

JIS

JAPANESE INDUSTRIAL STANDARD

Methods for Determination of Nickel in Ores

JIS M 8126—1988

Translated and Published

by

Japanese Standards Association

Printed in Japan

In the event of any doubt arising,
the original Standard in Japanese is to be final authority.

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Methods for Determination of Nickel in Ores

M 8126-1988

1. Scope

This Japanese Industrial Standard specifies the methods for determination of nickel in ores. However, this Standard does not apply to the ores of which methods for determination of nickel are specified in other JIS.

2. General Matters

General matters common to the methods for determination shall be in accordance with the following:

JIS K 0050

JIS K 0115

JIS K 0121

JIS K 0116

3. Methods for Sampling and Handling of Samples

3.1 Sampling and Preparation of Samples The sampling and preparation of samples shall be in accordance with JIS M 8101 and JIS M 8083.

3.2 Handling and Weighing Method of Sample The handling and weighing method of sample shall be as given in the following:

- (1) In weighing out the sample, mix well taking care so that the sample represents the mean composition.

Applicable Standards:

JIS K 0050-General Rules for Chemical Analysis

JIS K 0115-General Rules for Absorptiometric Analysis

JIS K 0116-General Rules for Emission Spectrochemical Analysis

JIS K 0121-General Rules for Atomic Absorption Spectrochemical Analysis

JIS K 8001-General Rules of Testing Methods for Reagents

JIS M 8083-Methods for Sampling of Non-Ferrous Flotation Concentrates in Bulk

JIS M 8101-Methods for Sampling of Metal Bearing Ores of Copper, Lead, Zinc, Tin, Gold, Silver and others

JIS Z 8401-Rules for Rounding off of Numerical Values

(2) Dry the sample for 2 h in an air bath regulated at $105 \pm 5^{\circ}\text{C}$, cool it down to an ordinary temperature in a desiccator and weigh its mass. Dry it in the same manner as foregoing, weigh after it has been left cooling, and repeat drying until the mass difference of before and after becomes within 0.1 %. However, the drying conditions (temperature, time, etc.) of the sample which is liable to change in property due to inclusion of sulfide and the like shall be as agreed on between the parties concerned.

(3) Weigh out the specified mass of the sample nearest to 0.1 mg.

4. Expression of Analytical Value and Tolerances

4.1 Expression of Analytical Value The expression method for analytical value shall be as given in the following:

- (1) Express the analytical value in mass percentage, and round it off to 3 places of decimals in accordance with JIS Z 8401.
- (2) Carry out 2 analyses repeatedly in the same analysis room, obtain their mean value when the difference between these is less than the room tolerance, hereinafter referred to as the "tolerance", round it off to 2 places of decimals in accordance with JIS Z 8401, and consider this to be the report value.
- (3) When the difference between two analytical values is not less than the tolerance, carry out 2 analyses again.
- (4) The tolerances shall be as given in Table 1.

Table 1. Tolerances

Unit: wt %

Method for determination	Division ⁽¹⁾	Tolerances (repetition)
Dimethylglyoxime gravimetric method	0.5 or over to less than 1	0.025
	1 or over to less than 5	0.050
	5 or over to less than 15	0.100
	15 or over to less than 30	0.150
	30 or over	0.200
Dimethylglyoxime separation EDTA titration	0.5 or over to less than 1	0.025
	1 or over to less than 5	0.050
	5 or over to less than 15	0.100
	15 or over to less than 30	0.150
	30 or over	0.200
Dimethylglyoxime absorptiometry	0.01 or over to less than 0.05	0.005
	0.05 or over to less than 0.1	0.010
	0.1 or over to less than 0.5	0.020
Atomic absorption method	0.01 or over to less than 0.05	0.005
	0.05 or over to less than 0.1	0.010
	0.1 or over to less than 0.5	0.020
Inductively coupled plasma emission spectro-chemical analysis	0.01 or over to less than 0.05	0.005
	0.05 or over to less than 0.1	0.010
	0.1 or over to less than 0.5	0.020

Note ⁽¹⁾ When two analytical values extend over 2 divisions, the tolerance of the division to which the mean value of the two analytical values corresponds shall be applied.

4.2 Caution on Analytical Operations In the analysis, blank tests shall be carried out through-out the overall operations to correct the measured values.

5. Classification of Methods of Determination

The methods of determination of nickel in ores shall be one of the following:

- (1) Dimethylglyoxime Gravimetric Method This method shall be applied to the sample of nickel content 0.5 wt % or over.
- (2) Dimethylglyoxime Separation EDTA Titration This method shall be applied to the sample of nickel content 0.5 wt % or over.
- (3) Dimethylglyoxime Absorptiometry This method shall be applied to the sample of nickel content 0.01 wt % or over to less than 0.5 wt %.
- (4) Atomic Absorption Method This method shall be applied to the sample of nickel content 0.01 wt % or over to less than 0.5 wt %.
- (5) Inductively Coupled Plasma Emission Spectrochemical Analysis This method shall be applied to the sample of nickel content 0.01 wt % or over to less than 0.5 wt %.

6. Dimethylglyoxime Gravimetric Method

6.1 Summary The sample shall be decomposed by hydrochloric acid, nitric acid and perchloric acid, and the solution is concentrated by heating to generate the fume of perchloric acid. After salts have been dissolved in hydrochloric acid and water, the solution shall be filtered. The filtrate shall be jointed by tartaric acid, alkalfied weakly by the addition of aqueous ammonia, and allowed to precipitate nickel by adding dimethylglyoxime. The precipitates shall be filtered out with a glass filter, dried and weighed for its mass.

6.2 Reagents The reagents shall be as follows:

- (1) Hydrochloric acid
- (2) Hydrochloric acid (1 + 1) (1 + 9)
- (3) Nitric acid
- (4) Perchloric acid
- (5) Hydrofluoric acid
- (6) Sulfuric acid (1 + 1)
- (7) Aqueous ammonia
- (8) Ammonium cleansing solution Add 1 to 2 drops of aqueous ammonia to about 500 ml of warm water. This solution shall be prepared on each occasion of use.

- (9) Sodium disulfate
- (10) Tartaric acid solution (40 w/v %)
- (11) Dimethylglyoxime solution Dissolve 1 g of dimethylglyoxime $[(CH_3)_2C_2(NO_2)_2]$ in 100 ml of ethanol (95 v/v %)
- (12) Standard nickel solution (40 $\mu\text{g Ni/ml}$) Decompose 1.000 g of nickel (99.9 % or over) with 20 ml of nitric acid (1 + 1), transfer it into a 1000 ml volumetric flask after cooling, dilute to the marked line and take it as a primary solution. Each occasion of use, dilute a required amount of this primary solution exactly to 25 times, and consider it to be the standard nickel solution.
- (13) Bromothymol blue ($C_{27}H_{28}Br_2O_5S$) solution (0.1 w/v %) Preparing method shall be in accordance with 4.3 (1) of JIS K 8001.

6.3 Mass of Sample to Be Weighed Out The mass of sample to be weighed out shall be as given in Table 2.

Table 2. Mass of Sample to Be Weighed Out⁽²⁾

Nickel content wt %	Mass of sample to be weighed out g
30 or over	0.5 to 1
15 or over to and excl. 30	1 to 2
5 or over to and excl. 15	0.5 to 1.5
0.5 or over to and excl. 5	1 to 2

Note (2) In the case where the nickel content is 15 wt % or over, weigh out the sample so that the amount of nickel becomes 300 to 600 mg, and where the nickel content is 5 wt % or over to and excluding 15 wt %, weigh out the sample so that the amount of nickel becomes 60 to 120 mg.

Further, in the case where the nickel content is less than 5 wt %, preferably weigh out so that the amount of nickel becomes 40 mg or over as far as possible.

6.4 Operations The determining operations shall be carried out in the following procedure:

- (1) Weigh out a sample, transfer it into a beaker (200 to 300 ml), cover with a watch glass, add 10 to 20 ml of the hydrochloric acid, add 5 ml of the nitric acid and 20 to 30 ml of perchloric acid when the amount of solution becomes about 5 ml by heating, continue heating to decompose, allow the fume of perchloric acid to generate sufficiently, and concentrate until the amount of solution becomes about 20 ml or under.

- (2) After it has been cooled, add 20 ml of the hydrochloric acid (1 + 1) and about 50 ml water, heat gently to dissolve soluble salts, then filter using filter paper (class 5 B), wash the residue 3 to 4 times with hydrochloric acid (1 + 9), and then wash with warm water sufficiently⁽³⁾.
- (3) Receive the filtrate and washings in a beaker (500 ml), and cool to an ordinary temperature. Further, in the case of a sample of nickel content 15 wt % or over, transfer the filtrate and washings into a transfer flask of 250 ml, dilute to the marked line with water, separate 50 ml from this, and transfer it into a beaker (500 ml).
- (4) Add 25 ml of tartaric acid solution to this solution, and make it up to about 300 ml by adding water. Add 2 to 3 drops of bromothymol blue solution as an indicator, drop aqueous ammonia until the colour of solution changes from yellow to green while stirring the solution, and further add 5 ml in excess⁽⁴⁾.
- (5) Heat this solution to 70°C⁽⁵⁾, add dimethylglyoxime solution in the ratio of 4 ml for each 10 mg of the resultant amount of nickel and cobalt in the solution while stirring, and, further add 20 ml in excess⁽⁶⁾. Next, after stirring up the solution violently for about 30 sec, continue heating and keep the temperature of solution at about 70°C for about 10 min, and further leave it to stand at room temperature for about 2 h to allow the precipitates of nickel methylglyoxime to mature⁽⁷⁾.
- (6) Filter out the precipitates with a glass filter (1 G 4) preliminarily given a constant mass by drying at 110 to 120°C, and wash well with the ammonium cleansing solution⁽⁸⁾.
- (7) Reduce the precipitates to a constant mass by drying at 110 to 120°C together with the glass filter and, after cooling it to ordinary temperature in a desiccator, weigh its mass.

Notes ⁽³⁾ In the case where nickel is included in the residue, transfer the residue together with the filter paper into a platinum crucible (No. 30), dry them and heat gently to incinerate. After it is cooled, add several drops of sulfuric acid (1 + 1) and 3 to 5 ml of hydrofluoric acid, heat to vaporize the silicon dioxide, and evaporate to dryness. Add 2 to 3 g of sodium disulfate, and heat gently to fuse. After cooling, dissolve the melt in a small amount of warm water and about 1 ml of the hydrochloric acid, filter with filter paper (class 5 B), wash 3 to 4 times with warm water, and mix the filtrate and washings with the main solution.

⁽⁴⁾ The pH of this solution shall be about 9.0.

- (5) In the case where precipitates are generated in this solution and where not less than 1 mg of copper, 10 mg of lead, 50 mg of zinc or 100 mg of manganese is included in this solution, carry out the operation of Note (7) after the operation of 6.4 (5) is carried out, since the amount of impurities included in the precipitates of nickel dimethylglyoxime increases then.
- (6) In the blank test, the amount of dimethylglyoxime solution to be added shall be 20 ml.
- (7) In the case where the precipitates of generated nickel dimethylglyoxime are likely to include impurities, filter out the precipitates with filter paper (class 5 A), wash with the ammonia cleansing solution, transfer the precipitates into the original beaker by washing with warm water, add 20 ml of hydrochloric acid and 10 ml of nitric acid and, after decomposition by heating, allow nickel dimethylglyoxime to reprecipitate by repeating the operations of 6.4 (4) and (5) (however, addition of tartaric acid solution shall be 10 ml and the excess of dimethylglyoxime solution shall be 5 ml). When anticipating a determination of nickel in the filtrate and washings in accordance with Note (8), preserve the filtrate and washings.
- (8) In the case of the sample of nickel content 15 wt % or over, and of the sample of nickel content less than 15 wt % and, further in the case where the sample to be weighed out is less than 1 g, preserve the filtrate and washings, and determine the nickel in the filtrate and washings in accordance with the following method.

Transfer the filtrate and washings thus preserved into a beaker (500 to 1000 ml), and if Note (7) is applied, further mix the filtrate and washings, heat to evaporate and concentrate until the amount of solution becomes about 100 ml, and add 50 ml of the hydrochloric acid. After cooled to an ordinary temperature, transfer into a volumetric flask of 200 ml, dilute with water to the marked line, measure absorbance by operating in accordance with the procedure of 9.4 (3), and obtain the amount of nickel from the working curve drawn up from the following operation.

Prepare several beakers (200 to 300 ml), add 25 ml of tartaric acid solution to each of them [when Note (7) is applied, 35 ml], and when Note (3) is applied, further add 2 to 3 g of sodium disulfate and dissolve, neutralize by adding aqueous ammonia, add 50 ml of hydrochloric acid, and after cooling transfer into a volumetric flask of 200 ml each with a small amount of water. Next, take gradual amounts of the