SLOVENSKI STANDARD
SIST EN 28050:2009

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Feronikelj v ingotih ali kosih - Vzorļenje za analizo (ISO 8050:1988)

Ferronickel ingots or pieces - Sampling for analysis (ISO 8050:1988)

Ferronickelbarren oder -stücke - Probenahme für Analyse (ISO 8050:1988)

Ferro-nickel en lingots ou en morceaux - Echantillonnage pour analyse (ISO 8050:1988)

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English version

Ferronickel ingots or pieces — Sampling for analysis

(ISO 8050 : 1988)

Ferro-nickel en lingots ou en morceaux — Échantillonnage pour analyse (ISO 8050 : 1988)

Ferronickelbarren oder -stücke — Probenahme für Analyse (ISO 8050 : 1988)

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CEN

European Committee for Standardization
Comité Européen de Normalisation
Europäisches Komitee für Normung

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Foreword

On the proposal of the CEN Central Secretariat, the Technical Board has decided by Resolution BT C157/1990 to submit the International Standard ISO 8050 : 1988 'Ferronickel ingots or pieces — Sampling for analysis' to the formal vote.

This European Standard EN 28050 was approved by CEN on 1992-05-08.

National standards identical to this European Standard shall be published at the latest by 1992-11-30 and conflicting national standards shall be withdrawn at the latest by 1992-11-30.

According to the CEN/CENELEC Internal Regulations, the following countries are bound to implement this European Standard:

Austria, Belgium, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 8050 : 1988 was approved by CEN as a European Standard without any modification.

Ferronickel ingots or pieces — Sampling for analysis

1 Scope

This International Standard specifies a method for sampling of ferronickel lots in ingot or piece form with a view to obtaining a representative laboratory sample for the determination of the chemical composition of the lot.

As agreed between the purchaser and the supplier, a choice is to be made between two procedures:

— The first procedure can be applied at the producer's plant during casting (description in clauses 3 and 5).

— The second procedure can be applied to lots as delivered at the buyer's premises[1] (description in clauses 4 and 5). It includes two alternatives for sample taking (drilling and milling).

Each party is entitled to participate in (or be represented at) sampling operations.

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 listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 513 : 1975, *Application of carbides for machining by chip removal — Designation of the main groups of chip removal and groups of application.*

ISO 3855 : 1977, *Milling cutters — Nomenclature.*

ISO 4957 : 1980, *Tool steels.*

ISO 6352 : 1985, *Ferronickel — Determination of nickel content — Dimethylglyoxime gravimetric method.*

ISO 6501 : 1988, *Ferronickel — Specifications and conditions of delivery.*

3 Sampling of each heat at the producer's plant

3.1 Taking the primary sample during casting

3.1.1 Each increment[2] shall be taken using a spoon and cast in a mould to obtain a suitable small ingot for chemical or physical analysis. The usual geometry of such ingots is that of a frustum. It is desirable that the dimensions lie within the following limits:

— height: 100 to 140 mm

— upper diameter: 35 to 50 mm

— lower diameter: 30 to 40 mm

The ingot mould shall be made of material that allows the sample to cool rapidly; a large block of copper would meet this requirement.

If necessary, the sample shall be killed to obtain a small ingot of sound metal (free of cracks and blowholes). Killing is most often done with aluminium, in wire or chip form, at 1 to 2 g of aluminium per kilogram.

The large height of the small ingots permits small shrinkholes to be confined to the upper part, thus ensuring that the lower part of the ingot is perfectly sound and homogenous and hence suitable for analysis. Generally, a height of 120 mm will guarantee that the sound portion extends at least 70 mm up from the base.

3.1.2 In general, the small ingots are used for physical analysis on solid metal after a disc is cut from them.

Whenever possible, this analysis should produce results as accurate as those obtained from chemical analysis of chips. To achieve such accuracy, it is often necessary to analyse several small ingots several times each.

A specified number of small ingots shall therefore be taken at regularly spaced intervals during the casting.

Examples are given in annex A to indicate the number of small ingots to be taken and the number to be analysed. It is suggested that four to eight small ingots be taken from each heat.

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1) This procedure can be applied at the producer's plant, at the buyer's premises or at an intermediate transit place, as agreed between the interested parties.

2) Increment: portion of the lot taken in a single operation; in this standard: portion of molten metal.

It may happen that for some exceptional reasons there are not enough small ingots available which are suitable either for accurate physical analysis or for accurate analysis of chips taken from them: some small ingots may no longer be available or may contain cracks or blowholes. In these circumstances, the ingots constituting the heat shall be sampled. Five ingots[1] shall be taken from the heat, and the procedure described beginning at clause 4.1.3[1) 2)] shall then be followed.

3.2 Secondary sampling of small ingots taken from the heat

3.2.1 Cutting

Each small ingot shall be cut in two about 10 or 15 mm from the bottom (the smaller-diameter end) with a cut-off wheel (made of carborundum or corundum, for example).

The use of water cooling is recommended, because it avoids heating of the sample and hence changes in the crystalline structure of the metal.

3.2.2 Use of the two pieces of the small ingot

3.2.2.1 The disc cut off the small ingot may be used for physical analysis (i.e. X-ray fluorescence or optical emission spectrometry) of the solid metal after adequate machining of the cut surface.

For the number of small ingots to be analysed and the number of determinations per small ingot, see annex A.

3.2.2.2 The larger piece may be used for preparing chips, using one of the two following methods:

a) Drilling

With the freshly cut surface facing upwards, the piece shall be drilled to a depth such that the pipe in the upper part of the small ingot (i.e. at the wider end) is not reached.

It is desirable to limit the depth of the drilled hole to 50 mm. By using a 20 mm diameter drill, more than 100 g of chips are obtained.

All the chips shall be collected. An assembly similar to that of figure 1 may be used for this purpose. It is particularly suitable when using a drill bit designed for oil cooling but fed with compressed air (see annex D, D.6.3). The assembly shall be made of materials which will not contaminate the chips. The recommended conditions for drilling are described in annex D.

b) Milling

The conical surface adjacent to the freshly cut surface shall be cleaned by grinding with a corundum (aluminium oxide) or carborundum grinding wheel and then milled to a depth of about 20 mm from the cut surface (this will provide 100 g of chips).

All the chips shall be collected.

The recommended conditions for milling are described in annex D.

All the chips obtained from the selected small ingots by one of the two techniques described above shall be combined to constitute the secondary sample which is treated according to the procedure in clause 5 to obtain the final laboratory sample.

4 Sampling of a lot of ingots or pieces

4.1 Sampling of ingots or pieces

4.1.1 Lots comprising one heat

The procedure as described in the last paragraph of 3.1 is used (5 ingots or pieces are taken in accordance with the rules for random sampling).

4.1.2 Lots comprising several heats

The minimum number N of ingots or pieces to be taken is given by the following rules[3]:

For lot tonnages between 5 and 80 t

$$N = 50$$

For lot tonnages between 80 and 500 t

$$N = 54 - \frac{T}{20} \text{ (see footnote 4)}$$

where T is the mass, in metric tons, of the lot.

1) The rules for random sampling given in annex B may be used.

2) A very small number of ingots constitutes a sufficiently representative sample of the heat, because the contents of different ingots from the same heat do not vary greatly (see annex C).

3) These rules have been established making the following practical assumptions:

- the lots are composed of heats each of mass about 20 t;
- the nickel contents of the heats in the lot lie within the range from k to $(k + 1)$ %, where k is an integer;
- variations in nickel content within ingots and between ingots from the same heat are negligible compared with the range k to $(k + 1)$ %.

Complete justification of these rules is given in annex C.

4) This rule applies only when the tonnage does not exceed 500 t. If, following an agreement between the purchaser and the supplier, a consignment is between 500 and 1 000 t, the interested parties may agree to use one of the following procedures:

- division of the consignment into lots of tonnages not exceeding 500 t;
- taking, from the whole consignment, only the number of ingots or pieces specified for a tonnage of 500 t, i.e. $N = 29$. This procedure considerably diminishes the quantity of work involved in taking secondary samples from ingots or pieces.

These rules are illustrated in table 1.

Table 1 — Number of ingots or pieces to be taken as a function of lot tonnage

Tonnage T of ferronickel t	Number N of ingots or pieces to be taken
5 to 80	50
100	49
140	47
200	44
240	42
300	39
340	37
400	34
440	32
500	29
500 to 1 000	29

The number of ingots or pieces may be increased by agreement between supplier and purchaser.

The rules for random sampling shall be respected. In order that such is the case for different methods of delivery, the procedure given in annex B is recommended.

4.1.3 The surface of each ingot or piece taken shall be carefully cleaned by washing, brushing or wiping as required in order to eliminate all foreign material (earth, dust, oil, etc.).

The selected ingots or pieces constitute the primary sample.

4.2 Taking chips from ingots or pieces

This procedure consists of producing turnings by either drilling or milling. These operations shall be performed in such a way that the chips are not contaminated by tool wear, dust or grease. In particular, the work shall be done dry.

For a detailed description of the conditions for machining, see annex D.

Certain types of ferronickel are extremely hard. Great care shall therefore be taken to ensure that suitable cutting tools and conditions are selected.

For hard types of ferronickel, heat treatment (tempering) of the solid metal (ingot, ingot part or piece) may be desirable, because it makes it much easier to take chips. (For details, see annex D, clause D.2.)

4.2.1 Drilling

Each ingot shall be drilled to half-thickness at one point using either a drill made of high-speed steel or a tungsten carbide drill. Drilling shall be done from the upper surface of one ingot and the lower surface of the next.

A drill between 12 and 20 mm in diameter is recommended; drills in the 15 to 17 mm range are most frequently used.

NOTE — Examples of suitable drills, and the conditions under which they should be used, are given in annex D.

Chips shall be discarded until the drill has worked its way into the material to its full diameter. All the remaining chips shall then be collected.

An assembly similar to that shown in figure 1 may be used for collection. It shall be made of materials that will not contaminate the chips.

For pieces, the drill shall penetrate to mid-thickness of the piece.

4.2.2 Milling

The ingots shall be cut with corundum (aluminium oxide) or carborundum wheels.

Either each ingot shall be cut once, and one of the two resulting pieces then milled, or a slice of thickness sufficient for milling shall be cut from the ingot.

The outside surface adjacent to the freshly cut surface to be milled shall be cleaned. This can be done with a corundum or carborundum grinding wheel.

The surface shall then be milled with a suitable milling cutter, and all the chips collected.

Pieces shall be cut and milled in the same way as ingots.

NOTE — Examples of suitable milling cutters, and the conditions under which they should be used, are given in annex D.

The chips obtained either by drilling or milling shall have a mass of a least 1 kg. These chips constitute the secondary sample which shall be treated as described in clause 5.

5 Treatment of chips

The secondary sample is constituted of chips obtained as described in 3.2.2.2 or 4.2.

5.1 Washing

It is strongly recommended that the complete secondary sample be washed twice with pure acetone (or once with pure acetone and once with pure ether) to remove from the surface of the metal any accidental contamination by lubricants, dust, etc., which is inevitably present on the machine-tool.

The bulk of the solvent is allowed to drain off, residual solvent allowed to evaporate in air and the sample dried for at least 0,5 h in an oven at 100 to 110 °C[1].

1) Pure organic solvents shall be used and then evaporated as completely as possible so that carbon and sulfur can be determined with automatic equipment using dry instrumental techniques.

5.2 Crushing

Sample chips from a single heat (see 4.1.1) need not be crushed because no problem of homogenization of the sample exists. They may therefore be treated directly as in 5.3.

For sample chips from lots comprising more than one heat (see 4.1.2), it is important to homogenize the sample. This process is much easier if the shape of the chips is such that they do not become entangled in one another. The shape of the chips is determined primarily by the drilling or milling technique used (see annex D). In all cases, homogenization will be facilitated by crushing the chips.

Whether the chips can be crushed or not depends on:

— the nickel content; if this exceeds 35 %, the alloy is ductile and cannot be crushed;

— the quantities of impurities present (especially carbon); high-carbon ferronickels can be crushed much more finely than low-carbon ferronickels.

When the ferronickel to be analysed can be crushed, this shall be done with a suitable crusher that does not introduce iron contamination. Laboratory vibratory crushers do this in 10 to 30 s. The crusher container shall ideally be made of tungsten carbide; if that material is unavailable, abrasion-resistant steel is acceptable. (Ball mills and rod mills are unacceptable.)

Crushing ferronickels of less than 35 % nickel content for 30 s will normally produce material of a fineness such that, if a sieving operation is performed, almost all of it will pass through

— a 2,5 mm (8 mesh) screen in the case of low-carbon (LC) ferronickels;

— a 0,8 mm (20 mesh) screen in the case of medium- and high-carbon (MC and HC) ferronickels.

If the crusher is not large enough to take the whole sample at once, the chips may be crushed in several successive portions.

5.3 Homogenization

The whole sample shall be thoroughly homogenized (alternate shovelling, several passes through a riffle divider keeping all the material, mechanical homogenization, etc.).

5.4 Division

The sample shall be divided into 100 g portions using a riffle divider or a rotary sampler.

For low-carbon ferronickels, each fraction shall be kept in a glass bottle with a stopper of such material that it cannot be contaminated by abrasion, particularly by carbon; no contact shall be permitted with paper, cardboard, rubber, cork or plastic material. The same care shall be taken at all stages of sampling. In particular, chips shall never be handled on paper (use, instead, aluminium foil, for instance).

With medium- and high-carbon (MC and HC) ferronickels, each fraction may be kept in a heavy-duty polyethylene bag.

The number of fractions will depend on the number of samples for analysis desired by each party. The minimum shall be

— 1 for the purchaser,

— 1 for the supplier,

— 1 for the referee,

— 1 as reserve.

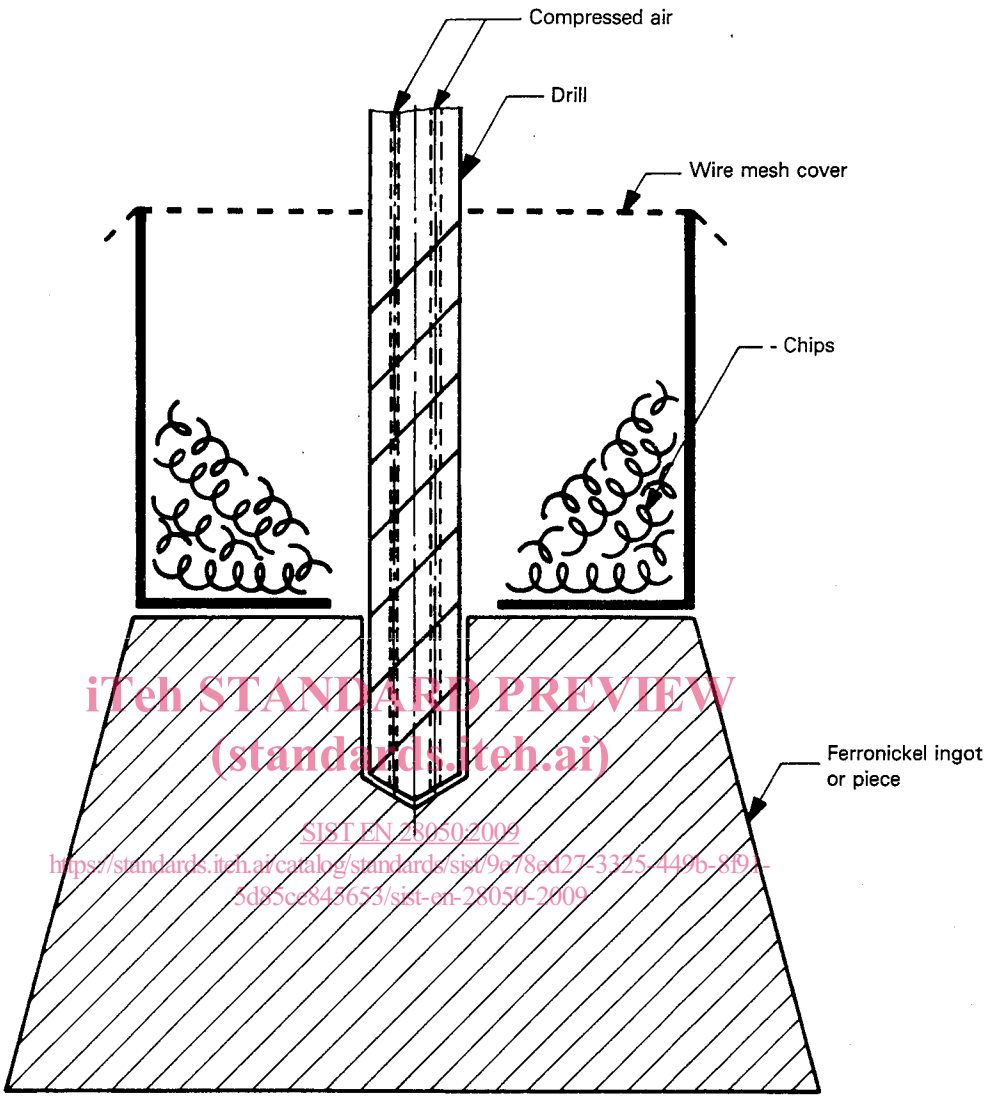

Figure 1 — Collection box for drilling chips

(For use especially when using a drill bit designed for oil cooling but fed with compressed air)

Annex A
(normative)

Number of small ingots to be taken and number to be analysed

A.1 When physical analysis on the solid metal is to be performed using small ingots taken during casting, the procedure shall be such that the precision approximates that of the chemical analysis of chips.

This is especially the case for the determination of nickel content[1].

To achieve this precision, the number of small ingots cast and analysed shall be sufficient to ensure that the sample is representative of the consignment. In addition, either a single determination shall be made on each small ingot and the mean calculated, or several determinations made on each small ingot and the mean calculated.

Moreover, the actual conditions under which casting, sampling and physical analysis are carried out may vary significantly from one manufacturer to another. No strict, general rule can therefore be given concerning the number of small ingots to be taken and the number to be analysed.

A.2 We therefore simply describe three practical examples in which the following general guidelines are observed:

- Number of small ingots taken : 4 to 8
- Number of small ingots analysed : 2 to 5
- Number of determinations per small ingot : 1 to 3

The number of small ingots taken is greater than the number analysed, because there must be enough small ingots to allow for exceptional cases in which some of them have defects such as cracks or blowholes[2].

Example 1

Eight small ingots are taken at regular intervals during casting. Five of the small ingots are then cut and a determination is performed on each of them.

In the exceptional case that a small ingot is found to be defective, one of the three remaining small ingots may be used.

The final result is the average of the five determinations.

Example 2

Five small ingots are taken at regular intervals during casting. After cutting, three of these ingots are selected and two determinations are performed on each of two ingots chosen out of the three. The average of the two determinations is then computed for each of the two ingots. If the difference between the two averages is less than 0,20 percentage points (for nickel content), the average of the four determinations is the final result.

If the difference exceeds 0,20 percentage points, a second set of analyses is performed on the three small ingots selected, after new machining of the cut surface. An average is obtained from the seven results, possibly with elimination of one or two outlying values.

Example 3

Use of small ingots to obtain chips.

Five small ingots are taken at regular intervals during casting. After cutting, three of them are selected and chips produced from the larger pieces according to the procedure described in 3.2.2.2. Chips may also be taken from the small ingots discussed in examples 1 and 2.

All the chips obtained are gathered and treated as described in clause 5.

The procedures described in the above examples are designed to obtain the desired degree of precision in the determination of the nickel content. In practice, they also serve as a simple method of obtaining sufficiently precise determinations of all the other elements to be analysed (see ISO 6501).

1) See ISO 6352.

2) In case of dispute, all the small ingots are cut, and the desired number of sound discs chosen and analysed.

Annex B
(informative)

Methods for taking a sample of size N in a supply of M items

B.1 General

It should be noted from the outset that in any method for drawing a sample from a population two stages can be distinguished:

a) the definition of the items to be sampled (ferronickel ingots or pieces);

b) the process of sampling itself.

It should also be recalled that in order to be representative a sample has to be drawn in such a way that any item of the sampled population has the same probability of being drawn.

B.2 Methods for defining the items constituting the sample

Two methods can be contemplated: one is random sampling of all the items of the sample; the other is systematic periodic sampling, only the first item being designated at random.

B.2.1 Random sampling of items

In this method, all possible samples of N items (or combinations of N objects taken from M) really have an equal probability.

Let us assume that the M items of the consignment bear some kind of identification which can be translated by a special numbering from 1 to M. The problem is then reduced to drawing N distinct integers at random among the first M integers.

To this purpose N random numbers shall first be selected from the uniform distribution in the interval 0 to 1. Some tables give such numbers directly. Others (such as table B.1) only give rows of numbers from 0 to 9, in random order, and real uniformly distributed numbers can easily be selected by taking an integer part equal to zero completed by a sequence of n decimals represented by n figures of the table.

Example:

Table B.2 is an extract from a table of random numbers which allows all the concrete cases to be found in this International Standard to be dealt with.

If numbers of the uniform distribution from 0 to 1 are needed with, say, five decimal places, groups of five digits shall be taken either by column or by row or by any other systematic means. Thus, by taking the first five digits of each line for example, the following sequences of digits are obtained:

10275
28415
34214
61817
etc.

and the numbers are: 0,102 75 — 0,284 15 — 0,342 14 — 0,618 17, etc.

NOTE — In table B.2, the spaces between rows and columns are only for improved readability of the table, which regroups digits from 0 to 9 in a random order.

Let $x_1, x_2, ..., x_N$ be a series of N numbers of the uniform distribution thus obtained. All these (real) numbers are multiplied by the integer M, which gives real numbers selected at random in the interval 0 to M.

$$Mx_1, Mx_2, ..., Mx_N$$

These real numbers are rounded up to the next highest integer:

$$E_1 = [Mx_1] + 1$$

$$E_2 = [Mx_2] + 1$$

$$...$$

$$E_N = [Mx_N] + 1$$

where $[Mx_i]$ = integer part of Mx_i.

The integers $E_1, E_2, ..., E_N$ then identify the items to be drawn from the population of M objects.

If this procedure results in drawing some equal numbers E_i, additional x_i numbers shall be drawn until N different values of E_i have been obtained.

B.2.2 Systematic periodic sampling of items

In this method, not all samples of N items constituted from M items of the supply have an equal probability of being obtained. Actually, this probability is zero for a very large number of them, although any specific item can have (at least approximately) the same probability of being part of the sample. This somewhat paradoxical result is explained by the non-independence of individual increments.

A whole quotient of M by N, i.e. Q, is calculated and, if the division gives a remainder R (less than N), it is neglected.