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Methods for chemical analysis of aluminum ores – Part 23: Determination of element contents X-ray fluorescence spectrometric method

铝土矿石化学分析方法

第23部分: X射线荧光光谱法测定元素含量

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Foreword

- YS/T 575-2007 "Methods for chemical analysis of aluminum ores" is consisted of 24 parts in total:
 - Part 1: Determination of aluminum 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 Potassium 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, 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-benzoyl-N-phenylhydroxylamine photometric method
 - Part 13: Determination of zinc content Flame atomic absorption spectrophotometric method
 - Part 14: Determination of total content of rare earth oxide Tribromoarsenazo photometric method

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Methods for chemical analysis of aluminum ores Part 23: Determination of element contents X-ray fluorescence spectrometric method

1 Scope

This part specifies the methods for the determination of aluminum oxide, silica, total iron (expressed by Fe₂O₃), titanium dioxide, potassium oxide, sodium oxide, calcium oxide, magnesium oxide, phosphorus pentoxide, manganese oxide, sulfur, vanadium, gallium and zinc, and the like, in the aluminum ores.

This part is applicable to the determination of the aluminum oxide, silica, total iron (expressed by Fe_2O_3), titanium dioxide, potassium oxide, sodium oxide, calcium oxide, magnesium oxide, phosphorus pentoxide, manganese oxide, sulfur, vanadium, gallium and zinc, and the like, in the aluminum ores. It is also applicable to the determination of the aforementioned elements in such samples as clay and kaolin. As for the determination range, SEE Table 1.

Table 1

Composition	Content range (mass fraction)/%	Composition	Content range (mass fraction)/%
$\mathrm{Al}_2\mathrm{O}_3$	30.00~80.00	SiO ₂	1.00~50.00
$\mathrm{Fe}_2\mathrm{O}_3$	0.30~30.00	${ m TiO_2}$	0.50~8.00
K_2O	0.050~3.00	Na ₂ O	0.050~3.00
CaO	0.050~5.00	MgO	0.030~3.00
$P_2 O_5$	0.010~5.00	S	0.050~3.00
MnO	0.0030~0.20	V	0.008 0~0.40
Ga	0.0020~0.050	Zn	0.001 5~0.30

2 Principle of the method

2.1 USE the anhydrous lithium tetraborate and lithium metaborate mixed reagent to melt the sample, in order to eliminate the mineral effects and particle size effects; and CAST it into the glass sheet of the shape suitable for the X-ray fluorescence spectrometer; MEASURE the fluorescence X-ray intensity of the measured element in the glass sheet. USE the calibration curve or equation to conduct analysis; the correction equation is established through using series standard samples, and CONDUCT inter-element disturbance effect correction.

4 Instruments and apparatus

- **4.1** Platinum-gold alloy crucible (95% Pt + 5% Au).
- **4.2** Platinum-gold alloy mold (95% Pt + 5% Au). The base thickness of the mold material is about 1 mm, making it difficult to deform.
- **4.3** The melting vessel and mold can be combined into one. If the sample is directly formed after being melted in the crucible, it requires the internal wall of the crucible bottom flat and smooth, so that the glass sheet is easily peeled off from the mold without being broken.
- **4.4** Melting machine: automatic flame melting or high-frequency induction melting machine. If using other types of melting machines, the temperature is not less than 1150 $^{\circ}$ C, AND the temperature shall be able to controlled, with the temperature control accuracy at \pm 15 $^{\circ}$ C.
- **4.5** Wavelength dispersive X-ray fluorescence spectrometer: end window rhodium target X-ray tube.
- **4.6** Oven: controllable temperature 105 °C \pm 5 °C.

5 Samples

- **5.1** The particle size of sample shall be less than 125 μm.
- **5.2** The sample shall be preheated in an oven (4.6) at 105 ° C \pm 5 °C for 2 h, placed in a desiccator and allowed to cool to room temperature.

6 Analysis procedures

6.1 Number of determinations

TAKE two samples for parallel determination, and TAKE their average value.

6.2 Sample

Based on the equipment used, accurately WEIGH the sample (5) and the flux (3.1) according to the mass ratio of the sample to the flux of 1: 8 ~ 1:10.

6.3 Calibration test

Together with the sample, ANALYZE the standard samples of the same type.

6.5.4 Calibration: As for the elements with spectral line overlap interference, it shall conduct the spectral line overlap interference correction. As for the zinc and gallium element, it shall use the RATIO between the intensity and the RhK α line Compton scattering intensity AND the recommended value regression analysis to conduct calibration. As for the inter-element absorption-enhancement effect, it shall use the theoretical α coefficient or the basic parameter method to correct it.

6.6 X-Ray fluorescence spectrometry

- **6.6.1** PREHEAT the wavelength dispersive X-ray fluorescence spectrometer (4.5) to stabilize it. Based on the X-ray tube type, ADJUST the tube voltage and tube current. Based on the X-ray fluorescence spectrometer model, SELECT the operating parameters (SEE Appendix A).
- **6.6.2** Measurement of monitoring sample: SET the monitoring sample name; MEASURE the X-ray intensity of the analyzed element in the monitoring sample. The reference intensity of the analyzed element in the monitoring sample must be measured in the same machine start-up with the standard sample, in order to ensure the validity of the drift calibration.
- **6.6.3** Measurement of standard sample: INPUT the standard sample name; MEASURE the X-ray intensity of the analyzed element in the standard sample.
- **6.6.4** Measurement of unknown samples: START the quantitative analysis program; MEASURE the monitoring sample; CONDUCT instrument drift correction. MEASURE the standard sample which is prepared in the same batch of the unknown sample. The element analysis results of the standard sample shall comply with the repeatability requirements as specified in Table 2. INPUT the unknown sample name and MEASURE the unknown sample.

7 Calculation of analysis results

MEASURE the X-ray intensity of the standard sample, in order to obtain the quadratic equation or the first order equation of the intensity and concentration. The quadratic equation can be calculated by the least squares method. CALCULATE the calibration curve constants a, b, c AND the spectral line overlap correction coefficient β_{ik} , and SAVE them in the quantitative analysis software of the computer. Based on the X-ray measurement intensity of the unknown sample, USE the computer software and the equation (2) to calculate the content and automatically PRINT the measurement results.

$$W_i = aI_i^2 + bI_i + c \qquad \cdots \qquad (2)$$

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