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Ferronickel shot - Sampling for analysis (ISO 8049:1988)

Ferronickelschrot - Probenahme für Analyse (ISO 8049:1988)

Ferro-nickel en grenailles - Echantillonnage pour analyse (ISO 8049:1988)

**Ta slovenski standard je istoveten z: EN 28049:1992**

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## Ferronickel shot — Sampling for analysis

(ISO 8049 : 1988)

Ferro-nickel en grenailles — Échantillonnage  
pour analyse  
(ISO 8049 : 1988)

Ferronickelschrott — Probenahme für Analyse  
(ISO 8049 : 1988)

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### CEN

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

Central Secretariat: rue de Stassart 36, B-1050 Brussels

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to this European Standard shall be published at the latest by 1992-11-30 and conflicting national standards shall be withdrawn at the latest by 1992-11-30.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

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# 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

$$15 \leq k \leq 59$$

$$1 \leq n \leq 5$$

$$16 \leq k + n \leq 60^{1)}$$

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 ingots<sup>2)</sup> be obtained (within-small-ingot homogeneity).

1) The case of non-blended lots (case  $n \leq 1$ ) 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  $N_p$ .<sup>1)</sup>

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  $N_p$ .<sup>2)</sup>

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 dimensions<sup>1)</sup> 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.

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 to 110 °C.<sup>1)</sup>

#### 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.<sup>2)</sup>

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 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 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- and high-carbon ferronickels (MC and 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

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

1) 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.

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



## 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 of this International Standard.

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.

The following notation is used :

$N_p$  is the number of primary increments.

$V_p$  is the primary variance estimating the scatter of nickel contents among primary increments.

This variance therefore gives an estimate of the slight heterogeneity which may be observed in the whole lot when it is discharged at the purchaser's.

This quantity is by nature a variance of integration (zero if homogeneity is perfect among primary increments). It is generally not computable from a theoretical scheme. Only empirical observations can be made when the lots are discharged.

$N_s$  is the number of secondary increments. This is the number of remelted small ingots.

$V_s$  is the secondary variance estimating the scatter of nickel contents among secondary increments (mass  $\leq 1$  kg).

The fact that the material is made up of separate shot particles and that the heats selected within a defined range of nickel contents are blended means that a minimum mass of blended material must be kept for the secondary sample to be representative.

This quantity is by nature a fundamental variance [which always exists in any fragmented material undergoing content variations from fragments to fragments, even if homogenization (or blending) is perfect].

There are mathematical models for calculating this quantity. Their application to ferronickel lots generally implies keeping a mass in excess of 1 kg. This is the main reason why several small ingots are remelted and why  $N_s$  and  $V_s$  are to be evaluated.

$V_e$  is the sampling variance.

We have

$$V_e = \frac{V_p}{N_p} + \frac{V_s}{N_s} \quad \dots (1)$$

NOTE — The intermediate sample is not taken into consideration in the procedure principle. It is only one sample among all the others handled during blending and mass-division operations. It is to be selected with a reasonable mass for transportation between the primary sampling location at the works and the laboratory.

If one analysis is carried out per remelted sample ingot,

$$V_{Ni} = \frac{V_p}{N_p} + \frac{V_s}{N_s} + \frac{V_r}{N_s} + V_A + V_L \quad \dots (2)$$

where

$V_{Ni}$  is the variance of nickel content over all "sampling and analysis" operations when several laboratories are compared;

$V_r$  is the variance of analytical repeatability<sup>1)</sup>;

$V_A$  is the variance of inter-analyst fluctuation (analytical)<sup>1)</sup>;

$V_L$  is the variance of inter-laboratory fluctuation (analytical)<sup>1)</sup>.

1) For the detailed significance of  $V_r$ ,  $V_A$ ,  $V_L$ , see annex B of ISO 6352.

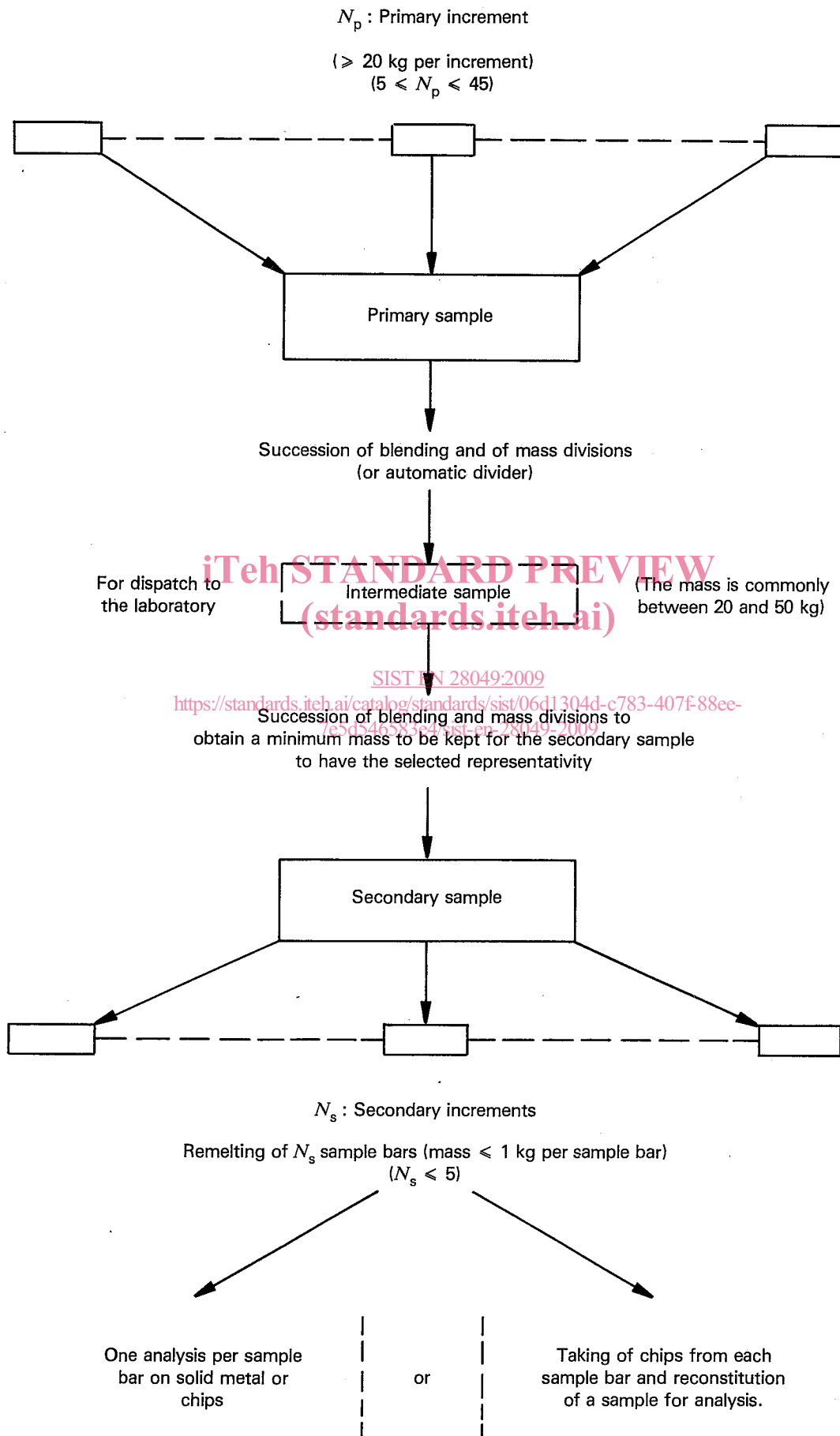


Figure A.1 — General scheme of increments

### A.3 Estimate of primary variance

Observations have been made by some producers either at dispatch from the manufacturing plant or at the time of delivery at the purchaser's.

Even in the case  $n = 5$  (maximum range of nickel content), very low values of  $V_p$  have always been observed.

The value  $V_p = 0,01$  has been retained for  $n = 5$ , which is noticeably in excess of any collected concrete observations. It can therefore be considered as a safe value.

Moreover, it has been assumed that the upper limit adopted for  $V_p$  could be reduced when  $n$  was reduced.

Again a slight diminution of the value of  $S_p = \sqrt{V_p}$  has been admitted by prudence, according to table A.1.

Table A.1 — Variation of  $S_p$

$n$	$S_p = \sqrt{V_p}$	$V_p$
1	0,06	0,003 6
2	0,07	0,004 9
3	0,08	0,006 4
4	0,09	0,008 1
5	0,10	0,010 0

$M$  is the total number of shot particles in the lot;

$N$  is the number of shot particles in the primary sample;

$\alpha$  is the coefficient, the numerical values of which have been calculated under three hypothetical conditions of nickel content distribution within the range, as follows :

Hypothesis 1 (most unfavourable case) : Half of the heats are at the lower limit  $(k - \varepsilon) \%$  and the other half at the upper limit  $(k + n + \varepsilon) \%$  :  $\alpha = 4$

Hypothesis 2 (less unfavourable case) : The contents, heat after heat, are uniformly distributed between the two limits :  $\alpha = 12$

Hypothesis 3 (more favourable case) : Normal (Gaussian) distribution of the mean in the middle of the range and the standard deviation at 1/6th of the range :  $\alpha = 36$

In practice, a producer tries to centre his production of shot on a desirable content for the heats intended for constituting the blended lot; the hypothesis of a normal distribution is then logical. For shot, hypothesis 2 remains consequently an unfavourable hypothesis.

### A.4 Estimate of secondary variance

The main conclusions drawn from a mathematical model for the estimation of this fundamental variance are :

**A.4.1** The fact that heats with different particle size ranges and nickel content are blended has no effect on the value of the fundamental variance if no correlation is found between the nickel content and the mass of each shot particle.

#### A.4.2 Estimation of the between-heats variance $V_c$

This is the variance which estimates the variations due only to the difference in nickel contents between heats (without taking into account the granular nature of the material).<sup>1)</sup>

This variance is of the form

$$V_c = \frac{(n + 2\varepsilon)^2}{\alpha} \times \frac{M - N}{M - 1} \quad \dots (3)$$

where

$\varepsilon$  is the numerical value expressing the uncertainty in the determination of the nickel content of a heat, which means that the range defined by  $k$  to  $(k + n) \%$  as planned to constitute the blended batch may have to be extended in practice from  $(k - \varepsilon)$  to  $(k + n + \varepsilon) \%$ . The theoretical range is  $n$ . The practical range is  $n + 2\varepsilon$ ;

#### A.4.3 Estimation of the required secondary sample mass for the desired uncertainty to be achieved (fundamental error)

The number  $N$  of shot particles to be taken for achieving an uncertainty characterized by standard deviation  $s_s$  is given by the formula :

$$N = \frac{(1 + \varrho_m^2) V_c}{s_s^2} + \varrho_m^2 \quad \dots (4)$$

in which parameter  $\varrho_m$  represents the coefficient of variation of shot mass  $m$ . This parameter, which would disappear if all shot particles had the same mass ( $\varrho_m = 0$ ), is used to account for the fact that the sampling variance depends on the product particle size heterogeneity. The presence of larger particles beside smaller particles has an unfavourable influence on the fundamental variance for a given total number  $N$  of particles in the sample.

The log-normal representation of the particle size distribution curve (i.e. of the cumulative proportion of undersize — or oversize — on a normal scale versus particle size on a logarithmic scale) is used to evaluate the quantity  $(1 + \varrho_m^2)$  as well as the quantities  $\tilde{m}$  and  $\sigma_{\ln m}^2$  which appear in the following equation for mean particle mass.

$$E(m) = \tilde{m} \exp \left( - \frac{\sigma_{\ln m}^2}{2} \right) \quad \dots (5)$$

1) This is the same case as for the sampling of ferronickels in ingots or pieces as dealt with in ISO 8050.

2) In  $m$  is the natural logarithm of  $m$ .