

ICS 73.060

CCS D42



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB/T 15923-2010

Replacing GB/T 15923-1995

**Method for chemical analysis of nickel ores - Determination of
nickel content**

镍矿石化学分析方法 镍量测定

Issued on: November 10, 2010

Implemented on: February 1, 2011

**Issued by: General Administration of Quality Supervision, Inspection and
Quarantine of the People's Republic of China;
Standardization Administration of China**

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1 Application Scope

GB/T 15923-2010 is the Chinese national standard on method for chemical analysis of nickel ores - determination of nickel content, in the field of mining and minerals. The /T suffix marks it as a recommended standard: it is not compulsory by itself, but it becomes binding as soon as a contract, a tender or a customer specification calls it up - which in practice is how most foreign buyers meet it. It was issued on 10 November 2010 by the General Administration of Quality Supervision, Inspection and Quarantine of the People's Republic of China; Standardization Administration of China, and has been in force since 1 February 2011. Classification: ICS 73.060, CCS D42. This page is published from the official record of the standard held by the Chinese standards administration: the identification, the dates, the classification and the issuing body are taken from there. The clause text, the tables and the numeric limits are in the document itself, which is delivered complete in English translation.

This Standard specifies the atomic absorption spectrometric method for the determination of nickel content in nickel ores, in which an acidic solution is prepared after acid dissolution or alkali fusion separation. This Standard applies to the determination of nickel in nickel ores. The measuring range: 0.1% ~ 6.0% of nickel.

2 Normative References

The provisions in following documents become the provisions of this Standard through reference in this Standard. For dated references, the subsequent amendments (excluding corrigendum) or revisions do not apply to this Standard, however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

GB/T 6682, Water for Analytical Laboratory Use - Specification and Test Methods

3 Principle

After sample is decomposed by hydrofluoric acid, aqua regia and perchloric acid, use hydrochloric acid to dissolve salts (or use sodium peroxide and sodium hydroxide to melt ores, use water to extract and filter, and use hydrochloric acid to dissolve the sediment); in a hydrochloric acid medium of 5%, use the air-acetylene flame to measure the absorbance of nickel on the atomic absorption spectrometer at the wavelength of 352.5 nm; and calculate the nickel content.

4 Reagents

Unless specified otherwise, the analytically pure reagents and the water for analytical laboratory use specified in GB/T 6682 shall be used in the analysis.

4.1 Sodium peroxide.

4.2 Sodium hydroxide.

4.5 Hydrofluoric acid (rho

1.13 g/mL). Warning: Hydrofluoric acid is toxic and corrosive, so gloves shall be worn during operation to avoid contact with the skin!

4.6 Perchloric acid (rho

1.67 g/mL). Warning: Explosive, handle with care!

4.7 Absolute ethyl alcohol.

4.8 Aqua regia: [hydrochloric acid (4.3) + nitric acid (4.4)] = (3 + 1), freshly prepared.

4.9 Hydrochloric acid (1 + 1).

4.10 Nitric acid (1 + 1).

4.11 Sodium hydroxide solution (20 g/l).

4.12 Nickel standard solution: weigh 0.500 0 g of metal nickel (of mass fraction $\geq 99.99\%$); place into a 100 mL beaker; put on the watch glass; add 20 mL of nitric acid (4.10) along

the beaker wall; heat to dissolve; and dry by distillation at low temperature. Use a small amount of water to purge the watch glass; add 5 mL of hydrochloric acid (4.3); dry by distillation at low temperature; and repeat once. Add 10 mL of hydrochloric acid (4.3) to dissolve nickel salt; cool down; use water to transfer into a 1 000 mL volumetric flask; dilute to volume; and shake up. Then the mass concentration of the solution nickel is 0.50 mg/mL.

5 Apparatus

5.1 Atomic absorption spectrometer. It is equipped with nickel element hollow cathode lamp.

5.2 Analytical balance: grade 3; sensitivity

0.1 mg. Place specimen (7.1) into corundum crucible; add 4 g of sodium peroxide (4.1); use 1 g of sodium hydroxide (4.2) to cover the top; place into a high temperature furnace whose temperature has been raised to 650°C; maintain the temperature until fully melted (about 10 min); and take out for cooling. Place the crucible into a 200 mL beaker; add 100 mL of warm water; put on the watch glass; after the drastic reaction stops, add several drops of absolute ethyl alcohol (4.7); boil on the electric furnace for 3 min ~ 5 min; and take down for cooling. Filter; use hot sodium hydroxide solution (4.11) to wash the beaker and crucible and precipitate for 6 times ~ 8 times; use hot hydrochloric acid (4.9) to dissolve and precipitate in the original beaker; wash out the crucible; and place the solution on the electric hot plate to evaporate to dryness. Add 5 mL of hydrochloric acid (4.9) and a small amount of water to dissolve salts; use water to transfer into a 50 mL volumetric flask; dilute to volume; and shake up.

7.5 Preparation of calibration solution series In a series of 50 mL volumetric flasks, add respectively

0.00 mL,

0.50 mL,

10.00 mL of nickel standard solution (4.12); add 10 mL of hydrochloric acid (4.9); use water to dilute to volume; and shake up. The mass concentrations of the serial solution nickels are respectively 0.00 µg/mL, 5.00 µg/mL, 10.00 µg/mL ... 100.00 µg/mL.

7.6 Determination Warning: Ignite or extinguish air-acetylene flame in accordance with the instructions to use of the atomic absorption spectrometer, in order to avoid causing possible explosion hazards! Use the flame atomic absorption spectrometer to measure the absorbance of nickel in the calibration solution series, blank solution and test solution (

7.4.1 and 7.4.2), at the wavelength of 352.5 nm, in accordance with the operating conditions of the instrument (refer to Annex A).

7.7 Plotting of calibration curve Plot the calibration curve using the nickel content as the abscissa and the absorbance as the ordinate; and obtain the corresponding nickel content from the calibration curve.

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