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NATIONAL STANDARD OF THE PEOPLE'S REPUBLIC OF CHINA

GB/T 14353.18-2014

Methods for chemical analysis of copper ores, lead ores and zinc ores - Part 18: Determination of copper content, lead content, zinc content, cobalt content and nickel content

铜矿石、铅矿石和锌矿石化学分析方法 第18部分：铜量、铅量、锌量、钴量和镍量测定

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1 Scope

Warning: persons using this part shall have practical experience of regular laboratory work. This part does not point out all the possible safety problems. It is the responsibility of the user to take suitable safety and health measures and to make sure that the conditions laid down by the relevant national regulations are met.

This part of GB/T 14353 lays down the method for the simultaneous determination of copper, lead, zinc, cobalt and nickel in copper ores, lead ores and zinc ores by inductively coupled plasma optical emission spectrometry.

This part applies to the simultaneous determination by inductively coupled plasma optical emission spectrometry of copper, lead, zinc, cobalt and nickel in copper ores, lead ores and zinc ores.

Determination range: 0.002 % to 8.5 % of copper, 0.01 % to 5 % of lead, 0.005 % to 3 % of zinc, 0.001 5 % to 0.5 % of cobalt, 0.003 % to 0.5 % of nickel.

Detection limits of the method: copper 0.000 66 %, lead 0.003 2 %, zinc 0.001 7 %, cobalt 0.000 47 %, nickel 0.001 0 %.

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition, including all amendments, applies.

GB/T 6682 Water for analytical laboratory use - Specification and test methods

GB/T 14505 Methods for chemical analysis of rocks and ores - General rules and regulations

3 Principle

After the test portion has been decomposed with hydrochloric acid, nitric acid, hydrofluoric acid and perchloric acid, the test solution, in a 20 % nitric acid medium containing 3 % hydrochloric acid, is introduced into a high-temperature plasma torch, where the elements to be determined are excited into ions and atoms and emit the characteristic spectral lines of the elements contained. The emission spectral intensity of the ions and atoms of each element is measured at the prescribed wavelength; the emission spectral intensity is proportional to the concentration of the element measured, and the content of the element measured is calculated by the computer built into the instrument using the calibration curve method.

4 Reagents

Unless otherwise stated, analytically pure reagents and water for analytical laboratory use complying with GB/T 6682 are used throughout the analysis.

4.1 Nitric acid (density 1.42 g/mL).

4.2 Hydrofluoric acid (density 1.13 g/mL). Warning: hydrofluoric acid is toxic and strongly corrosive; corrosion-resistant gloves shall be worn during the operation and contact with the skin avoided.

4.3 Hydrochloric acid (density 1.19 g/mL).

4.4 Perchloric acid (density 1.68 g/mL). Warning: explosive, handle with care.

4.5 Mixed acid (nitric acid plus hydrochloric acid = 4+1).

4.6 Nitric acid (1+9).

4.7.1 Copper standard solutions. a) Copper stock standard solution, mass concentration of copper 1.00 mg/mL: 0.500 0 g of copper metal of purity greater than 99.99 % is weighed

into a 250 mL beaker, covered with a watch glass, 10 mL of nitric acid (1+1) is added down the wall of the beaker and the solution warmed gently; after dissolution is complete 10 mL of sulfuric acid (1+1) is added, the solution is evaporated until white fumes of sulfur trioxide appear, taken off and cooled, the copper salt dissolved in water, the watch glass washed with water, and the solution transferred after cooling into a 500 mL volumetric flask, diluted to the mark with water and mixed. b) Copper standard solution, mass concentration of copper 100.0 μg/mL: 25.00 mL of the copper stock standard solution [4.7.1a)] is taken into a 250 mL volumetric flask, diluted to the mark with hydrochloric acid (5+95) and mixed. c) Copper standard solution, mass concentration of copper 20.0 μg/mL: 50.00 mL of the copper standard solution [4.7.1b)] is taken into a 250 mL volumetric flask, diluted to the mark with hydrochloric acid (5+95) and mixed.

4.7.2 Lead standard solutions. a) Lead stock standard solution, mass concentration of lead 1.00 mg/mL: 1.000 0 g of lead metal of purity greater than 99.99 % is weighed into a 250 mL beaker, covered with a watch glass, 10 mL of nitric acid (1+1) is added down the wall of the beaker and dissolved with heating; the watch glass is washed with a little water and the solution transferred into a 1 000 mL volumetric flask, diluted to the mark with water and mixed. b) Lead standard solution, mass concentration of lead 20.0 μg/mL: 20.00 mL of the lead stock standard solution [4.7.2a)] is taken into a 1 000 mL volumetric flask, diluted to the mark with water and mixed.

4.7.3 Zinc standard solutions. a) Zinc stock standard solution, mass concentration of zinc 1.00 mg/mL: 1.000 0 g of zinc metal of purity greater than 99.99 % is weighed into a 250 mL beaker, covered with a watch glass, 10 mL of hydrochloric acid (1+1) is added down the wall of the beaker and the mixture left to dissolve by itself, more acid being added if there is not enough; after dissolution is complete the watch glass is washed with water and the solution transferred into a 1 000 mL volumetric flask, diluted to the mark with water and mixed. b) Zinc standard solution, mass concentration of zinc 20.0 μg/mL: 20.00 mL of the zinc stock standard solution [4.7.3a)] is taken into a 1 000 mL volumetric flask, diluted to the mark with water and mixed.

4.7.4 Cobalt standard solutions. a) Cobalt stock standard solution, mass concentration of cobalt 100 μg/mL: 0.100 0 g of cobalt metal of purity greater than 99.99 % is weighed into a 100 mL beaker, covered with a watch glass, 20 mL of nitric acid (1+1) is added down the wall of the beaker, dissolved with heating and evaporated to dryness at low temperature; the watch glass is rinsed with a little water, 5 mL of hydrochloric acid (4.3) is added and the solution evaporated to dryness at low temperature, and the operation is repeated once. 10 mL of hydrochloric acid (4.3) is added to dissolve the cobalt salt, the solution is cooled, transferred with water into a 1 000 mL volumetric flask, diluted to the mark and mixed, and kept for use. b) Cobalt standard solution, mass concentration of cobalt 10.0 μg/mL: 50.00 mL of the cobalt stock standard solution [4.7.4a)] is transferred into a 500 mL volumetric flask, diluted to the mark with hydrochloric acid (1+99) and mixed.

4.7.5 Nickel standard solutions. a) Nickel stock standard solution, mass concentration of nickel 100 μg/mL: 0.100 0 g of nickel metal of purity greater than 99.99 % is weighed into a 100 mL beaker, covered with a watch glass, 20 mL of nitric acid (1+1) is added down the wall of the beaker, dissolved with heating and evaporated to dryness at low temperature; the watch glass is rinsed with a little water, 5 mL of hydrochloric acid (4.3) is added and the solution evaporated to dryness at low temperature, and the operation is repeated once. A further 10 mL of hydrochloric acid (4.3) is added to dissolve the nickel salt, the solution is cooled, transferred with water into a 1 000 mL volumetric flask, diluted to the mark and mixed. b) Nickel standard solution, mass concentration of nickel 10.0 μg/mL: 50.00 mL of the nickel stock standard solution [4.7.5a)] is transferred into a 500 mL volumetric flask, diluted to the mark with hydrochloric acid solution (1+99) and mixed.

5 Apparatus

5.1 Inductively coupled plasma optical emission spectrometer.

5.2 Analytical balance: class three, sensitivity 0.1 mg.

6 Test sample

6.1 The particle size of the test sample prepared according to the relevant provisions of GB/T 14505 shall be less than 97 μm.

6.2 The test sample is dried in an oven at 60 °C to 80 °C for 2 h to 4 h and cooled to room temperature in a desiccator before use.

7 Analytical procedure

7.1 According to the content of the element to be determined in the test sample, the test portion is weighed as given in Table 1 to the nearest 0.1 mg.

Table 1, test portion mass, has seven columns: the element, and then two sets of three columns giving the content in units of ten to the power minus two, the mass of test portion in grams and the make-up volume in millilitres. For Co the first set is 0.001 5 to 0.1 with 0.25 g made up to 25.00 mL and the second set is 0.1 to 0.5 with 0.1 g made up to 100.0 mL; for Cu, 0.002 to 0.1 with 0.25 g to 25.00 mL and 0.1 to 8 with 0.1 g to 100.0 mL; for Ni, 0.003 to 0.1 with 0.25 g to 25.00 mL and 0.1 to 0.5 with 0.1 g to 100.0 mL; for Pb, 0.01 to 0.1 with 0.25 g to 25.00 mL and 0.1 to 5 with 0.1 g to 100.0 mL; for Zn, 0.005 to 0.1 with 0.25 g to 25.00 mL and 0.1 to 3 with 0.1 g to 100.0 mL.

The note below Table 1 states that, for one and the same sample, when the mass fractions of copper, lead, zinc, cobalt and nickel differ by more than four orders of magnitude, 0.25 g and 0.1 g of the sample may be weighed separately, decomposed and made up into two test solutions of 25.00 mL and 100 mL and determined at the same time; the result