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YS/T 820.23-2012

Methods for Chemical Analysis of Laterite Nickel Ores - Part 23: Determination of Cobalt, Iron, Nickel, Phosphorus, Aluminium Oxide, Calcium Oxide, Chromium Oxide, Magnesium Oxide, Manganese Oxide, Silicon Dioxide and Titanium Dioxide Content - Wavelength Dispersive X-ray Fluorescence Spectrometry

红土镍矿化学分析方法 第 23 部分：钴、铁、镍、磷、氧化铝、氧化钙、氧化铬、氧化镁、氧化锰、二氧化硅和二氧化钛量的测定 波长色散 X 射线荧光光谱法

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Foreword

This Part was drafted in accordance with the rules given in GB/T 1.1-2009.

YS/T 820-2012, *Methods for Chemical Analysis of Laterite Nickel Ores*, includes 26 parts:

-- *Part 1: Determination of Nickel Content - Flame Atomic Absorption Spectrometry;*

-- *Part 2: Determination of Nickel Content - Dimetylglyoxime Spectrophotometry*;

-- *Part 3: Determination of Total Iron Content - Potassium Dichromate Titration*;

-- *Part 4: Determination of Phosphorus Content - Phosphorus Molybdenum Blue Spectrophotometry*;

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-- *Part 6: Determination of Copper Content - Flame Atomic Absorption Spectrometry*;

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-- *Part 20: Determination of Aluminum Content - EDTA Titration*;

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-- *Part 24：Determination Hygroscopic Moisture Content - Gravimetric Method*;

-- Part 25：*Determination of Combined Water Content - Gravimetric Method*;

-- *Part 26: Determination of Loss on Ignition - Gravimetric Method*.

This Part is Part 23 of YS/T 820-2012.

This Standard shall be under the jurisdiction of National Technical Committee 243 on Non-ferrous Metals of Standardization Administration of China (SAC/TC 243).

The responsible drafting organizations of this Standard are Beijing General Research Institute of Mining and Metallurgy, Bayuquan Entry-exit Inspection and Quarantine Bureau of the People's Republic of China, Jinchuan Non-ferrous Group Co., Ltd.

The drafting organizations of this Part: Ningbo Entry-exit Inspection and Quarantine Bureau of the People's Republic of China, Bayuquan Entry-exit Inspection and Quarantine Bureau of the People's Republic of China.

The contributory drafting organizations of this Part: Changshu Entry-exit Inspection and Quarantine Bureau, Nantong Entry-exit Inspection and Quarantine Bureau.

The main drafters of this Part: Zhang Jianbo, Lin Li, Chu Ning, Hou Jin, Zhu Jiayu, Li Weigang, Dou Huaizhi, Chen Shaohong, Hou Jianguo.

m_3--the mass of specimen, in g.

The result of calculation shall be rounded off to the second decimal place.

7.3 Preparation of specimen fuse pieces

One of the following methods may be used to weigh specimen:

a) weigh accurately 0.600 0 g of burned specimen, 6.000 0 g of mixed solvent of lithium tetraborate and lithium metaborate (4.1) into the crucible (5.2); add 1 ml of lithium nitrate-lithium bromide solution (4.4) after mixing up; and dry on the electric furnace. The result obtained with this measuring method is the ignition basis result of sample.

b) If the X-ray fluorescence spectrometer software has the correction function of loss on ignition, directly weigh 0.600 0 g of specimen (6.1) and 6.000 0 g of mixed solvent of lithium tetraborate and lithium metaborate (4.1) in the crucible (5.2); add 1 ml of lithium nitrate-lithium bromide solution (4.4) after mixing up; dry on the electric furnace; and input the loss on ignition (L) into the software for correction. The result obtained with this measuring method is the air-dried basis result of sample.

NOTE Appropriate specimens and the weight of solvents may be chosen in accordance with the size of the die, but the mass ratio 1:10 must be maintained unchanged.

Specimen and solvent fuse at 1 020°C; and rotate and/or shake from time to time until they are fully dissolved and the melt is uniform. If some fuse beads hang on the wall of the crucible, shake the crucible to fuse them down. After 15 min of fusion, pour the melt into the die preheated for more than 3 min; take out; and cool down.

An automatic fusion furnace may also be used to fuse specimen; shake and rotate the crucible during the fusion process; and the glass pieces made are in the crucible or poured into the die for forming.

The fuse pieces of specimen shall be uniform glass vitreous bodies whose surface shall be flat and smooth without inclusions of bubbles, infusible particles etc.; or else, they shall be prepared once again.

7.4 Plotting of calibration curve

7.4.1 Standard samples

Use reagents of high purity, solution of nitric acid or water medium of known concentration and/or multiple certified standard substances to prepare standard samples. See Annex A for the specific method for preparation. Multiple certified standard substances are recommended as the basis for the preparation of standard samples.

The reagents of high purity used shall be pure oxides or carbonates, if possible. The

Annex A

(Informative)

Preparation of Calibration Samples

A.1 Basic principle

The fusion process of samples is also the homogenization process of samples. For major components, add different masses of high-purity oxides or carbonates; for minor components, add different volumes of solutions of nitric acid medium or water medium of known concentration. Homogenize all components through the fusion process of samples. If multiple certified standard substances of similar compositions are used as the basis, it can significantly accelerate the preparation speed of calibration samples. For minor components, if the solutions of nitric acid medium or water medium of known concentration are not available, it is also acceptable to fuse high-purity oxides or carbonates and fusion agents together, before crushing, weighing and adding.

The high-purity reagents used shall be pure oxides or carbonates, if possible. The reagents weighed during fusion shall not contain water or carbon dioxide; or, it is corrected. When the first grade national standard solutions of appropriate concentration of nitric acid medium or water medium are available, priority shall be given to the use of the first grade national standard solutions.

A.2 Preparation

A.2.1 For the high-content components including magnesium oxide, aluminium oxide, silicon dioxide and ferric oxide, directly weigh air-dried high-purity oxides; and record the mass, accurate to 0.1 mg. Meanwhile, measure the loss on ignition of the oxide at 1 050°C; and correct the mass weighed.

A.2.2 Phosphorus: directly use first grade standard solution; or weigh certain mass of diammonium hydrogen phosphate dried (for 2 h at 105°C) to dissolve in water; and add water dropwise to volume. Calculate the concentration of phosphorus in accordance with the weighed mass and constant volume. The concentration of the solution is around 1 000 mg/ml.

A.2.3 Chromium oxide, nickel, manganese, cobalt: weigh 1.0 g of standard potassium dichromate to dissolve in water; record the mass, accurate to 0.1 mg; add water dropwise to 100 ml; and calculate the concentration of potassium dichromate. Weigh 1.0 g of high-purity metal nickel ($w_{Ni} \geq 99.99\%$); record the mass, accurate to 0.1 mg; dissolve in diluted nitric acid; add diluted nitric acid dropwise to 100 ml; and calculate the concentration of manganese. For cobalt, directly use first grade national standard solution. Because potassium oxide is introduced in potassium dichromate, determine potassium oxide to correct its effects on the measurement.

NOTE For metal nickel and manganese, use diluted acid to remove the oxide layer on the surface before use.

A.2.4 Titanium dioxide and calcium oxide: weigh 0.06 g of high-purity titanium dioxide burned (at 1 050°C), 1.0 g of standard calcium carbonate dried (for 2 h at 220°C) and 6.000 g of fusion agent; record the mass of titanium dioxide and calcium carbonate, accurate to 0.1 mg; crush them after fusion; and calculate the concentration of titanium dioxide and calcium oxide in the mixture. Titanium dioxide and calcium carbonate may also be fused with fusion agent before cooling and crushing. When preparing magnesium oxide, aluminium oxide, silicon dioxide, ferric oxide etc. of relatively low content, this method may also be used.

A.2.5 In accordance with the set concentration of calibration samples, calculate the mass or volume of all components required respectively based on that the total mass is the samples' weight; then weight corresponding weight of all components and fusion agent into the crucibles; and record the actual mass, accurate to 0.1 mg. After mixing up, use a micropipette to add to all solutions of corresponding volumes; then add 1 ml of lithium nitrate-lithium bromide mixed solution; and cast into glass pieces in accordance with the preparation method of specimen fusion pieces after drying on the electric furnace. If the components of weighed parts contain fusion agent, adjust the mass of fusion agent weighed finally to make the total mass of fusion agent consistent with the mass of fusion agent during specimen fusion. Calculate the concentration of all components in calibration samples in accordance with the actual mass and volume.

A.2.6 Under normal conditions, there is certain differences between the calculated mass and the actual sample weight. For the software having the loss-on-ignition correction function, such differences may be dealt with the loss-on-ignition correction function; and for the software without such function, first weigh all components, record the total mass of all components after fusion; and then weigh fusion agent in accordance with the relationship of 1:10.

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