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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Ferronickel ingots or pieces — Sampling for analysis

Ferro-nickel en lingots ou en morceaux — Échantillonnage pour analyse

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 8050 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys.*

Annex A forms an integral part of this International Standard. Annexes B, C and D are for information only.

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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.

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.

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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.

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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.

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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.

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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.

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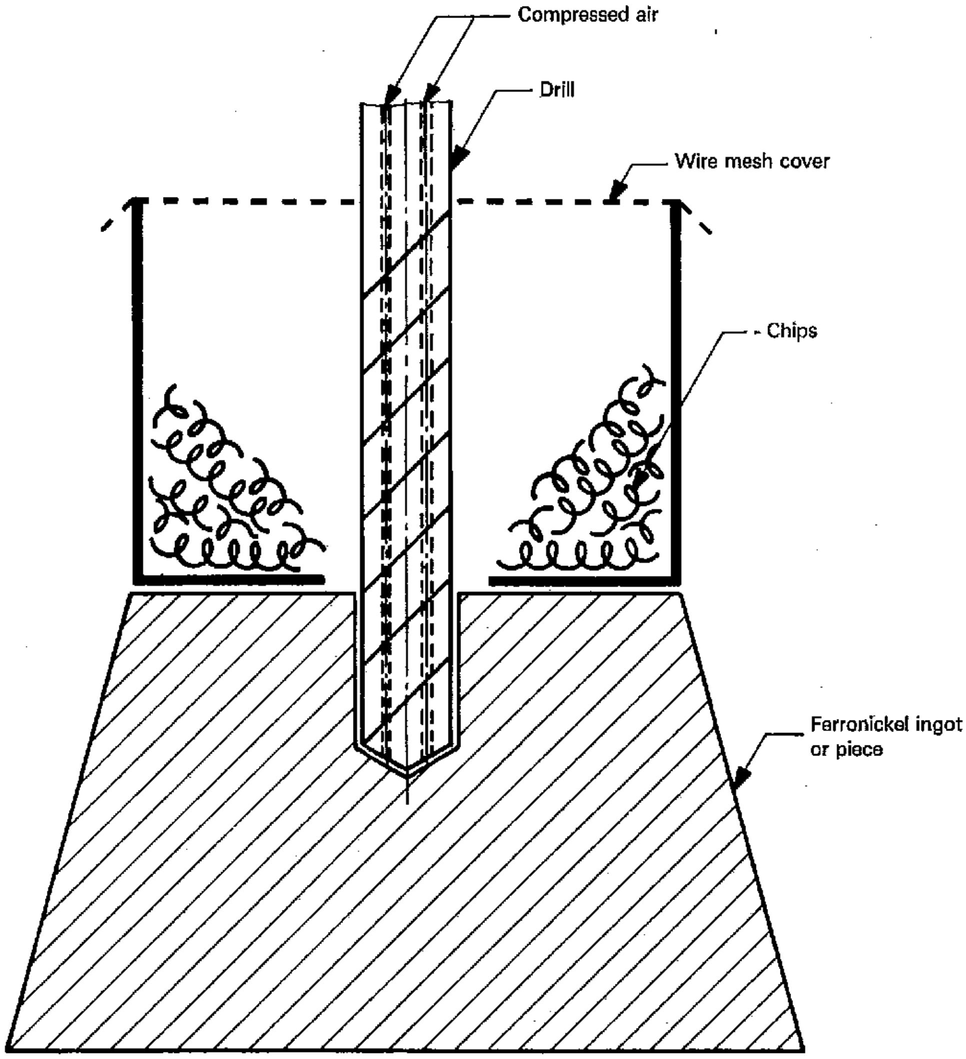

Figure 1 — Collection box for drilling chips

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

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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.

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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.

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

$$H, Q + H, 2Q + H, ..., (N - 1)Q + H$$

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.

B.3 Sorting out of the N ingots or pieces identified

N ingots or pieces having, in theory, been identified by the integers $E_1, E_2, ..., E_N$ 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.

B.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 $E_1, E_2, ..., E_N$ to make up the primary sample needed.

B.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 table B.1 as an example.

Let the numbers $E_1, E_2, ..., E_N$ which designated the ingots or pieces and which were previously defined by one of the methods indicated in clause B.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 B.3.1.

Table B.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	1 109
G	100	1 300	1 288

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Table B.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

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Annex C
(informative)

Formulae giving the number N of ingots or pieces to be taken to form the primary sample

C.1 General

The mathematical reasoning and numerical examples 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:

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

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:

$$V_e = \frac{1}{N} \times \left(\frac{V_f}{r} + V_l + V_c\right) \qquad \ldots (2)$$

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 fluctuations in nickel content within an ingot or piece (variance within ingot or piece);

V_l 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.

C.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.

C.2.1 Most unfavourable case

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

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

C.2.2 Middle case

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

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

i.e. for V_c a value three times less than in the preceding case.

In the case where $N/M < 0{,}10$ 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.

C.3 Estimation of V_f and V_l

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).

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C.4 Value of V_e

Applying equation (2) shows that terms V_f and V_i are small with regard to V_c. 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 $V_a \approx 0{,}01$, which corresponds to a standard deviation of 0,1 % for individual determinations (in content percentage points).

To obtain $a = 1$ and $V_e = V_a = 0{,}01$, with the numerical values mentioned above for V_c, V_i 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 a is greater than 1; the value V_a/a is decreased and moreover if the importance of V_e with regard to V_a/a is to be minimized, the above values of N can be increased. It is, however, to be noted that experimental cost is larger when N is doubled than when a 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.

C.5 Value of N according to the lot size

C.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.

C.5.2 Lot of tonnage between 20 and 80 t

These lots are composed of 2 to 5 casts of 16 to 20 t each.

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

C.5.3 Lot between 80 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 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.

NOTE — 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 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 C.2.2.

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

C.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 ingot 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 C.5.1, C.5.2, and C.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 $V_e < V_a/a$, 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 B).

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Annex D
(informative)

Technical conditions for drilling and milling

D.1 General

The hardness of ferronickel ingots or pieces may vary considerably, depending on the grades (nickel content) and especially on the quantity of impurities present (mainly C and Si).

The ingot or piece must be considered very hard when it falls between 180 and 600 in Vickers hardness (or equivalent hardness scales).

The cutting tools used and the conditions under which they are used must be carefully selected. Cutting is extremely difficult, because the work must always be done dry to avoid any contamination.

NOTE — Machining by drilling or milling are the most common procedures. Shaping with planing tools meeting the general requirements below is also possible. Work is then done on cut-off faces as in milling. This type of machining is, however, slower.

D.2 Case of very hard ferronickel

When machining is very difficult and involves tool wear and subsequent significant chip contamination or even impossible sampling, it is advisable to heat treat (temper) the material. The practical procedure is dependent on the metal hardness and crystalline structure. For guidance, tempering is generally useful when hardness exceeds 180 Vickers. Tempering may be carried out on ingots, on cut-off ingots parts or on pieces, as follows:

The ingots, ingots parts or pieces are placed in a furnace at 650 to 800 °C for 2 to 24 h. The furnace is then turned off and allowed to cool slowly overnight.

If a shorter period is required, the ingots, ingot parts or pieces may be cooled in a few hours by immersing them in sand at a temperature less than 200 °C.

This treatment induces oxidation of the surface when conducted in the presence of air, and decarburization to a depth of 0,5 to 1 mm, whatever the atmosphere in the furnace. Chips shall therefore not be taken from the surface of the metal after heat treatment. Either a 2 to 3 mm thickness should be cut from the surfaces and the resultant block machined, or the chips obtained from the first 2 to 3 mm of the sample during machining should be discarded.

D.3 Selection of cutting tools

The cutting tools used shall be made of suitable types and grades of steel, so that tool wear and resultant contamination of the chips will be reduced as much as possible.

For high-speed steel tools, table D.1 gives the specifications from ISO 4957, *Tool steels*.

Table D.1 — Steels for high-speed tools

Grade	S 9	S 10	S 11	S 12
Designation	HS 12-1-5-5	HS 10-4-3-10	HS 2-9-1-8	HS 7-4-2-5
C %	1,45 to 1,60	1,20 to 1,35	1,05 to 1,20	1,05 to 1,20
Co %	4,70 to 5,20	9,50 to 10,5	7,50 to 8,50	4,70 to 5,20
Cr %	3,50 to 4,50	3,50 to 4,50	3,50 to 4,50	3,50 to 4,50
Mo %	0,70 to 1,00	3,20 to 3,90	9,00 to 10,0	3,50 to 4,20
V %	4,75 to 5,55	3,00 to 3,50	0,90 to 1,40	1,70 to 2,20
W %	11,5 to 13,0	9,00 to 10,0	1,30 to 1,90	6,40 to 7,40
Minimum Rockwell hardness after tempering (HRC)[1]	65	66	66	66

1) 66 HRC is equivalent to approximately 900 Vickers.

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The high carbon, chromium and cobalt contents ensure tool hardness; the molybdenum prevents the chips from sticking to the tool.

For hard ferronickels (for example of hardness greater than 180 Vickers), experience has shown that it is indispensable to have a steel with a cobalt content greater than or equal to 7,5 %; type S 11 appears to be the most suitable.

For less hard ferronickels, one may be satisfied with a quality containing about 5 % cobalt, for example type S 12.

For tungsten carbide tools: a type shall be chosen that affords a compromise between resistance to wear and toughness in order to avoid tool chafing or rupture. Selection has therefore to be made between types M 10, M 20 or M 30 of ISO 513.

NOTE — The data in this clause are for guidance only. They correspond to present experience of laboratories having experimented in the field.

D.4 Additional precautions

Vibration between the cutting tool and the metal being machined shall be avoided as far as possible.

When the metal is to be drilled, very short drills that are not too thin shall be used (the diameter should be no less than 12 mm and preferably between 15 and 20 mm). A low helix angle is also favourable; for example, a drill with a 15° helix angle could be used, as opposed to a standard drill with a helix angle of 30°.

The use of a taper-shank drill (Morse taper socket No.2 or 3) is highly desirable.

Milling cutters shall also be short in relation to their diameter.

Finally, the machine shall be very rigid. This is readily achieved with a milling machine but more difficult to accomplish with a drilling machine, whether equipped with a milling table or not.

In either case, it shall be possible to mount the tool on the spindle using a sufficiently solid intermediate: standard ISO taper SA 40 or SA 50.

D.5 Machining parameters

The machining specifications shall be such that

— there is very little heating of the tool, so that it does not become worn; tool wear can be detected by examining the chips produced during machining: slight yellowing is acceptable, but they must never be blue.

— the feed per tooth does not fall below a minimum value during either drilling or milling, so that the material does not undergo work hardening; this is necessary for normal operation of the tool, free from vibration, wear and abnormal heating.

The parameters given in table D.2 must be considered to achieve a good compromise.

Table D.2 — Parameters to be considered

Symbol	Parameter	Unit of measurement
N	Speed of rotation of the tool	revolution per minute
D	Diameter of drill or milling cutter	millimetre
d	Number of teeth[1)]	
V_1	Linear cutting speed	metre per minute
V_2	Rate of longitudinal feed or cross-feed (milling) or of vertical feed (drilling)	millimetre per minute
a	Feed per tooth	millimetre per tooth

1) In mechanical terms, each tooth corresponds, in the case of a drill (examples in D.6.1, D.6.2 and D.6.3) or in the case of end mill cutters (examples in D.6.4, D.6.5 and D.6.6.) to one flute.

The relationships of these parameters are expressed in the following equations:

$$V_1 = \frac{\pi DN}{1\ 000}$$

$$a = \frac{V_2}{Nd}$$

(valid with the above-mentioned units)

Good machining conditions are obtained by selecting appropriate values for V_1 and a and then adjusting N and V_2 accordingly on each machine[1)].

Examples of recommended specifications are given in table D.3.

Table D.3 — Examples of recommended specifications

	V_1 maximum m/min	V_1 desirable m/min	a maximum mm/tooth	a desirable mm/tooth	a minimum mm/tooth
High-speed steel drill	4	2 to 3	0,05	0,04	0,03
Tungsten carbide drill	10 to 12	4 to 7	0,03	0,02	0,015
High-speed steel chip-breaking milling cutter	6	2 to 3	0,03	0,015 to 0,02	0,01
High-speed steel end milling cutter	6	2 to 4	0,05	0,03 to 0,04	0,02

1) In the case of milling, the linear cutting speed is applied in a rigorous manner; the above-mentioned diameter D is always the diameter corresponding to the cutting point of the rotating tool. In the case of a drill, the formula V_1 normally applies to the cutting portion of the drill which is located on the outer diameter; but V_1 has bad significance for the working zones of the drill on a smaller diameter and above all for the point, which penetrates by pressure rather than by cutting. This is why the set of cutting parameters can be controlled better in milling than in drilling.

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Understandably, for low-hardness metal machining, the maximum values given in table D.3 may be increased.

To achieve these specifications in practice, machining shall be done within the following limits:

N = 30 to 100 rpm when drilling
40 to 100 rpm when milling

V_2 = 3 to 10 mm/min when drilling
5 to 20 mm/min when milling

D.6 Examples of suitable tools

The descriptions below are given for guidance only. They should facilitate research for the reader and also testing for selecting the most appropriate cutting tools from the various producers in each country.

Only a life duration test with macrographical cutting lip examination can be used for drawing reliable conclusions. A high-performing tool type or sharpening obtained at one supplier may perform less well at another. The following are examples only.

D.6.1 High-speed steel drill

Diameter : 15 to 20 mm
Morse taper : No. 2 or 3
Useful length : 60 to 70 mm
Helix angle : 15° (or, failing this, 30°)
Point angle : 140° (or, failing this, 130°, but no less)
Clearance angle : 5° to 7°
Back-clearance angle : About 15°
Rake angle : Positive (i.e. in the same direction as the helix) but may be 3° to 6°
Drill point grinding : 3 rake angles : clearance, back-clearance and web clearance

Web thinning permits having a cross-edge of 1 to 2 mm.

D.6.2 Carbide drill

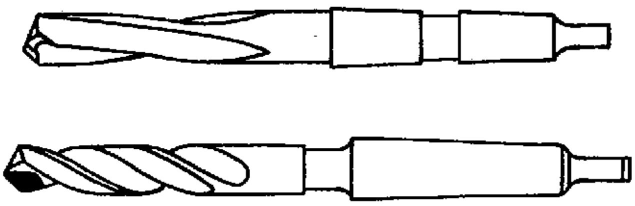

Figure D.1 — Carbide drill

Diameter : Approximately 15 mm
Useful length : Approximately 35 mm
Morse taper : No. 2
Helix angle : 10° to 15° (or, failing these, 30°)
Point angle : 130°
Clearance angle : 2° to 4°
Back-clearance angle : About 15°
Rake angle : Positive (i.e. in the same direction as the helix) but may be 2° to 5°
Sharpening : 3 rake angles : clearance, back-clearance and web thinning.

The remark for web thinning as in D.6.1 applies here. Here it is very important not to reduce the cross-edge to less than 1 mm. Otherwise, the risk of rupture of the point is very high.

This type of drill is not to be used with the hardest ferronickels. The risk of carbide flaking or point rupture increases when hardness increases. And the steel land wear beyond the carbide tip becomes very important due to blocked chips inside the hole inducing friction. (The latter disavantage is suppressed when using solid carbide tools.)

D.6.3 Oil-hole drill

See table D.4 and figure D.2.

Table D.4 — Characteristics of oil-hole drills

	Drill diameter	
	15,875 mm	19,050 mm
Total length	241,3 mm	266,7 mm
Groove length	123,825 mm	149,225 mm
Helix angle	34°	34°
Point angle	118°	118°
Clearance angle 1)	10°	10°
Back-clearance angle 2) (approximately)	10°	10°

1) As measured at the intersection of the side flank with the land (margin).

2) This angle shall be altered to improve performance. Such sharpening or so-called "flatted-lip" reduces the effective inclination of the axis with respect to the side flank and has proved advantageous for many applications.

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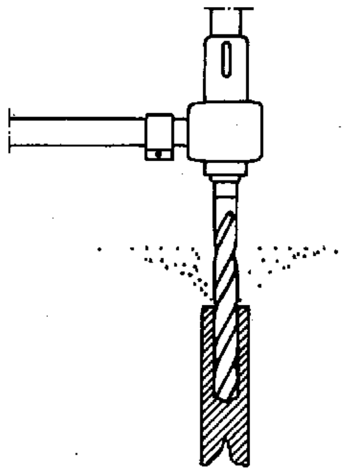

Figure D.2 — Oil-hole drill

These drills are used with compressed air instead of oil feeding. A special coupling ring is used for this purpose.

Such drills are not made of steels with high cobalt and molybdenum contents. Hence, they cannot be used for very hard ferronickels.

Chip recovery as shown in figure 1 shall be used with chips drilling technique.

D.6.4 Chip-breaking mill cutter[1)]

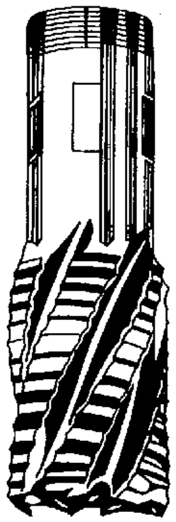

Figure D.3 — Chip-breaking mill cutter

Diameter : 20 to 30 mm
Number of teeth (or flutes) : 4, 5 or 6
Profile of the longitudinal edge of the length of each flute : Rough-formed cutting edge, round profile
Useful length : 30 to 45 mm
Clearance angle : 3° to 4°
Rake angle : 2° to 5°
Mounting : By an intermediate socket with SA 40 or SA 50 taper

This type of milling cutter is suitable for working with the cylindrical part.

Cut depth : 0,5 to 2 mm, depending on the hardness of the material

D.6.5 End-mill cutter[1)]

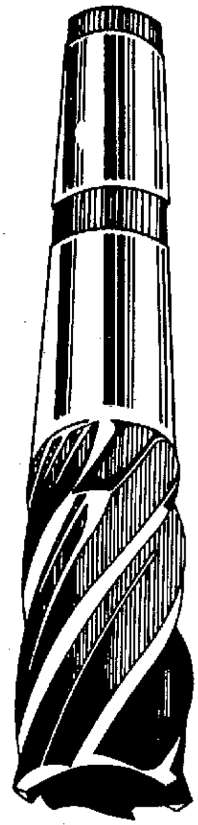

Figure D.4 — End-mill cutter

Diameter : 20 to 50 mm
Morse taper : No. 3 or 4
Number of teeth (or flutes) : 4, 5 or 6
Useful length : 35 to 75 mm
Clearance angle : 4° to 6°
Rake angle : 2° to 5°

This type of milling cutter is more suitable for working with the end than with the cylindrical part.

Cut depth : 0,5 to 2 mm, depending on the hardness of the material

1) For the terminology of milling cutters, see ISO 3855.

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D.6.6 Diesinking cutter head

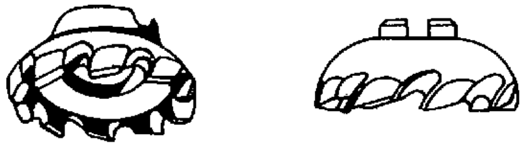

Figure D.5 — Diesinking cutter head

Diameter : 50 or 80 mm
Number of teeth (or flutes) : 6 or 10
Useful length : 10 to 15 mm
Clearance angle : 4° to 6°
Rake angle : 2° to 5°
Mounting : by an intermediate socket with socket with SA 40 or SA 50 taper

This type of milling cutter is used only on end but under the same conditions as an end-mill cutter.

Cut depth : 0,5 to 2 mm, depending on the hardness of the material

From life duration tests made by some producers, it appears that the lowest wear (or greatest permissible number of cuts before wearing out and new sharpening of the milling cutter) is obtained with this type of cutter.

NOTE — Tungsten carbide milling cutters have a shorter life than steel milling cutters for ferronickel machining.

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UDC 669.15'24-198:620.11

Descriptors : ferroalloys, ferronickel, ingots, sampling.

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