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Refined nickel — Sampling

Nickel raffiné — Échantillonnage

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 7156 was prepared by Technical Committee ISO/TC 155, *Nickel and nickel alloys*.

Annexes A and B of this International Standard are for information only.

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Refined nickel — Sampling

1 Scope

This International Standard specifies sampling procedures for up to 25 tonnes (metric tons) of refined nickel of the same composition, size and shape and manufactured under similar conditions of production.

NOTE 1 These procedures are not primarily intended for routine or production lot sampling. However, they can, in certain cases, be used for lot transactions between purchaser and supplier.

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 indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 6283:1979, *Refined nickel*.

ISO 6372-2:1989, *Nickel and nickel alloys — Terms and definitions — Part 2: Refinery products*.

3 Presentation of the product

Refined nickel is usually delivered in one of the following forms:

— whole-sheet cathodes which weigh about 50 kg for a thickness most often between 6 mm and 12 mm;

— drums containing metal pieces. The pieces may be cut cathodes (generally squares with 25 mm, 50 mm or 100 mm edges), briquettes, pellets, shot, granules or powder. The capacity of the drums is most often 50 kg, 250 kg or 1 000 kg.

4 Principle of sampling procedure

4.1 Symbols

For the purposes of this International Standard, the following symbols are used.

U The total number of units of packaging in a lot of 25 tonnes or less. These units may be whole-sheet cathodes or drums.

N The number of units which constitute the primary sample. These N units are the primary increments.

n The number of increments taken from each of the primary increments.

$N \times n$ The number of secondary increments which constitute the secondary sample.

NOTE 2 The justification of the number of primary and secondary increments is given in annex A.

4.2 Sample selection

4.2.1 From the U units contained in the lot, N units are selected to constitute the primary sample. The selection of these units shall respect the rules of random sampling.

4.2.2 From each of the N primary increments, n secondary increments are taken. The $(N \times n)$ secondary increments are combined and constitute the secondary sample.

4.2.3 An adequate complementary treatment to reduce the mass of the secondary sample results in the final laboratory sample for chemical analysis. Cathodes or briquettes are machined to obtain a final sample in the form of fine chips. Pellets, shot or granules are either taken as they are or, when their particle size analysis allows, machined to obtain chips. Powders are homogenized and reduced by riffling until the final sample is obtained.

5 Sample preparation

5.1 The laboratory sample shall be prepared as directed in the clauses dealing with various product forms.

5.2 The laboratory sample shall be of sufficient mass for the chemical analysis planned. For fine chips or powder, it is recommended to divide a sample of at least 200 g between two parties and to keep two portions in reserve in case of dispute. For larger pieces, such as pellets, granules or shot, a minimum mass of 500 g is recommended for each party and for reserve.

5.3 Precautions in sample preparation

5.3.1 Given the high purity of certain qualities of nickel, extremely strict precautions shall be taken in order not to contaminate the sample. Contamination of a sample may occur from tools, utensils and containers used in the sampling operation. Care shall therefore be taken in their selection and use to eliminate or minimize such contamination.

5.3.2 Contamination from cutting tools by elements such as cobalt, chromium, molybdenum, vanadium and tungsten shall be avoided. All machining operations shall be carried out without using lubricants. Experience has shown that high-speed steel cutting tools are better for nickel metal than tungsten carbide tools.

5.4 Final preparation of the laboratory sample

5.4.1 Any sample which has been through a mechanical preparation and, in particular, machining into chips, will inevitably be contaminated on the surface of the metal by, at least, the element iron. It is essential, therefore, that the chips or pieces are cleaned by etching with acid before the test sample is taken for analysis. Unless otherwise specified in the International Standard to be used for the analytical method, the laboratory shall be instructed to clean the test sample as directed in 5.4.2.

5.4.2 Place the chips in a beaker and cover with a few millilitres of concentrated hydrochloric acid. Heat at low temperature and, as soon as dissolution starts (evolution of a few bubbles), add a large quantity of distilled or demineralized water to stop this dissolution. Pour off the diluted acid and wash the chips several times with water, by decantation of the water from the beaker, until acid-free. Wash the chips with high-purity acetone and dry them in a low-temperature oven. Take the test sample to be analysed from the clean chips and keep the remainder for future analyses.

6 Sampling of whole-sheet cathodes

6.1 Primary sampling

6.1.1 Determine the number of units U of whole-sheet cathodes in the lot and select, at random, N units (primary increments) using table 1 as a guide. The number of units in a lot of a given mass and the number of units which constitute the primary sample in table 1 are based on a unit mass of 50 kg.

6.1.2 If the mass per cathode is significantly different from 50 kg, the number of primary increments N shall be selected on the basis of the mass of the lot, as given in column 1 of table 1, and the number N of selected units as given in column 3 of table 1.

6.2 Secondary sampling

6.2.1 From each of the primary increments, n secondary increments as given in column 4 of table 1 shall be taken. Where values of n are coupled, e.g. 3 and 2, or 2 and 1, these are to be distributed randomly among the N primary increments to give $(N \times n)$ as given in column 5 of table 1.

6.2.2 The secondary increments may be taken by any one of the three following methods.

a) Drilling a hole through the cathode using a drill, preferably with a diameter between 15 mm and 25 mm. It is possible to obtain chips by appropriate shaping of the drill. Strands of metal shall be cut into small pieces.

b) Milling a hole using a cylindrical milling cutter with a diameter between 15 mm and 25 mm to produce chips directly. It may be necessary to drill a small pilot hole, e.g. 2 mm to 3 mm, when using certain designs of cutter.

c) Punching out a disc of about 15 mm to 25 mm diameter and milling the disc to produce chips.

6.2.3 For the position of the secondary increment, five geometrical positions are defined on a diagonal of a whole-sheet cathode. Position 1 is taken at a distance of 25 mm to 30 mm from one corner. The distance from position 1 to the centre of the cathode is then divided into five equal intervals to define positions 2 to 5. Thus position 5 is a little before the middle of the cathode. A template may be used to define the positions of the holes along the diagonal of the cathode. The starting corner for the template shall be alternated for each cathode to be sampled, to avoid bias due to starting at the top or bottom of the cathode.

6.2.4 For lots of greater than 3,75 tonnes (where $n = 1$) the secondary increment from the first cathode shall be taken at position 1, from the second at position 2, etc., and back to position 1 for the sixth cathode. When n is greater than 1, the $(N \times n)$ increments shall be distributed among the N cathodes so that positions 1 to 5 are used nearly the same number of times.

6.2.5 Combine the chips from the $(N \times n)$ increments constituting the secondary sample and mix thoroughly. The mass of the chips shall be at least 200 g. Divide the sample by riffling or by fractional shovelling to provide the required number of laboratory samples.

7 Sampling of drums containing forms requiring comminution or machining

Nickel forms such as cathode shapes, briquettes, shot or large granules or pellets require comminution or machining to obtain chips or pieces of a suitable size for the laboratory sample.

7.1 Primary sampling

7.1.1 The number of units U in a lot is the number of drums of given mass. Select the number of drums N to constitute the primary sample as given in table 1 for drums of 50 kg, table 2 for drums of 250 kg and table 3 for drums of 1 000 kg.

7.1.2 For drums of intermediate mass, the numbers N and n shall be adjusted such that the total number of secondary increments $(N \times n)$ is the constant number given in table 1 to table 3 for a given tonnage. The number of units N shall be as high as possible as a function of the number of units U in the lot, and n shall be as small as possible for a given value of $(N \times n)$ specified in table 1 to table 3.

The following example illustrates this point:

5 tonne lot, by drums of 400 kg
U = 13 drums
From table 2, $N \times n$ = 23 and N = 20
but N cannot be greater than U
Therefore n is 2 or 1 and N = 13
i.e. n = 2 for 10 of the drums and 1 for 3 of the drums.

7.2 Secondary sampling

7.2.1 From each drum constituting the primary sample, take at random one or more pieces (secondary increments) as given in table 1 to table 3.

7.2.2 Machine the secondary increments by milling or drilling and cutting, as indicated in 6.2.2, to obtain chips. Briquettes shall be machined from the edge to the centre such that one half of each piece is consumed in the milling process. Large pellets or shot may be pressed between platens for further machining or cutting.

7.2.3 Combine the chips from the $(N \times n)$ increments constituting the secondary sample and mix thoroughly. The mass of the chips shall be at least 200 g. Divide the sample by riffling or by fractional shovelling to provide the required number of laboratory samples.

NOTE 3 For pellet or shot, it may be necessary to increase the value of n to obtain sufficient material for the laboratory samples.

8 Sampling of drums containing forms not requiring comminution or machining

Nickel forms such as powder, fine shot and granules and small pellets can be sampled directly and do not need further preparation such as comminution.

8.1 Primary sampling

8.1.1 Select the number of units (primary increments) to constitute the primary sample as directed in 7.1.1.

8.2 Secondary sampling

8.2.1 One secondary increment of at least 500 g shall be taken from each drum constituting the primary sample. The masses of each of these secondary increments shall be approximately equal. For products which are known to be homogeneous, material may be scooped from the top of the drum. Otherwise, secondary increments shall be obtained by fractional shovelling or by riffling the whole drum.

8.2.2 Secondary increments of nickel powder produced by chemical methods shall be obtained by successive riffling of the entire contents of each drum through 1:1 splitters until a mass of about 500 g is obtained from each of the primary increments.

8.2.3 Combine the $(N \times n)$ secondary increments constituting the secondary sample and homogenize. Reduce the bulk of the sample by consecutive riffling through 1:1 splitters to obtain the required number of laboratory samples.

9 Sampling report

The sampling report shall include the following particulars:

a) reference to the appropriate clauses of this International Standard;

b) any operation not included in this International Standard or which is regarded as optional;

c) any unusual features noted during sampling or any deviations made from the sampling procedure.

Table 1 — Sample selection from units of 50 kg

Mass of lot tonnes	Total number of units in lot	N 1)	n 2)	$N \times n$ 3)
0,050	1	1	5	5
0,100	2	2	3	6
0,150	3	3	3 and 2	7
0,200	4	4	2	8
0,250	5	5	2 and 1	9
0,300 to 0,400	6 to 8	6	2 and 1	9
0,450 to 0,550	9 to 11	7	2 and 1	10
0,600 to 0,700	12 to 14	8	2 and 1	11
0,750 to 0,850	15 to 17	9	2 and 1	11
0,900 to 1,050	18 to 21	10	2 and 1	12
1,100 to 1,300	22 to 26	11	2 and 1	13
1,350 to 1,500	27 to 30	12	2 and 1	14
1,550 to 1,750	31 to 35	13	2 and 1	15
1,800 to 2,050	36 to 41	14	2 and 1	15
2,100 to 2,350	42 to 47	15	2 and 1	16
2,400 to 2,650	48 to 53	16	2 and 1	17
2,700 to 3,000	54 to 60	17	2 and 1	18
3,050 to 3,350	61 to 67	18	2 and 1	19
3,400 to 3,750	68 to 75	19	2 and 1	20
3,800 to 4,150	76 to 83	20	1	20
4,200 to 4,550	84 to 91	21	1	21
4,600 to 4,950	92 to 99	22	1	22
5,000 to 5,450	100 to 109	23	1	23
5,500 to 5,900	110 to 118	24	1	24
5,950 to 6,400	119 to 128	25	1	25
6,450 to 6,900	129 to 138	26	1	26
6,950 to 7,450	139 to 149	27	1	27
7,500 to 8,000	150 to 160	28	1	28
8,050 to 8,550	161 to 171	29	1	29
8,600 to 9,150	172 to 183	30	1	30
9,200 to 9,750	184 to 195	31	1	31
9,800 to 10,400	196 to 208	32	1	32
10,450 to 10,950	209 to 221	33	1	33
11,000 to 11,750	222 to 235	34	1	34
11,800 to 12,400	236 to 248	35	1	35
12,450 to 13,150	249 to 263	36	1	36
13,200 to 13,850	264 to 277	37	1	37
13,900 to 14,600	278 to 292	38	1	38
14,650 to 15,400	293 to 308	39	1	39
15,450 to 16,150	309 to 323	40	1	40
16,200 to 17,000	324 to 340	41	1	41
17,050 to 17,800	341 to 356	42	1	42
17,850 to 18,650	357 to 373	43	1	43
18,700 to 19,550	374 to 391	44	1	44
19,600 to 20,400	392 to 408	45	1	45
20,450 to 21,350	409 to 427	46	1	46
21,400 to 22,250	428 to 445	47	1	47
22,300 to 23,200	446 to 464	48	1	48
23,250 to 24,150	465 to 483	49	1	49
24,200 to 25,000	484 to 500	50	1	50

1) N is the number of units sampled (primary increments).

2) n is the minimum number of secondary increments from each of the N sampled units.

3) The pairs of values for n (e.g. 3 and 2) shall be distributed randomly over the N sampled units so as to obtain the number ($N \times n$) of secondary increments indicated.

Table 2 — Sample selection from units of 250 kg

Mass of lot tonnes	Total number of units in lot	N 1)	n 2)	$N \times n$ 3)
0,250	1	1	9	9
0,500	2	2	5	10
0,750	3	3	4 and 3	11
1,000	4	4	3	12
1,250	5	5	3 and 2	12
1,500	6	6	3 and 2	13
1,750	7	7	2	14
2,000	8	8	2 and 1	15
2,250	9	9	2 and 1	16
2,500	10	10	2 and 1	16
2,750	11	11	2 and 1	17
3,000	12	12	2 and 1	18
3,250	13	13	2 and 1	19
3,500	14	14	2 and 1	19
3,750	15	15	2 and 1	20
4,000	16	16	2 and 1	21
4,250	17	17	2 and 1	22
4,500	18	18	2 and 1	22
4,750	19	19	2 and 1	23
5,000	20	20	2 and 1	23
5,250	21	21	2 and 1	24
5,500	22	22	2 and 1	24
5,750	23	23	2 and 1	25
6,000	24	24	2 and 1	25
6,250	25	25	2 and 1	26
6,500 to 6,750	26 to 27	26	2 and 1	27
7,000 to 7,250	28 to 29	27	1	27
7,500 to 7,750	30 to 31	28	1	28
8,000 to 8,250	32 to 33	29	1	29
8,500 to 9,000	34 to 36	30	1	30
9,250 to 9,500	37 to 38	31	1	31
9,750 to 10,250	39 to 41	32	1	32
10,500 to 10,750	42 to 43	33	1	33
11,000 to 11,500	44 to 46	34	1	34
11,750 to 12,250	47 to 49	35	1	35
12,500 to 12,750	50 to 51	36	1	36
13,000 to 13,500	52 to 54	37	1	37
13,750 to 14,250	55 to 57	38	1	38
14,500 to 15,000	58 to 60	39	1	39
15,250 to 15,750	61 to 63	40	1	40
16,000 to 16,750	64 to 67	41	1	41
17,000 to 17,500	68 to 70	42	1	42
17,750 to 18,250	71 to 73	43	1	43
18,500 to 19,250	74 to 77	44	1	44
19,500 to 20,000	78 to 80	45	1	45
20,250 to 21,000	81 to 84	46	1	46
21,250 to 22,000	85 to 88	47	1	47
22,250 to 22,750	89 to 91	48	1	48
23,000 to 23,750	92 to 95	49	1	49
24,000 to 25,000	96 to 100	50	1	50

1) N is the number of units sampled (primary increments).

2) n is the minimum number of secondary increments from each of the N sampled units.

3) The pairs of values for n (e.g. 2 and 1) shall be distributed randomly over the N sampled units so as to obtain the number ($N \times n$) of secondary increments indicated.

Table 3 — Sample selection from units of 1 000 kg

Mass of lot tonnes	Total number of units in lot	N 1)	n 2)	$N \times n$ 3)
1,00	1	1	12	12
2,00	2	2	8 and 7	15
3,00	3	3	6	18
4,00	4	4	5	20
5,00	5	5	5 and 4	23
6,00	6	6	5 and 4	25
7,00	7	7	4 and 3	27
8,00	8	8	4 and 3	29
9,00	9	9	4 and 3	31
10,00	10	10	4 and 3	33
11,00	11	11	4 and 3	34
12,00	12	12	3 and 2	35
13,00	13	13	3 and 2	37
14,00	14	14	3 and 2	38
15,00	15	15	3 and 2	40
16,00	16	16	3 and 2	41
17,00	17	17	3 and 2	42
18,00	18	18	3 and 2	43
19,00	19	19	3 and 2	44
20,00	20	20	3 and 2	45
21,00	21	21	3 and 2	46
22,00	22	22	3 and 2	47
23,00	23	23	3 and 2	48
24,00	24	24	3 and 2	49
25,00	25	25	2	50

1) N is the number of units sampled (primary increments).

2) n is the minimum number of secondary increments from each of the N sampled units.

3) The pairs of values for n (e.g. 3 and 2) shall be distributed randomly over the N sampled units so as to obtain the number ($N \times n$) of secondary increments indicated.

Annex A
(informative)

Justification of the number of primary and secondary increments selected

A.1 General

This annex gives the statistical justification for the number of primary and secondary increments to be taken from a lot to produce a representative sample while minimizing the sampling uncertainty and effort required.

A.2 Principle

The basic reasoning was developed for the case of the highest tonnages per lot (25 tonnes) and using the same basis for whole-sheet cathodes as for drums. The first two objectives are

- to select the values of the number (N) of primary increments and the number (n) of secondary increments to obtain the lowest possible uncertainty of sampling for a constant number ($N \times n$) of increments;
- to select the value of ($N \times n$) to obtain a given level of uncertainty for sampling.

The conclusions are then extrapolated towards the lowest tonnages, i.e. a situation in which the number of units (whole-sheet cathodes or drums) is small.

A.3 Mathematical model for high tonnages

A.3.1 Symbols

In addition to the symbols defined in 4.1 (U, N, n and $N \times n$), the following symbols are used:

V_1 within-lot variance (between primary increments) for a particular impurity;

V_2 within-unit variance (between secondary increments issued from one primary increment) for the same impurity;

V_e variance attributable to the selection of samples.

A.3.2 Basic equation

If it is assumed that the physical treatment applied to the ($N \times n$) increments taken introduces a negligible uncertainty as regards V_e, it can be said that V_e is the uncertainty of the whole sampling procedure.

These parameters are related by the equation

$$V_e = \frac{V_1}{N} + \frac{V_2}{N \times n} \qquad \text{. . . (A.1)}$$

Putting

$$s_1 = \sqrt{V_1}\,,\; s_2 = \sqrt{V_2} \text{ and } s_e = \sqrt{V_e}$$

(where s is the corresponding standard deviation) in equation (A.1) gives

$$s_e = \sqrt{\left[\frac{1}{N}\left(s_1^2 + s_2^2/n\right)\right]} \qquad \text{. . . (A.2)}$$

The numerical values of s_1 and s_2 can vary over a very wide range, depending on the impurity considered and the concentration of this impurity. Moreover, for a given value of s_1, the value of s_2 can vary significantly depending on the methods used to manufacture the metal and to package the lots.

Generally, it is assumed that s_2 has a high probability of lying between the limits s_1 and $s_1/3$.

Some numerical values of s_e, calculated using equation (A.2), are given by way of example in table A.1.

A.4 Value to adopt for n

The numerical values in table A.1 demonstrate that, in all cases, for a given value of $N \times n$ fixed increments, the minimum value of s_e is obtained by taking n = 1 and, consequently, N as high as possible.

Example:

Consider the case in which $s_1 = s_2 = 10$ g/t and $N \times n = 50$

If $N = 2$ and $n = 25$, then $s_e = 7{,}2$

If $N = 50$ and $n = 1$, then $s_e = 2{,}0$

A.5 Value to adopt for N

Having selected a value for n, it is necessary to determine which value to adopt for N so that $s_e = \sqrt{V_e}$ does not exceed a chosen limit.

So that the number N of primary increments does not decrease too rapidly as the number of units decreases, it is usual in sampling to use the following relationship:

$$N = k\sqrt{U} \qquad \ldots (A.3)$$

where k is a coefficient which permits the value of s_e to be fixed at the chosen level.

Moreover, it is preferable for N to depend only on the tonnage of the lot and not on the number U of units. Consequently, it is necessary to include, on the right-hand side of equation (A.3), a second coefficient, the value of which depends on the mass of the units in the lot.

Thus

$$N = ka\sqrt{U} \qquad \ldots (A.4)$$

where

a = 1 for units of 50 kg;

a = 2,25 for units of 250 kg;

a = 4,5 for units of 1 000 kg.

The value of N therefore depends on the tonnage and the coefficient k corresponding to the level chosen for s_e. In practice, the value of k selected is 2,25, which results in N = 50 for a 25 tonne lot.

NOTE 4 It is the combination of k = 2,25 and a = 1 or a = 2,25 or a = 4,5 which allows a round number (50) to be chosen for N for a 25 tonne lot.

If the nickel is packaged in 1 tonne drums, U = 25 for a 25 tonne lot; thus N cannot be greater than 25. The value selected for n is therefore 2, so that $N \times n$ = 50. Every drum in the lot shall be emptied and the contents sampled randomly at a minimum of two different points.

A.6 Lower tonnages

When considering the total number ($N \times n$) of increments, it can be seen that, for a very low tonnage, i.e. a small number of units, using equation (A.4) when n = 1 gives a very low total number of primary increments, because N cannot be greater than U. This is all the more true as the mass per unit is higher. In the case of a 1 tonne drum, the procedure described would give only one item, whereas the same mass in 50 kg drums would give N = 10, i.e. not less than 10 items, one for each of the drums selected.

Thus, in practice, as the tonnage decreases, the number ($N \times n$) of increments is progressively increased in accordance with the two conditions n = 1 and $N = ka\sqrt{U}$.

Examples:

For a lot of 1 tonne, $N \times n$ is taken as 12. This is true whether the mass of the drums is 50 kg, 250 kg or 1 000 kg.

For a lot consisting of a single drum of 250 kg, N is taken as 1 and n as 9 ($N \times n$ = 9); for a lot consisting of 5 drums of 50 kg, N is taken as 5 and ($N \times n$) as 9.

For a lot consisting of a single drum of 50 kg, N is taken as 1 and n as 5.

For each tonnage (or number of drums), table 1, table 2 and table 3 give the appropriate values of N and n. When n is a combination of two values, the operator selects randomly from the series of N sampled drums.

It is necessary to select a value of n greater than 1

— when the lot weighs less than 3,8 tonnes and consists of 50 kg units (i.e. U = 76);

— when the lot weighs less than 7 tonnes and consists of 250 kg units (i.e. U = 20);

— for any lot consisting of 1 tonne units.

A.7 Whole-sheet cathodes

In a lot of whole-sheet cathodes, the mass of each cathode is very often close to 50 kg and is always less than 100 kg. The figures in table 1, calculated for 50 kg drums, are therefore also used for whole-sheet cathodes.

In the case of whole-sheet cathodes, it is particularly important to bear in mind that certain impurities can be present at a concentration a little higher near the edge of the cathode than at the centre. Any discrepancy remains, however, within limits which are perfectly compatible with the level being aimed at for s_e provided that

— the five geometrical positions specified in 6.2.3 of the procedure are respected;

— the ($N \times n$) increments are taken at random to give a balanced distribution between these five positions.

Table A.1 — Examples of numerical values of s_e

s_1 (g/t)			100	100	10	10	1	1
s_2 (g/t)			100	33	10	3,3	1	0,33
N	n	$N \times n$	s_e	s_e	s_e	s_e	s_e	s_e
2	1	2	100	74	10,0	7,4	1,0	0,74
2	3	6	82	72	8,2	7,2	0,82	0,72
2	9	18	75	71	7,5	7,1	0,75	0,71
2	25	50	72	71	7,2	7,1	0,72	0,71
10	1	10	45	33	4,5	3,3	0,45	0,33
10	3	30	37	32	3,7	3,2	0,37	0,32
10	9	90	33	32	3,3	3,2	0,33	0,32
10	25	250	32	32	3,2	3,2	0,32	0,32
30	1	30	26	19	2,6	1,9	0,26	0,19
30	3	90	21	19	2,1	1,9	0,21	0,19
30	9	270	19	18	1,9	1,8	0,19	0,18
30	25	750	19	18	1,9	1,8	0,19	0,18
50	1	50	20	15	2,0	1,5	0,20	0,15
50	3	150	16	14	1,6	1,4	0,16	0,14
50	9	450	15	14	1,5	1,4	0,15	0,14
50	25	1 250	14	14	1,4	1,4	0,14	0,14

Annex B
(informative)

Technical conditions for drilling and milling

B.1 General

The most common procedures for the preparation of nickel samples are milling and drilling. The hardness of nickel metal may vary considerably, depending mainly on the conditions of the metallurgical process of production. If the metal is not very hard, there is usually no problem in machining without contaminating the chips. Sometimes, however, the metal is very hard and very strict precautions must be taken to avoid an unacceptable level of contamination, particularly with very pure nickel with its very low levels of impurities.

B.2 Reference

ISO 4957:1980, *Tool steels.*

B.3 Selection of cutting tools

Experience shows that tungsten carbide tools are not very suitable for machining nickel, as the metal sticks to the cutting tool so that attrition is high. High-speed steel tools are recommended. Table B.1 gives the specification from ISO 4957 for some such tool steels. The high carbon, chromium and cobalt contents ensure tool hardness; the molybdenum prevents the chips from sticking to the tools. It is useful to have a very high cobalt content. Grade S-11 appears to be the most suitable; grade S-12 is also suitable.

B.4 Machining parameters

B.4.1 All machining operations must be carried out without the use of lubricants or cooling oils, to avoid contamination. Vibration between the cutting tool and the metal being machined should be avoided as much as possible. The drills or milling cutters should be very short, with a diameter between 15 mm and 25 mm. It should be possible to mount the tool on the spindle of the machine using a sufficiently solid intermediate, for example ISO taper SA40 or SA50.

B.4.2 The machining conditions should be such that there is very little heat generated so that the sample does not become hot. The feed per tooth should not fall below a minimum value during either drilling or milling so that work hardening of the material is avoided. The following parameters should be considered to achieve a good compromise:

		Units of measurement
N	= speed of rotation of tool	r/min
D	= diameter of drill or milling cutter	mm
d	= number of teeth	
V_1	= linear cutting speed	m/min
V_2	= rate of longitudinal feed or cross feed (milling) or of vertical feed (drilling)	mm/min
a	= feed per tooth	mm/min

The relationships between these parameters are expressed by the following equations:

$$V_1 = \frac{\pi DN}{1000}$$

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

where each parameter is measured in the units given above.

B.4.3 Good machining conditions are obtained by selecting appropriate values for V_1 and then adjusting N and V_2 accordingly on each machine. Examples of recommended conditions are given in table B.2. To achieve these conditions in practice, machining should be done within the following limits (the values are dependent on the diameter of the machining tool):

N = 40 r/min to 140 r/min when drilling;

N = 100 r/min to 40 r/min when milling;

V_2 = 4 mm/min to 20 mm/min when milling or drilling.

For machining low-hardness metals, the maximum values given in table B.2 may be increased.

Table B.1 — Composition of high-speed tool steels

Grade Designation	S 9 HS 12-1-5-5	S 10 HS 10-4-3-10	S 11 HS 2-9-1-8	S 12 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
HRC[1)]	65	66	66	66

1) Minimum Rockwell hardness after tempering. 66 HRC is equivalent to approximately 900 Vickers.

Table B.2 — Examples of recommended conditions

Type of drill or cutter	Linear cutting speed		Feed per tooth		
	Maximum V_1 m/min	Desirable V_1 m/min	Maximum a mm/tooth	Desirable a mm/tooth	Minimum a mm/tooth
High-speed steel drill	6	3 - 4	0,08	0,05	0,3
High-speed steel chip-breaking milling cutter	8	3 - 4	0,05	0,03	0,01
High-speed steel end milling cutter	8	3 - 5	0,08	0,05	0,02