

Ferronickel shot — Sampling for analysis

The European Standard EN 28049 : 1992 has the status of a British Standard

Ferro-nickel en grenailles — Échantillonnage
pour analyse

Ferronickelschrott — Probenahme für Analyse

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National foreword

This British Standard has been prepared under the direction of the Non-ferrous Metals Standards Policy Committee and is the English language version of EN 28049 : 1992, 'Ferronickel shot — Sampling for analysis' published by the European Committee for Standardization (CEN). It is identical with ISO 8049 : 1988 published by the International Organization for Standardization (ISO).

Compliance with a British Standard does not of itself confer immunity from legal obligations.

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English version

Ferronickel shot — Sampling for analysis

(ISO 8049 : 1988)

Ferro-nickel en grenailles — Échantillonnage
pour analyse
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Ferronickelschrott — Probenahme für Analyse
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Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

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CEN

European Committee for Standardization
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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 8049 : 1988 'Ferronickel shot — Sampling for analysis' to the formal vote.

This European Standard EN 28049 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to this European Standard shall be published at the latest by 1992-11-30 and conflicting national standards shall be withdrawn at the latest by 1992-11-30.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 8049 : 1988 was approved by CEN as a European Standard without any modification.

Ferronickel shot — Sampling for analysis

1 Scope

This International Standard defines a method of sampling for analysis of ferronickel lots in the form of shot as specified in ISO 6501 in those cases where lots are constituted either heat by heat or by taking from blended stock.

The purpose is to determine the contents of the various elements :

- either from slugs by physical analysis methods (such as X-ray fluorescence or emission spectral analysis);
- or from chips by dry methods (carbon, sulfur) or chemical analysis (other elements).

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 — Specification and delivery requirements.*

3 Form and packaging of product

Grain size : between 2 and 50 mm

Lot tonnage : equal to or greater than 5 t

In the case of lots taken from blended stock, the nickel content range k to $(k + n)$ % of the blended heats shall be chosen as

$$\begin{aligned} 15 < k < 59 \\ 1 < n < 5 \\ 16 < k + n < 60^{1)} \end{aligned}$$

The ferronickel shot is generally delivered in bulk form in units which may be trucks, containers, or railroad cars, of which the contained masses normally range from 5 to 30 t, although in the case of railroad cars, loads may have masses up to 60 t.

This type of ferronickel can also be delivered drum-packed (the contained mass of which may be 250 kg).

4 Principle

In a single heat, intergrain homogeneity is practically ensured. It is therefore very easy to obtain a representative "primary sample" from a small number of "primary increments".

In the case of a blended lot composed of several heats, a greater number of primary increments N_p has to be taken, but the whole still constitutes the primary sample.

After blending and mass division of the primary sample, an "intermediate sample" is obtained having a reasonable mass for laboratory treatment. The treatment of the intermediate sample gives a "secondary sample", which may be divided in N_s "secondary increments" not exceeding a mass of 1 kg individually. Each secondary increment is then remelted under appropriate conditions so that no variation in composition can be observed and that N_s homogeneous small ingots²⁾ be obtained (within-small-ingot homogeneity).

1) The case of non-blended lots (case $n < 1$) is not dealt with in this International Standard.

2) It is generally accepted that 1 kg is the maximum mass which can be accommodated in a laboratory furnace for re-casting under the required conditions. According to the grain size distribution of shot, it is often necessary for the secondary sample to exceed 1 kg in order to be representative. Hence the necessity of melting several small ingots. See the statistical justification in annex A.

The small ingots are then either used for physico-chemical analysis or machined into chips for chemical analysis. (This procedure is summed up in figure A.1 of annex A.)

5 Taking of the primary sample and then of the intermediate sample

5.1 Blended lots

5.1.1 Bulk sampling in the case of a suitable system for taking the primary sample

This can be performed, for example, by emptying the shot into a bin with reclaim by belt conveyor. From the conveyor discharge, two possibilities are

- to have a true sampling system respecting the rules of the art for sampling of particulate material (such as a cross-stream sampler);
- to take increments at regularly spaced intervals, using a power shovel with a dipper intercepting the shot stream in a representative manner.

The mass of each primary increment shall be, in this case, not less than 20 kg, and is generally between 20 and 50 kg.

The number of primary increments N_p to be selected is shown in table 1.

The primary sample shall then be mass-divided into smaller units, in order to obtain an intermediate sample having a mass which can reasonably be sent to the laboratory for further preparation, 20 to 50 kg, say.

This can be accomplished with automatic mass dividers (such as rotary dividers) of suitable size with respect to the particle size of the product being handled.

Failing such equipment, the division can be made by alternate shovelling from the primary sample stockpile. As a precaution

against material spill during shovelling, it is recommended that a scoop or coal-miner's-type shovel be used.

For example, every fifth shovelful or less would be taken and this division would be repeated a sufficient number of times to obtain the desired sample mass of 20 to 50 kg.

5.1.2 Sampling of bulk material when no adequate primary sampling system is available

In this case, hand sampling shall be performed by alternate shovelling on each unit to be checked (truck, railroad car, container, etc.). The number of units to be checked is the number N_p in table 1 or the total number of units if it is less than N_p .¹⁾

For example, when unloading a 20 t truck on to the ground, sampling could proceed as follows :

- Shovel the 20 t, setting aside every fifth shovelful.
- From the 4 t obtained, set aside every fifth shovelful.
- From the 800 kg obtained, set aside every fifth shovelful.
- From the 160 kg obtained, set aside every fifth shovelful.
- Send the 32 kg obtained to the laboratory.

In this example, an intermediate sample is obtained for the checked unit.

If more than one unit is checked in the same lot, intermediate samples in each unit can be blended and mass division carried out again until an intermediate sample representative of the lot is obtained. In this case, the intermediate sample mass can be reduced to 10 to 20 kg.

5.1.3 Sampling of a drum-packed lot

The number of drums from which increments have to be taken is the number N_p in table 1 or the total number of drums if this is less than N_p .²⁾

Table 1 — Minimum number of primary increments to be selected

Numbers of primary increments N_p	Tonnage	Range of nickel contents n				
		$n < 1$	$1 \leq n < 2$	$2 \leq n < 3$	$3 \leq n < 4$	$4 \leq n \leq 5$
	5 to 50	5	10	15	20	30
	50 to 200	7	12	17	22	35
	200 to 500	10	15	20	25	40
	500 to 2 500	15	20	25	30	45
Number of secondary increments N_s ¹⁾		1	2	3	4	5

1) This indicates the number of small ingots to be remelted in the hypothesis of 1 kg per small ingot. (If the maximum mass which can be remelted is $1/x$ kg, the number of small ingots to be remelted is $x \cdot N_s$.)

1) For this purpose, the rules for random sampling given in annex B can be applied.

2) In general, drum-packaging is used for low-tonnage lots. The first line of the table is therefore applicable in most cases.

A minimum of 1 kg of shot or more, if required, per selected drum is taken to obtain a mass in excess of 20 kg, generally between 20 and 50 kg.

If the contents of each drum are assumed to be homogeneous, the sample may be taken from the top of the drum. If not, the drums shall be emptied and the sample taken by alternate shovelling.

5.2 Particular case of a lot made up of one single heat

As inter-grain homogeneity is ensured, it is sufficient to take the minimum quantity of material for small ingot remelting (1 kg for example).

To have more adequate guarantee, a small number of primary samples, for example 3 to 5, can be taken (either by bulk sampling or sampling from drums), then blended and mass-divided in order to obtain an intermediate sample of 5 to 10 kg.

If the lot is not assumed to be made up of a single heat, one of the procedures described in 5.1 shall be applied.

6 Treatment of the intermediate sample and taking of the secondary sample

This is generally carried out in the laboratory sampling shop.

6.1 Blended lot

The intermediate sample is blended, then mass-divided, preferably using a riffle divider of appropriate dimensions¹⁾ or, failing this, by alternate shovelling, until a mass equal to or slightly exceeding the mass, in kilograms, in the last line of table 1 is obtained. In the table, N_s is the number of small ingots to be remelted when 1 kg of material can be melted in one operation. (If melting is achieved by masses of $1/x$ kg, the number of small ingots to be remelted is $x \cdot N_s$.)

The mass defined by the rule above is the mass to be remelted and to be used for representative analysis. If a sampling reject or second unmelted secondary sample is to be kept, the corresponding quantity of material shall be set aside at the time of mass divisions.

6.2 Lot made up of a single heat

To be representative, a small ingot having a mass of 250 to 1 000 g shall be obtained. This is obtained by blending and mass division of the intermediate sample made in accordance with 5.2 until the mass required for remelting is obtained.

7 Remelting of the secondary sample

Remelting shall be performed in conditions such that no variation in content (of either Ni or the impurities to be checked) occurs either during melting or casting of the final sample (slugs, rondelles or small ingots).

In practice, the melting shall be done by induction heating in order to be carried out rapidly; it generally requires argon protection. The melted sample can be cooled and solidified in the melting pot itself, provided that argon protection is provided. However, it is much better to cast after melting by centrifuging. This ensures

- an excellent homogeneity of the sample produced as a result of mixing the molten metal during its injection into the mould;
- a uniform crystalline structure which fosters a good repeatability of the measurements for physical analysis methods. The argon protection should preferably be maintained during centrifuging.

It is recommended that a reagent (such as aluminium chips in a proportion of 1 to 2 g/kg) be introduced to kill the shot to be remelted. Naturally, the dilution undergone by the sample can be taken into account to correct the nickel content found during final analysis.

8 Use of small ingots (secondary increments)

8.1 The small ingots produced are truncated near their base to obtain a slice having a thickness of some 15 to 20 mm.

The slices obtained can be used for physical analysis and the average value of the analyses is calculated.

8.2 It is also possible to take chips by drilling or milling on the remaining parts of the small ingots. Chips coming from all the small ingots are conditioned for analysis by dry methods (sulfur and carbon) or chemical analysis (for the other elements).

8.2.1 Precautions for chip machining

Machining (and preferably milling) shall be carried out in such a way that chips cannot be contaminated (either by cutting tool wear or by dust or grease). In particular, the work shall be carried out under dry conditions.

For the detailed technical conditions of machining, see annex C.

Some ferronickel types are very hard, hence the need to select appropriate cutting tools and cutting conditions with great care.

Machining will generally be easier if the small ingot is previously annealed.

8.2.2 Treatment of chips

8.2.2.1 Washing

When surface contamination of chips (by lubricants, dust, etc., inevitably present when working with machine tools) is feared,

1) The colander width shall be at least three times the mean diameter of the largest shot.