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Ferronickel shot — Sampling for analysis

Ferro-nickel en grenailles — Échantillonnage pour analyse

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Contents

Page

Foreword

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The committee responsible for this document is ISO/TC 155, *Nickel and nickel alloys*.

This second edition cancels and replaces the first edition (ISO 8049:1988). The following change has been made: 5.1.4 has been added.

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 documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 513:2012, *Classification and application of hard cutting materials for metal removal with defined cutting edges — Designation of the main groups and groups of application*

3 Form and packaging of product

Grain size: between 3 mm 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 follows:

— $15 \leq k \leq 59$;

— $1 \leq n \leq 5$;

— $16 \leq k + n \leq 60$.

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

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 t 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, should 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 ingots be obtained (within-small-ingot homogeneity).

NOTE 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.)

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 as follows:

— 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 kg and 50 kg.

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

Table 1 — Minimum number of primary increments to be selected

Sample	Tonnage	Range of nickel contents, n				
		$n < 1$	$1 \leq n < 2$	$2 \leq n < 3$	$3 \leq n < 4$	$4 \leq n \leq 5$
Numbers of primary increments N_p	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[a]		1	2	3	4	5

[a] 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 1lx kg, the number of small ingots to be remelted is $x \times N_s$.)

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 kg 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 kg 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. For this purpose, the rules for random sampling given in Annex B may be applied.

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 kg to 20 kg.

5.1.3 Sampling of a drum-packed lot

The number of drums from which increments should be taken is the number N_p in Table 1 or the total number of drums if this is less than N_p.

NOTE 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 kg 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.

5.1.4 Sampling of a container-packed lot

5.1.4.1 Principle

This sampling method is applicable only for the determination of the nickel content (Ni).

The aim of this proposal is to simplify the sampling mode of a ferronickel delivery at customer site. Indeed generally,

— the end user does not have the appropriate means to proceed rigorously with this standard to sample the product, and

— when the end user gets the analytical results on the delivery, the lot is already partially or totally consumed, and consequently a further contradictory sampling is not possible.

This way of doing can be only used in the case of a blended lot which has been constituted with several heats (presenting different chemical analysis) stored in a big stand. When the stand is full, the homogenization of the stand shall be carried out and the parameters of exactness and reliability of the stand should be determined.

The exactness is the difference in Ni content between the first and the last heat.

The reliability is the biggest difference in Ni content between two heats.

The values of those both parameters will determine the way to go with the customer lot analysis:

— under certain values the customer lot analysis will be the one of the stand;

— above these values the customer lot will be sampled during the containers loading and the customer lot analysis will be the one of the representative sample of the customer lot.

5.1.4.2 Sampling method

If the conditions described previously are fulfilled for the stand, only one container (taken at random) of the customer lot can be sampled.

EXAMPLE To sample a 20 t container, sampling could proceed as follows:

— take a minimum of 16 portions of ~5 kg shots, largely scattered in the metal mass (into the container or spread on a clean ground), 8 at the surface and 8 inside the mass, to obtain a sample of approximately 80 kg;

— homogenize this sample using a suitable riffle divider (D62 is minimum) or by alternate shovelling;

— make successive divisions using a suitable riffle divider or by alternate shovelling to finally obtain two twin samples of ~5 kg to be packed in sealed plastic bags with lot reference labelling.

— one sample is provided to the laboratory for preparation and analysis, the second is kept for a possible other control.

Ni content is then determined with the appropriate analytical method and compared with the Ni content of the stand as follows:

— If $x - 3' \sigma_S < X_C < x + 3' \sigma_S$, the customer lot is in accordance with the supplier analysis certificate;

— If X_C is out of the interval, the customer lot is not in accordance with the supplier analysis certificate

where

X_C is the Ni content (obtained by the customer at lot reception) in the sampled container;

x is the Ni content of the supplier analysis certificate;

σ_S is the calculated standard deviation of Ni contents in the containers;

where

$$\sigma_s = \sigma_e \left(1 - \rho_h\right)$$

where

σ_e is the calculated standard deviation of Ni content in the heats constituting the stand;

ρ_h is the homogenization rate of the stand (determined by the supplier).

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 kg 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

6.1 General

This is generally carried out in the laboratory sampling shop.

6.2 Blended lot

The intermediate sample is blended, then mass-divided preferably using a riffle divider of appropriate dimensions 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 $x \times N_S$.)

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

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.

6.3 Lot made up of a single heat

To be representative, a small ingot having a mass of 250 g 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 the following:

— 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 g/kg 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 mm 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, it is strongly recommended that the chips be washed twice in pure acetone (or once in pure acetone and once in pure ether).

The solvent is drained off. Residual solvent is then evaporated in the air and the sample is dried for a minimum of 0,5 h in an oven maintained at 100 °C to 110 °C.

The use of pure organic solvents and their utmost removal is required for later determination of carbon and sulfur with automatic devices according to dry instrumental techniques.

8.2.2.2 Crushing

If chips come from a single small ingot, due to the fact that cast small ingots are very homogeneous, it is not necessary to crush the chips.

NOTE This is all the more valid the finer the chips. Millings are finer than drillings.

If several small ingots have been cast it is useful, when possible, to crush the chips in order to achieve homogeneity between the chips from various small ingots.

In practice, crushability depends on the following:

— the nickel content, if it exceeds 35 %, the alloy becomes ductile and is difficult to crush;

— the impurity contents (above all carbon): high-carbon ferronickels can be crushed much finer than low-carbon ferronickels.

In the case of crushable ferronickels, a suitable crusher shall be used which does not introduce contamination with iron. Vibration mill laboratory crushers used for a duration of 10 s to 30 s are suitable. It is desirable that the crushing container be of tungsten carbide or, if this is not possible, of special anti-wear steel (ball-type or bar-type crushers are not permissible).

In the case of ferronickels having nickel contents less than 35 %, 30 s crushing gives such fine material that almost all can be considered as undersize in case of sieving:

— on a sieve having a 2,5 mm aperture size (8 mesh), for low-carbon ferronickel (LC);

— on a sieve having a 0,8 mm aperture size (20 mesh) for medium-carbon ferronickels (MC) and high-carbon ferronickels (HC).

8.2.2.3 Homogenization and bottling

When the chips derive from several small ingots, it is necessary to achieve homogenization (using a mechanical homogenizer or repeated alternative shovelling, or several passes through a riffle divider keeping all the material, etc.).

The sample shall be subdivided in several portions using a riffle divider or a sample distributor. The number of fractions will depend on the required number of test samples for analysis to be kept by the interested parties.

The minimum distribution shall be the following:

— one for the purchaser,

— one for the vendor,

— one for the referee,

— one reserved.

For low-carbon ferronickels (LC), all handling operations shall be carried out so that no carbon contamination can occur (no contact with paper, cardboard, rubber, cork, or plastics; metallic materials and aluminium foils can be used).

The same care shall be exercised for bottling.

For medium-carbon ferronickels (MC) and high-carbon ferronickels (HC), samples can be stored in bottles of, for example, glass or aluminium, or in thick, heavy-quality polyethylene bags.

Annex A
(informative)

Justification of the number of primary and secondary increments

A.1 General

The reasoning below applies to blended lots.

The definition of the product is given in Clause 3.

The adopted procedure is derived from the following preliminary considerations:

a) excellent homogeneity within a granulated heat. No content variation is detected (for nickel and the various impurities: carbon, cobalt, chromium, sulfur, silicon) either among granules of the same particle size range or among the various particle size ranges within the same heat;

b) particle size distributions may vary considerably from one heat to another in a blended lot;

c) it is possible to remelt ferronickel shot under argon without varying the content of nickel, cobalt, chromium, silicon, sulfur. However, slight reductions in carbon contents have been observed.

In practice, the maximum known capacity of remelting furnaces is 1 kg and the numerical values of N_S in Table 1 have been chosen on this basis.

The study has been carried out mainly on nickel contents, which are the figures on which the greatest accuracy is sought.

A.2 Sampling scheme

The general principle adopted is summed up in Figure A.1.