculated according to Annex C.

9.2 Constitution of samples

When samples are to be constituted from the increments, the following shall be taken into consideration:

a) quality characteristics to be determined;

b) required overall precision;

c) coefficient of variation (CV) in mass of increments taken by mass-basis sampling.

9.3 Division rules

In order to obtain the specified precision of sample preparation the following aspects of division shall be taken into consideration:

a) minimum mass of the sample after division, specified for each quality characteristic to be determined;

b) method and type of division to be adopted;

c) nominal top size of the sample to be divided.

9.4 Method and type of division

One or more of the following methods of sample division shall be conducted individually or jointly:

a) manual increment division method (see 12.1);

b) manual riffle division method (see 12.2);

c) mechanical division methods (see ISO 3082).

NOTE 4 Each sample preparation stage has its own variance and these variances are additive.

This International Standard specifies two methods of manual division a) and b), which shall be applied to increments or partial samples as shown in table 4.

NOTE 5 "Increment" in the increment division method is not the same as that taken by the sampling procedure from the lot and means a quantity taken by the division method specified in 12.1. See also the definition in ISO 11323. "Increment" taken by the sampling procedure is referred to hereafter as "Increment (primary)", if necessary.

Combining of increments taken by time-basis sampling and mass-basis sampling shall incorporate the procedures specified in clause 11.

Table 4 — Application of manual division method

Division of	Condition of increment	Manual division method to be applied	
		Constant mass division	Fixed rate division
	CV %	Increment division method	Riffle division method
Increment	< 20	yes[1)]	yes
(primary)	⩾ 20	yes	no[1)]
Partial sample		yes	yes
Gross sample		yes	yes

1) "yes" denotes applicable and "no" denotes not applicable.

9.5 Split use and multiple use of sample

When a sample taken from the lot meets the respective requirements for the determination of moisture content, size analysis and chemical analysis, the test samples may be obtained from split use or multiple use samples.

9.6 Crushing and grinding

The crushing and grinding shall be conducted with a crusher and a grinder suitable for the size and mechanical strength of the DRI particles.

The crusher and grinder should be purged just before use with DRI from the same source.

Precautions shall be taken to minimize overheating and reoxidation, and to avoid the production of "plates" of metal.

9.7 Mixing

By mixing the sample thoroughly, it can be made homogeneous and consequently the errors in sample division can be lessened.

The mixing may be conducted either by a mechanical mixer or by hand. The mixer shall be selected to suit the sample and its particle size.

9.8 Requirements for sample preparation

9.8.1 Sample preparation shall be carried out so that there is no significant contamination or introduction of material other than the sample and no change in the quality.

9.8.2 Check experiments for precision and bias shall be carried out regularly on the sample preparation process in accordance with ISO 3085 and ISO 3086,

respectively, so that the precision of sample preparation is known and that any bias in the results caused by the preparation process may be detected.

10 Apparatus for sample preparation

The following apparatus, which shall be thoroughly cleaned and examined before and after use, shall be provided for sample preparation.

a) Crushers and grinders, e.g. jaw crusher, cone crusher, vertical mill, ring grinder, and agate pestle and mortar.

b) Mixers, e.g. double-cone mixer.

c) Riffles, details of which are given in Annex A.

d) Scoops, for increment division, details of which are given in figure 2.

11 Combining increments for sample preparation

The method of combining increments shall be selected according to the type of sampling employed for taking increments, viz. whether the increments have been taken by mass basis sampling or by time basis sampling. Systematic sampling is classified into two types, viz. mass basis and time basis. Stratified and two-stage sampling are performed on a mass basis.

11.1 Combining increments taken by mass-basis sampling

11.1.1 Constitution of partial samples or gross sample from increments

The increments either as-taken or after having been prepared individually by constant-mass or fixed-rate division at an appropriate stage shall be combined into partial samples or a gross sample.

When the variation in mass of individual increments is 20 % or over ($CV \geq 20$ %), the increments as-taken shall not be combined into partial samples or a gross sample. The increments shall be prepared individually by constant-mass division at a practical stage before being combined into partial samples or gross sample (see table 4). Otherwise, each increment shall be prepared separately and then subjected to the determination of quality characteristics.

11.1.2 Constitution of gross sample from partial samples

The partial samples constituted according to 11.1.1 can, with or without division, be combined into a gross sample.

When division is carried out on each partial sample to constitute the gross sample, the division shall be carried out as follows:

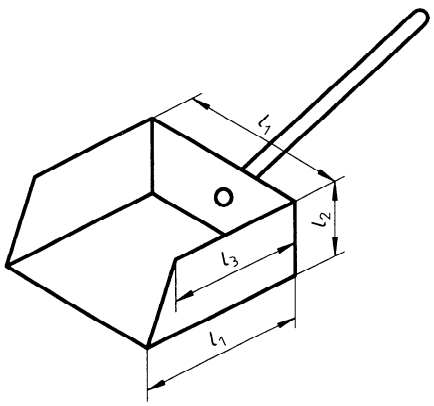
a) if the partial samples consist of an equal number of increments, constant-mass or fixed-rate division may be applied;

b) if the partial samples consist of different number of increments, only fixed-rate division shall be applied.

11.2 Combining increments taken by time-basis sampling

11.2.1 Constitution of partial or gross samples from increments

11.2.1.1 The increments as taken shall be combined into partial samples or a gross sample, irrespective of the variation in mass of increments.



Scoop number	Dimensions of scoop mm			Thickness of metal sheet mm	Approximate volume ml
	l_1	l_2	l_3		
31,5 D	90	60	80	2	450
22,4 D	80	45	70	2	270
16 D	70	40	60	2	180
10 D	60	35	50	1	110
5 D	50	30	40	1	65
2,8 D	40	25	30	0,5	35
1 D	30	15	25	0,5	10
0,5 D	20	10	20	0,5	4
0,25 D	15	10	12	0,3	2

Figure 2 — Scoop for increment division and its dimensions

11.2.1.2 When division is carried out on each increment and the divided increments are combined into partial samples or a gross sample, the division shall be carried out on each increment by fixed-rate division at any stage (see table 4).

11.2.2 Constitution of gross sample from partial samples

11.2.2.1 The partial samples constituted according to 11.2.1 shall, with or without division, be combined into a gross sample, irrespective of the variation in mass of partial samples.

11.2.2.2 When division is carried out on each partial sample and the divided partial samples are combined into a gross sample, the division shall be carried out on each partial sample by fixed-rate division at any stage (see table 4).

12 Manual methods of division

12.1 Manual increment division method

The manual increment division method will provide the specified precision in spite of the high division ratio. The manual increment method shall be applied to DRI of up to 31,5 mm nominal top size.

However, this method should not be applied to reduced pellets, which roll freely and/or segregate easily (see 12.2). When the reduced pellets have been crushed to a sufficiently small particle size, this method may be applied satisfactorily.

The manual increment division method shall be carried out using a scoop for increment division.

12.1.1 Mass of increments

The mass of each increment shall be as specified in table 5.

Table 5 — Minimum mass of each increment by manual increment division method

Nominal top size mm		Minimum mass of each increment g
Over	Up to and including	
22,4	31,5	1 000
16	22,4	600
10	16	400
5	10	250
2,8	5	150
1	2,8	80
0,5	1	25
0,25	0,5	10
	0,25	5

12.1.2 Number of increments

The number of increments to be taken shall be as specified in table 6.

Table 6 — Number of increments to be taken by manual increment division method

Division of	Minimum number of increments
Gross sample	20
Partial sample	12
Increment (primary)	4

A lesser number may be taken provided it has been demonstrated that no significant bias and/or lack of precision is introduced (see ISO 3085 and ISO 3086).

12.1.3 Procedure

Sample division by manual increment division shall be carried out as follows.

12.1.3.1 Form the sample to be divided on a smooth and flat plate (non-moisture absorbing) into a flat rectangle with a uniform layer thickness as specified in table 7.

12.1.3.2 Arrange the rectangle in the same number of parts as the minimum number of increments specified in table 6.

Table 7 — Nominal top size, thickness of spread sample and scoop for increment division

Dimensions in millimetres

Nominal top size		Thickness of spread sample for increment division	Scoop for increment division
Over	Up to and including		Number
22,4	31,5	60 to 80	31,5 D
16	22,4	50 to 60	22,4 D
10	16	40 to 50	16 D
5	10	30 to 40	10 D
2,8	5	25 to 35	5 D
1	2,8	20 to 30	2,8 D
0,5	1	10 to 20	1 D
0,25	0,5	5 to 10	0,5 D
	0,25	5 to 10	0,25 D

12.1.3.3 Select an appropriate scoop as designated in figure 2, according to the nominal top size. Take a scoopful of sample from each of the parts (the place of taking such an increment being selected at random in each part) and combine these scoopfuls of sample.

The scoop shall be thrust into the bottom of the sample layer by the above procedure. It is recommended that a bump plate be held vertically in front of the scoop. The bump plate shall be thrust into