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Methods of sampling for chemical
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(Part 4 : Ferronickel)

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at the Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently **JIS G 1604 : 1985** is replaced with **JIS G 1604 : 2000**.

In this revision, **ISO** Standards have been employed as Annexes 1, 2 and 3 for the purpose of conforming to the International Standards.

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Methods of sampling for chemical analysis of ferroalloys (Part 4 : Ferronickel)

Introduction This Standard is the Japanese Industrial Standard, whose Annex 1 and Annex 2 are prepared based on **ISO 8050** *Ferronickel ingots or pieces—Sampling for analysis*, published in 1988, and Annex 3 is prepared based on **ISO 8049** *Ferronickel shot—Sampling for analysis*, published in 1988, without modifying the technical contents.

The underlined parts with dots in these Annexes show the matters not specified in the corresponding International Standards.

1 Scope This Standard specifies the sampling methods of ferronickel in ingots, pieces or shot form or the sampling method of ferronickel from molten metal, with a view to obtaining a representative laboratory sample for physical analysis as fluorescent X-ray analysis, emission spectral analysis and so on, carbon and sulfur contents analysis by dry-type method, or chemical analysis method for other components.

Remarks: The International Standards corresponding to this Standard are listed as follows.

ISO 8049 : 1988 *Ferronickel shot—Sampling for analysis*

ISO 8050 : 1988 *Ferronickel ingots or pieces—Sampling for analysis*

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. If the indication of the year of publication is given to these referred standards, only the edition of indicated year constitutes the provision of this Standard but the revision and amendment made thereafter are not applied. The normative references without the indication of the year of publication apply limiting to the most recent edition (including amendment).

JIS G 1326 *Method for chemical analysis of ferronickel*

Remarks : **ISO 6352** : 1985 *Ferronickel—Determination of nickel content—Dimethylglyoxime gravimetric method* is identical with a part of the said standard.

JIS G 2316 *Ferronickel*

Remarks : **ISO 6501** : 1988 *Ferronickel—Specification and delivery* is identical with a part of the said standard.

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*

3 Types of sampling methods The types of sampling methods of ferronickel shall be as given in Table 1.

Table 1 Sampling methods of ferronickel

Sampling method	Scope	Annex (normative) number
Sampling method in ingot or piece form	Sampling from ingot or piece form	Annex 1
Sampling method from molten metal	Sampling from molten metal	Annex 2
Sampling method in shot form	Sampling from shot form	Annex 3

Annex 1 (normative)

Sampling methods from ingots or pieces

Introduction This annex is prepared based on **ISO 8050 Ferronickel ingots or pieces—Sampling for analysis**, published in 1988 without modifying the technical contents.

The underlined part with dots in this Annex shows the matters not specified in the corresponding International Standard.

1 Scope This Annex 1 specifies methods for sampling of ferronickel lots in ingot or piece form with a view to obtaining a representative laboratory sample for the determination of chemical composition of the lot.

The sampling methods shall be applied to the lot delivered to the purchaser's yard⁽¹⁾. There are two procedures (drilling and milling).

Buyer and purchaser (or the representative) are entitled to participate in sampling operations.

Note ⁽¹⁾ This sampling procedure may 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 Normative references The following standards contain provisions which, through reference in this Annex, constitute provisions of this Annex. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Annex 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 Sampling

3.1 Sampling of ingots or pieces

3.1.1 Lots comprising one heat Take five ingots⁽²⁾ from one heat and follow the procedure as described in 3.1.3⁽²⁾⁽³⁾ and the follows.

Informative reference : One heat means one ladle of molten material to be drawn up to cast into the ingot or the like.

Notes ⁽²⁾ The rules for random sampling given in Annex 1 Informative reference A.

- (³) A very small number of ingots constitutes a representative sample of the heat, because the contents of different ingots from the same heat do not vary greatly (see Annex 1 Informative reference B).

3.1.2 Lots comprising several heats The minimum number N of ingots or pieces to be taken is given by the following rules(⁴).

For lot tonnages between 5 t and 80 t

$$N = 50$$

For lot tonnages between 80 t and 500 t

$$N = 54 - (T/20) \quad [\text{see Note } (^5)]$$

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

The relations between tonnage of lot and number N are shown in Annex 1 Table 1.

Annex 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. The procedure given in Annex 1 Informative reference A is recommended.

Notes (⁴) 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 1 Informative reference B.

- (5) 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 t 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.

3.1.3 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.

3.2 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 1 Informative reference C.

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 1 Informative reference C clause C.2.)

3.2.1 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 mm and 20 mm in diameter is recommended; drills in the 15 mm to 17 mm range are most frequently used.

Remarks : Examples of suitable drills, and the conditions under which they should be used, are given Annex 1 Informative reference C.

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 Annex 1 Fig. 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.

3.2.2 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.

Remarks : Examples of suitable milling cutters, and the conditions under which they should be used, are given in Annex 1 Informative reference C.

The chips obtained either by drilling or milling shall have a mass of at least 1 kg. These chips constitute the secondary sample which shall be treated as described in clause 4.

4 Treatment of chips The secondary sample is constituted of chips obtained as described in 3.2.

4.1 Washing It is 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 °C to 110 °C⁽⁶⁾.

Note ⁽⁶⁾ 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.

4.2 Crushing Sample chips from a single heat (see 3.1.1) need not be crushed because no problem of homogenization of the sample exists. They may therefore be treated directly as in 4.3 and after.

For sample chips from lots comprising more than one heat (see 3.1.2), 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 1 Informative reference C). 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 can be crushed, this shall be done with a suitable crusher that does not introduce iron contamination. Laboratory vibratory crushers do this in 10 s 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.

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

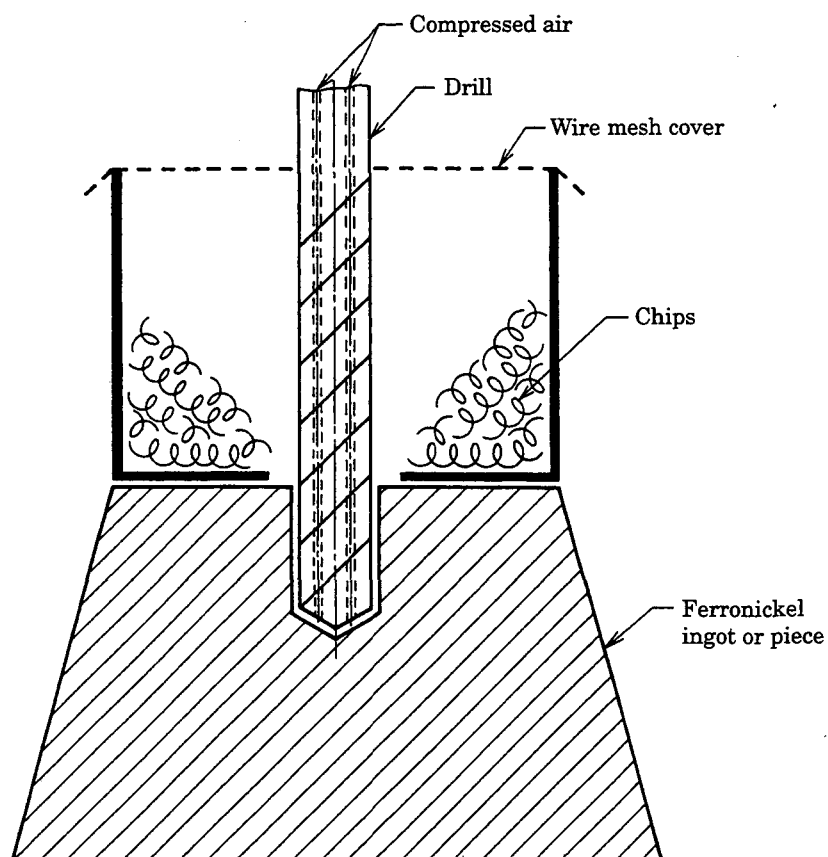
4.4 Division The sample shall be divided into 100 g portions using a riffle divider or a rotary sampler.

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.



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

Annex 1 Fig. 1 Collection box for drilling chips

Annex 1 Informative reference A
Methods for taking a sample of size N
in a supply of M items

A.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.

A.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 by random starting.

A.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 purposes 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 Annex 1 Informative reference A Table 2) 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:

Annex 1 Informative reference A Table 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.

Remarks : In Annex 1 Informative reference A Table 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 $x_1, x_2, \dots, x_N$ 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 .

$$Mx_1, Mx_2, \dots, Mx_N$$

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 $E_1, E_2, \dots, E_N$ 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 E_i have been obtained.

A.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.

An integer is then chosen at random in the sequence 1, 2, ..., $Q - 1, Q$, for example by the method described in A.2.1. Let this number be H . The items composing the sample are then defined by integers:

$$H, Q + H, 2Q + H, \dots, (N - 1)Q + H$$

It can be seen that by this method $M - NQ$ items are ignored by sampling but that only one drawing is necessary from the table of random numbers.

Because of the unequal probability of all possible samples of N items being drawn, it is also necessary to specify that the theoretical formulae for calculating the sampling variance do not apply in this case, except if the lot of items has been mixed carefully, which is hardly practicable.

A.3 Sorting out of the N ingots or pieces identified N ingots or pieces having, in theory, been identified by the integers $E_1, E_2, \dots, E_N$ among M items constituting the lot, the physical operation of sampling remains to be carried out without losing sight of the fact that the ingots or pieces generally do not bear an identification mark.

Two cases can be considered: the consignment to be sampled is either in bulk or it is physically split up into pallets, lorries, wagons, etc.

A.3.1 Case of a consignment delivered in bulk It is widely recognized that a consignment delivered in bulk can be sampled correctly only if all its elements can be moved.

When doing this, it will be possible to separate the ingots or pieces coming with the numbers $E_1, E_2, \dots, E_N$ to make up the primary sample needed.

A.3.2 Case of a consignment split up into sub-lots or sub-assemblies In this case, it is possible to avoid moving all the ingots or pieces in the consignment through a prior identification of the sub-assemblies (pallets, or wagons) from which ingots or pieces should be picked out.

To do this, a list of the sub-assemblies indicating the number of ingots or pieces in each of them is drafted, and the cumulative number of ingots or pieces in these batches when they appear in succession computed, as shown in the third column of Annex 1 Informative reference A Table 1 as an example.

Let the numbers $E_1, E_2, \dots, E_N$ which designated the ingots or pieces and which were previously defined by one of the methods indicated in clause A.2 be, for example:

110, 132, 167, 404, 489, 827, 859, 959, 1 109, 1 288,

then it will be easy, by comparison of these numbers with the cumulative numbers of sub-assemblies in the sequence of batches, to determine the particular sub-assembly from which to take each ingot or piece of the sample.

It may happen that no ingot or piece need be taken from some batches (B and D in our example). With important consignments made up of a large number of sub-lots, this may be the case for most of them, especially if the sample size is small. This will result in a substantial saving in handling operations.

The ingots or pieces so identified will be sampled from their respective sub-assemblies by the method described in A.3.1.

Annex 1 Informative reference A Table 1 Cumulative number of ingots or pieces

Sub-assembly	Number of ingots or pieces	Cumulative number of ingots or pieces	Order number of ingots or pieces to be taken from sub-assemblies
A	200	200	110, 132, 167
B	200	400	
C	150	550	404, 489
D	250	800	
E	250	1 050	827, 859, 959
F	150	1 200	
G	100	1 300	1 109
			1 288

Annex 1 Informative reference A Table 2 Table of random numbers

10 27 53 96 23	71 50 54 36 23	54 31 04 82 98	04 14 12 15 09	26 78 25 47 47
28 41 50 61 88	64 85 27 20 18	83 36 36 05 56	39 71 65 09 62	94 76 62 11 89
34 21 42 57 02	59 19 18 97 48	80 30 03 30 98	05 24 67 70 07	84 97 50 87 46
61 81 77 23 23	82 82 11 54 08	53 28 70 58 96	44 07 39 55 43	42 34 43 39 28
61 15 18 13 54	16 86 20 26 88	90 74 80 55 09	14 53 90 51 17	52 01 63 01 59
91 76 21 64 64	44 91 13 32 97	75 31 62 66 54	84 80 32 75 77	56 08 25 70 29
00 97 79 08 06	37 30 28 59 85	53 56 68 53 40	01 74 39 59 73	30 19 99 85 48
36 46 18 34 94	75 20 80 27 77	78 91 69 16 00	08 43 18 73 68	67 69 61 34 25
88 98 99 60 50	65 95 79 42 94	93 62 40 89 96	43 56 47 71 66	46 76 29 67 02
04 37 59 87 21	05 02 03 24 17	47 97 81 56 51	92 34 86 01 82	55 51 33 12 91
63 62 06 34 41	94 21 78 55 09	72 76 45 16 94	29 95 81 83 83	79 88 01 97 30
78 47 23 53 90	34 41 92 45 71	09 23 70 70 07	12 38 92 79 43	14 85 11 47 23
87 68 62 15 43	53 14 36 59 25	54 47 33 70 15	59 24 48 40 35	50 03 42 99 36
47 60 92 10 77	88 59 53 11 52	66 25 69 07 04	48 68 64 71 06	61 65 70 22 12
56 88 87 59 41	65 28 04 67 53	95 79 88 37 31	50 41 06 94 76	81 83 17 16 33
02 57 45 86 67	73 43 07 34 48	44 26 87 93 29	77 09 61 67 84	06 69 44 77 75
31 54 14 13 17	48 62 11 90 60	68 12 93 64 28	46 24 79 16 76	14 60 25 51 01
28 50 16 43 36	28 97 85 58 99	67 22 52 76 23	24 70 36 54 54	59 28 61 71 96
63 29 62 66 50	02 63 45 52 38	67 63 47 54 75	83 24 78 43 20	92 63 13 47 48
45 65 58 26 51	76 96 59 38 72	86 57 45 71 46	44 67 76 14 55	44 88 01 62 12
39 65 36 63 70	77 45 85 50 51	74 13 39 35 22	30 53 36 02 95	49 34 88 73 61
73 71 98 16 04	29 18 94 51 23	76 51 94 84 86	79 93 96 38 63	08 58 25 58 94
72 20 56 20 11	72 65 71 08 86	79 57 95 13 91	97 48 72 66 48	09 71 17 24 89
75 17 26 99 76	89 37 20 70 01	77 31 61 95 46	26 97 05 73 51	53 33 18 72 87
37 48 60 82 29	81 30 15 39 14	48 38 75 93 29	06 87 37 78 48	45 56 00 84 47
68 08 02 80 72	83 71 46 30 49	89 17 95 88 29	02 39 56 03 46	97 74 06 56 17
14 23 98 61 67	70 52 85 01 50	01 84 02 78 43	10 62 98 19 41	18 83 99 47 99
49 08 96 21 44	25 27 99 41 28	07 41 08 34 66	19 42 74 39 91	41 96 53 78 72
78 37 06 08 43	63 61 62 42 29	39 68 95 10 96	09 24 23 00 62	56 12 80 73 16
37 21 34 17 68	68 96 83 23 56	32 84 60 15 31	44 73 67 34 77	91 15 79 74 58
14 29 09 34 04	87 83 07 55 07	76 58 30 83 64	87 29 25 58 84	86 50 60 00 25
58 43 28 06 36	49 52 83 51 14	47 56 91 29 34	05 87 31 06 95	12 45 57 09 09
10 43 67 29 70	80 62 80 03 42	10 80 21 38 84	90 56 35 03 09	43 12 74 49 14
44 38 88 39 54	86 97 37 44 22	00 95 01 31 76	17 16 29 56 63	38 78 94 49 81
90 69 59 19 51	85 39 52 85 13	07 28 37 07 61	11 16 36 27 03	78 86 72 04 95
41 47 10 25 62	97 05 31 03 61	20 26 36 31 62	68 69 86 95 44	84 95 48 46 45
91 94 14 63 19	75 89 11 47 11	31 56 34 19 09	79 57 92 36 59	14 93 87 81 40
80 06 54 18 66	09 18 94 06 19	98 40 07 17 81	22 45 44 84 11	24 62 20 42 31
67 72 77 63 48	84 08 31 55 58	24 33 45 77 58	80 45 67 93 82	75 70 16 08 24
59 40 24 13 27	79 26 88 86 30	01 31 60 10 39	53 58 47 70 93	85 81 56 39 38
05 90 35 89 95	01 61 16 96 94	50 78 13 69 36	37 68 53 37 31	71 26 35 03 71
44 43 80 69 98	46 68 05 14 82	90 78 50 05 62	77 79 13 57 44	59 60 10 39 66
61 81 31 96 98	00 57 25 60 59	46 72 60 18 77	55 66 12 62 11	08 99 55 64 57
42 88 07 10 05	24 98 65 63 21	47 21 61 88 32	27 80 30 21 60	10 92 35 36 12
77 94 30 05 39	28 10 99 00 27	12 73 73 99 12	49 99 57 94 82	96 88 57 17 91
78 83 19 76 16	94 11 68 84 26	23 54 20 86 85	23 86 66 99 07	36 37 34 92 09
87 76 59 61 81	43 63 64 61 61	65 76 36 95 90	18 48 27 45 68	27 23 65 30 72
91 43 05 96 47	55 78 99 95 24	37 55 85 78 78	01 48 41 19 10	35 19 54 07 73
84 97 77 72 73	09 62 06 65 72	87 12 49 03 60	41 15 20 76 27	50 47 02 29 16
87 41 60 76 83	44 88 96 07 80	83 05 83 38 96	73 70 66 81 90	30 56 10 48 59

Annex 1 Informative reference B**Formulae giving the number N of ingots or pieces to be taken to form the primary sample**

B.1 General The mathematical reasoning and numerical samples are based on nickel content determination. In practice, the accuracy is sufficient for the determination of the contents of the other elements.

The variance V attached to nickel determination includes a sampling component and an analysis component:

$$V = V_e + \frac{V_a}{a} \dots\dots\dots (1)$$

where,

V_e is the variance due to sampling;

V_a is the variance due to analysis;

a is the number of analyses.

The sampling variance may be factorized as follows:

$$V_e = \frac{1}{N} \times \left(\frac{V_f}{r} + V_1 + V_c \right) \dots\dots\dots (2)$$

where,

N is the number of ingots or pieces taken;

r is the number of sampling points per ingot or piece (number of drilled holes for drilling or number of milled faces for milling);

V_f is the variance translating the fluctuation in nickel content within an ingot or piece (variance within ingot or piece);

V_1 is the variance translating the fluctuations in nickel content between ingots or pieces from the same heat (variance between ingots or pieces or within heat);

V_c is the variance translating the fluctuations in nickel content between the various heats composing a lot (variance between heats).

In practical cases, the fluctuations between heats occur within a range of $(k - \varepsilon)$ to $(k + 1 + \varepsilon)$, k being an integer and ε the uncertainty of nickel determination for each heat when the heats are classified in ranges from k to $(k + 1)$ % Ni.

In numerical applications, ε has been chosen as 0.15 %. The fluctuation range then covers a content range of 1.30 % amplitude. This figure is larger than reality in all cases but preserves the representativity of the primary sample.

B.2 Calculation of V_c V_c can be calculated in a purely mathematical way in well-defined cases if it is considered that sampling N ingots or pieces at random is performed exhaustively (drawing without replacement) among the M ingots or pieces in the lot. Calculation leads to the following formulae in two well-defined cases.

B.2.1 Most unfavourable case The lot is composed of two heats, the first having a nickel content $(k - \varepsilon)$ and the second $(k + 1 + \varepsilon)$:

$$V_c = \frac{(1+2\varepsilon)^2}{4} \times \frac{M-N}{M-1} \dots\dots\dots (3)$$

B.2.2 Middle case A lot is composed of heats having regularly distributed nickel contents between $(k - \varepsilon)$ and $(k + 1 + \varepsilon)$:

$$V_c = \frac{(1+2\varepsilon)^2}{12} \times \frac{M-N}{M-1} \dots\dots\dots (4)$$

i.e. for V_c a value one-third of the preceding case.

In the case where $N/M \leq 0.10$ no significant error is introduced by assuming non-exhaustive drawing, i.e. replacing the term

$$\frac{M-N}{M-1}$$

by unity because such an approach can only increase the value of V_c :

With $\varepsilon = 0.15$, we obtain

$V_c = 0.42$ in the first case,

$V_c = 0.14$ in the second case.

B.3 Estimation of V_f and V_1 These are experimental observations made on heats at the producer's plant. The numerical values lie in the range from 0.005 to 0.05 (a maximum value encountered by exception only).

B.4 Value of V_e Terms V_f and V_1 are small with regard to V_c . And it may be concluded that

- it is useless to choose several sampling points per ingot or piece. $r = 1$ is therefore chosen;
- the value of V_e essentially results from the variance between heats and from the number N of ingots or pieces taken at random.

In practice, it is known that $V_a \cong 0.01$, which corresponds to a standard deviation of 0.1 % for individual determinations (in content percentage points).

To obtain $\alpha = 1$ and $V_e = V_a = 0.01$, with the numerical values mentioned above for V_c , V_1 and V_f , the following is to be chosen for N :

$N = 50$ in the most unfavourable case;

$N = 20$ in the middle case.

When α is greater than 1; the value V_a/α is decreased and moreover if the importance of V_e with regard to V_a/α is to be minimized, the above value of N can be increased. It is, however, to be noted that experimental cost is larger when N is doubled than when α is doubled, whereas the effect on the determination of V is the same when V_e is equal to V_a before N is doubled.

B.5 Value of N according to the lot size

B.5.1 Lot comprising a single heat It is known that the variances within and between ingots are very low and in this case V_c is nil. By choosing $N = 5$, one is certain to be very prudent and to have $V_e < 0.01$ with certainty.

B.5.2 Lot of tonnage between 20 t and 80 t These lots are composed of 2 to 5 cases of 16 t to 20 t each.

For the sake of prudence, the most unfavourable case is applied. One has thus $N = 50$.

B.5.3 Lot between 80 t and 500 t (maximum tonnage) In the case of 500 t, there is a strong probability of being in the middle case described above or in a more favourable case. It is then logical that the number of ingots or pieces to be taken is progressively decreased when the lot tonnage progressively increases from 80 t to 500 t.

This gives the equation

$$N = 55 - \frac{T}{15}$$

where T is the tonnage of the lot.

As a further measure of caution, the following equation has finally been adopted:

$$N = 54 - \frac{T}{20}$$

which slightly increases the values of N .

Remarks : In principle, the tonnage of a lot should not be greater than 500 t (see **ISO 6501**). If by special agreement between the purchaser and the supplier a consignment is between 500 t and 1 000 t, it is evident that the above formula no longer applies because for 1 000 t, $N = 4$ is obtained, which is less than the value for a single heat.

In fact, the conditions of 20 t heat blending are practically the same for 500 t (25 heats) or for 1 000 t (50 heats) and it may then be considered that one is constantly in the mean case as described in **B.2.2**.

It can then be considered that the number of ingots or pieces to be taken is constant ($N = 29$) from 500 t to 1 000 t.

Two procedures may therefore be applied in practice:

- a division of the consignment in lots of tonnages not exceeding 500 t;
- the drawing of $N = 29$ ingots or pieces from the whole consignment.

It is evident that the second solution notably decreases the quantity of work for chip taking from ingots or pieces while ensuring the same accuracy provided that analyses are repeated on the single lot so that the analytical uncertainty does not exceed the uncertainty obtained when analysing two or more lots.

B.6 Conclusions For a single heat, it is generally enough to take 2 to 4 ingots or pieces for them to be representative. By adopting $N = 5$, one is sure to be very prudent.

For a blend of several heats, the uncertainty of primary ingots or piece sampling is related almost uniquely to the range in which the heats are classified to compose the lot and to the number N of ingots or pieces being taken.

In the three examples in **B.5.1**, **B.5.2**, and **B.5.3**, recommended sampling is sufficiently prudent to guarantee $V_e < 0.01$.

It remains true that if the analysis itself is repeated several times and if it is required that $V_e \leq V_a/a$, it might be required that the values of N as given above be increased.

It is finally of importance to specify that the reasoning and conclusions above are valid if sampling of N ingots or pieces among the M of the lot is carried out on a random sampling basis (see Annex 1 Informative reference A).