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Replacing YS/T 575.23-2009

Methods for chemical analysis of bauxite - Part 23: Determination of element contents - X-ray fluorescence spectrometry method

铝土矿石化学分析方法 第 23 部分：元素含量的测定 X 射线荧光光谱法

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Foreword

This document was drafted in accordance with the provisions of GB/T 1.1-2020 "Directives for standardization - Part 1: Rules for the structure and drafting of standardizing documents".

This document is Part 23 of YS/T 575 "Methods for chemical analysis of bauxite". YS/T 575 has published the following parts:

- Part 1: Determination of aluminium oxide content EDTA titrimetric method;
- Part 2: Determination of silicon dioxide content - Gravimetric-molybdenum blue photometric method;
- Part 3: Determination of silicon dioxide content molybdenum blue photometric method;
- Part 4: Determination of iron oxide content - Dichromate titrimetric method;
- Part 5: Determination of iron oxide content - Orthophenanthroline photometric method;
- Part 6: Determination of titanium dioxide content - Diantipyrylmethane photometric method;
- Part 7: Determination of calcium oxide content - Flame atomic absorption spectrophotometric method;
- Part 8: Determination of magnesium oxide content - Flame atomic absorption spectrophotometric method;
- Part 9: Determination of potassium oxide and sodium oxide content - Flame atomic absorption spectrophotometric method;
- Part 10: Determination of manganese oxide content - Flame atomic absorption spectrophotometric method;
- Part 11: Determination of chromium oxide content - Flame atomic absorption spectrophotometric method;
- Part 12: Determination of vanadium pentoxide content - N-benzoy-N-phenylhydroxylamine photometric method;
- Part 13: Determination of zinc content flame - Atomic absorption spectrophotometric method;
- Part 14: Determination the total content of rare earth oxide - Tribromo-arsenazo

photometric method;

- Part 15: Determination of gallium oxide content - Rhodamine B-extraction photometric method;
- Part 16: Determination of phosphorus pentoxide content - Molybdenum blue spectrophotometric method;
- Part 17: Determination of sulfur content - Direct combustion-iodometric method;
- Part 18: Determination of total carbon content - Non-aqueous titrimetric method after combustion;
- Part 19: Determination of the loss on ignition Gravimetric method;
- Part 20: Preparation of pre-dried sample;
- Part 21: Determination of organic carbon content titrimetric method;
- Part 22: Determination of hydroscopic moisture on gravimetric method;
- Part 23: Determination of element contents - X-ray fluorescence spectrometric method;
- Part 24: Determination of carbon content and sulfur content - Infrared absorption method;
- Part 25: Determination of sulfur content - Coulometric titration method.

This document replaces YS/T 575.23-2009 "Methods for chemical analysis of bauxite - Part 23: Determination of element contents - X-ray fluorescence spectrometry method". Compared with YS/T 575.23-2009, in addition to structural adjustments and editorial changes, the main technical changes are as follows:

a) MODIFY the measurement range of "Al_2O_3" content (see Chapter 1; Chapter 1 of the 2009 edition);

b) MODIFY the measurement range of "S" content (see Chapter 1; Chapter 1 of the 2009 edition);

c) DELETE the "correction factor" of flux (see 3.1.2 of the 2009 edition);

d) ADD the pre-oxidant "lithium nitrate" (see 5.2.2);

e) MODIFY the "sample" (see Chapter 7; Chapter 5 of the 2009 edition);

f) MODIFY the "test material" (see 8.2; Chapter 6.2 of the 2009 edition);

g) ADD the "high temperature muffle furnace" (see 6.6);

Methods for chemical analysis of bauxite - Part 23: Determination of element contents - X-ray fluorescence spectrometry method

Warning - People using this document shall have practical experience in formal laboratory work. This document does not point out all possible safety issues. The user is responsible for taking appropriate safety and health measures and ensuring that the conditions specified in relevant national regulations are met.

1 Scope

This document specifies the determination method of aluminum oxide, silicon dioxide, total iron (expressed as Fe_2O_3), titanium dioxide, potassium oxide, sodium oxide, calcium oxide, magnesium oxide, phosphorus pentoxide, manganese oxide, sulfur, vanadium, gallium, zinc in bauxite.

This document is applicable to the determination of aluminum oxide, silicon dioxide, total iron (expressed as Fe_2O_3), titanium dioxide, potassium oxide, sodium oxide, calcium oxide, magnesium oxide, phosphorus pentoxide, manganese oxide, sulfur, vanadium, gallium, zinc in bauxite. The determination range is as shown in Table 1.

2 Normative references

The contents of the following documents constitute the essential provisions of this document through normative references in the text. Among them, for dated references, only the version corresponding to that date applies to this document; for undated references, the latest version (including all amendments) applies to this document.

GB/T 8170 Rules of rounding off for numerical values and expression and judgement of limiting values

YS/T 575.19 Methods for chemical analysis of bauxite - Part 19: Determination of loss on ignition - Gravimetric method

3 Terms and definitions

There are no terms and definitions that need to be defined in this document.

4 Principle

The sample is melted with anhydrous lithium tetraborate and lithium metaborate mixed flux. Ammonium nitrate solution or lithium nitrate is added as a pre-oxidant; a small amount of saturated lithium bromide or ammonium iodide solution is added as a demolding agent. It is melted in an automatic melting machine and cast into a glass sample. The fluorescence X-ray intensity of each element in the sample is measured by an X-ray fluorescence spectrometer; a calibration curve is established. The calibration equation is used to correct the absorption enhancement effect between elements; the content of each element in the sample is calculated from the calibration curve.

5 Reagents or materials

Unless otherwise specified, only reagents confirmed as analytically pure and distilled or deionized water or water of relatively pure quality are used in the analysis.

5.1 Flux

5.1.1 Anhydrous lithium tetraborate and lithium metaborate mixed flux, which has a mass ratio of 12:22; after burning at 700 °C for 2 h, place in a desiccator for use.

5.1.2 Anhydrous lithium tetraborate and lithium metaborate mixed flux, which has a mass ratio of 67:33; after burning at 700 °C for 2 h, place in a desiccator for use.

5.2 Pre-oxidant

5.2.1 Ammonium nitrate solution (500 g/L).

5.2.2 Lithium nitrate.

5.3 Release agent

Lithium bromide saturated solution or ammonium iodide solution (300 g/L).

8.2.1 Burning-based samples

According to the mass ratio of sample (7.1) to flux (5.1.1) of 1:8 ~ 1:10 (the recommended sample weight is 0.5 g ~ 0.7 g), accurately weigh the flux (5.1.1) and sample (7.1) in a platinum-gold alloy crucible (6.1), to an accuracy of 0.1 mg; stir evenly; add 1 drop of release agent (5.3).

8.2.2 Dry-base test material

a) Conventional sample: According to the mass ratio of sample (7.2) to flux (5.1.1) of 1:8 ~ 1:10 (the recommended sample weight is 0.5 g ~ 0.7 g), accurately weigh the sample (7.2) and flux (5.1.1) into a platinum-gold alloy crucible (6.1); stir evenly; add 2 mL of ammonium nitrate solution (5.2.1); add 5 drops of release agent (5.3); place in a 700 °C high-temperature muffle furnace (6.6) for pre-oxidation for 5 min.

b) Samples with high sulfur content: According to the mass ratio of sample (7.2), flux (5.1.2) and pre-oxidant (5.2.2) of 1:10:1.5 (the recommended sample weight is 0.5 g ~ 0.7 g), accurately weigh each substance into a platinum-gold alloy crucible (6.1); stir evenly; add 5 drops of release agent (5.3); place in a 600 °C ± 25 °C high temperature muffle furnace (6.6) for pre-oxidation for 15 min.

Note: When using the dry-based sample measurement method, attention shall be paid to the difference in loss-on-ignition of different types of bauxite. If the fluorescence instrument software has a loss-on-ignition correction function, the sample can be prepared directly according to the above method, meanwhile the sample loss-on-ignition can be input into the software for correction. If the instrument's loss-on-ignition correction function is not used, the difference in loss-on-ignition of different types of bauxite must be considered, meanwhile the bauxite samples must be classified to establish the working curve and measurement.

8.3 Preparation of measurement samples

8.3.1 Melting

Put the sample (8.2) into the melting machine, to melt it at 1070 °C ~ 1150 °C for 10 min ~ 15 min. During the melting process, rotate the crucible to allow the small molten beads and samples adhering to the crucible wall to enter the melt. The melting machine automatically shakes the crucible, at regular intervals to drive out bubbles and mix the melt.

8.3.2 Casting

Pour the melt (8.3.1) in the crucible into the mold (6.2), which was heated to 800 °C. Remove the mold from the flame. After cooling, peel off the formed glass sample from the mold. If the sample is directly formed after melting in the crucible, the crucible shall be shaken before cooling to drive out bubbles. The formed glass sample shall be uniform and transparent, with a smooth surface, no bubbles, no crystallization.

8.3.3 Sample storage

A label shall be attached to the non-measurement surface of the glass sample and stored in a desiccator to prevent moisture absorption and contamination. When measuring, only the edge of the sample shall be held, to avoid contamination of the measurement surface.

8.4 Determination

8.4.1 Measurement conditions

The measurement conditions depend on the equipment. Please refer to Appendix A to set the working parameters.

8.4.2 Calibration

8.4.2.1 Selection of calibration samples: National standard substances can be selected as calibration samples. Each element shall have a standard series, which have a sufficient content range and a certain gradient. If the above standard samples cannot meet the requirements, appropriate artificially prepared calibration samples shall be added. For high sulfur content samples, artificially prepared calibration samples can be obtained by adding national pyrite standard substances.

8.4.2.2 Calibration curve establishment: Prepare the selected calibration samples into calibration samples according to steps 8.2 ~ 8.3. Measure the fluorescence intensity of each element in the calibration sample, according to the selected measurement conditions. Draw the calibration curve, where the y-axis represents the net intensity of fluorescence X-rays and the x-axis represents the mass concentration. Select the theoretical α coefficient or the basic parameter method, to calibrate the absorption enhancement effect between elements, according to the calibration curve.

8.4.2.3 Calibration of interference spectral line: For elements with spectral line overlap interference, spectral line overlap interference calibration is required.

8.4.2.4 Instrument drift calibration: Use the monitoring sample (5.4) to perform instrument drift calibration. The first measurement of fluorescence intensity of the monitoring sample (5.4) shall be the same as the start-up measurement of the calibration sample, to ensure the effectiveness of drift calibration.

8.4.3 Measurement of unknown samples

Prepare national standard materials and unknown samples into standard material samples and unknown samples, respectively, according to 8.2 ~ 8.3. Start the quantitative analysis program, to measure the standard material samples. If the analysis results of the elements in the standard material samples meet the repeatability

------ The End ------