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Iron ores - Determination of calcium, silicon, manganese, titanium, phosphorus, magnesium, aluminium and barium content - Wavelength dispersive X-ray fluorescence spectrometric method

铁矿石 钙、硅、镁、钛、磷、锰、铝和钡含量的测定 波长色散 X 射线荧光光谱法

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Iron ores - Determination of calcium, silicon, manganese, titanium, phosphorus, magnesium, aluminium and barium content - Wavelength dispersive X-ray fluorescence spectrometric method

WARNING - Persons using this standard shall have practical experience in formal laboratory work. This Part does not address all possible safety issues. It is the user's responsibility to take appropriate safety and health measures and ensure compliance with the conditions stipulated in the relevant national laws and regulations.

1 Scope

This standard specifies the method for determining the content of calcium, silicon, magnesium, titanium, phosphorus, manganese, aluminum, barium in iron ore, by wavelength dispersive X-ray fluorescence spectrometer.

This standard is applicable to the determination of the content of 8 elements, as listed in Table 1, in iron ore and artificial rich ore. The determination range (mass fraction) of each element is as shown in Table 1.

Table 1 -- Determination range of each element

Element	Determination range (mass fraction)/%
Ca	0.02～15.00
Si	0.08～15.00
Mg	0.15～5.00
Ti	0.004～8.00
P	0.005～5.00
Mn	0.009～3.00
Al	0.02～5.00
Ba	0.02～3.00

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For the dated references, the subsequent amendments

(excluding corrections) 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 6379 Precision of test methods - Determination of repeatability and reproducibility for a standard test method by interlaboratory tests (GB/T 6379-1986, neq ISO 5725:1986)

GB/T 6730.1 Methods for chemical analysis of iron ores - Preparation of predried test samples for chemical analysis (GB/T 6730.1-1986, eqv ISO 7764:1985)

GB/T 10322.1 Iron ores - Sampling and sample preparation procedures (GB/T 10322.1-2000, idt ISO 3082:1998)

JJG 810-1993 National Metrology Verification Regulations of the People's Republic of China - Wavelength Dispersive X-Ray Fluorescence Spectrometers

3 Principles

The sample is prepared as a borate glass-like molten sample, to measure the X-ray fluorescence intensity of the element to be measured. Measure the background at the position of the analysis line of the blank molten sample; subtract it as the sample background. The melt samples are synthesized and corrected by pure chemical reagents, to obtain the results, after correcting the matrix effect between elements, by applying the principle of self-consistent correction.

4 Reagents and materials

Unless otherwise stated in the analysis, only approved reagents of analytical grade are used.

4.1 Ferric oxide (Fe_2O_3), guaranteed reagent.

Burn ferric oxide at 1000 °C for at least 1 h. Then cool it in a desiccator.

4.2 Silicon dioxide (SiO_2), guaranteed reagent.

Heat the silica to 1000 °C. Burn for at least 1 h. Then cool it in a desiccator.

4.3 Calcium carbonate ($CaCO_3$), guaranteed reagent.

Bake the calcium carbonate at 105 °C for 1h. Then cool it in a desiccator.

4.4 Magnesium oxide (MgO), guaranteed reagent.

Burn the magnesium oxide at 1000 °C for 1 h. Then place it in a desiccator. Weigh it immediately, after cooling.

4.5 Aluminum oxide (Al_2O_3), guaranteed reagent, α-type.

Burn the aluminum oxide at 1000 °C for at least 2 hours (if the aluminum oxide is not in the α-type, it shall be heated to 1250 °C for at least 2 hours, to convert it into the α-type); then cool it in the desiccator.

4.6 Titanium dioxide (TiO_2), guaranteed reagent.

Burn the titanium dioxide at 1000 °C for at least 1 h. Then cool it in a desiccator.

4.7 Manganese oxide (Mn_3O_4), guaranteed reagent.

Place the manganese dioxide (MnO_2) in a platinum crucible. Burn it at 1000 °C for 24 h. Cool it. Break the resulting bulk material into fine powder. Sinter it at 550 °C for 1 h. Then cool it in a desiccator.

4.8 Potassium dihydrogen phosphate (KH_2PO_4), guaranteed reagent.

Bake the potassium dihydrogen phosphate at 105 °C for 1h. Then cool it in a desiccator.

4.9 Barium carbonate ($BaCO_3$), guaranteed reagent.

Dry the barium carbonate at 105 °C for 1 h. Then cool it in a desiccator.

4.10 Potassium carbonate (K_2CO_3), guaranteed reagent.

Bake the potassium carbonate at 105 °C for 1 h. Then cool it in a desiccator.

4.11 Sodium nitrate ($NaNO_3$)

Bake the sodium nitrate at 105 °C for 1 h. Then cool it in a desiccator.

4.12 Ammonium iodide (NH_4I)

Ammonium iodide does not need to be dried, BUT shall be stored in a desiccator.

4.13 Desiccant

The desiccant shall be freshly regenerated self-indicating silica gel.

4.14 Flux

Use guaranteed reagent anhydrous lithium tetraborate ($Li_2B_4O_7$). Burn it at 500 °C for 4 hours. Then cool and store it in a desiccator.

4.15 Synthetic calibration sample (S)

950 °C).

5.3 Crucible and mold

The crucible and the mold (or the crucible also used as a mold) are made of non-wetting platinum-gold or platinum-gold-rhodium, which is not easy to deform, after heating and melting operation. The crucible shall have sufficient capacity, to hold the flux and specimen required for melting. The mold shall have a flat bottom, which is thick enough to prevent deformation. (It should not be used, when the thickness of the bottom is less than 1 mm).

Since the bottom surface of the melted sample is the analysis surface, the inner surface of the bottom of the mold shall be flat; it is regularly polished with a diamond abrasive of about 12 μm.

6 Sampling and specimen preparation

Take samples and prepare specimens, according to the provisions of GB/T 10322.1.

Prepare pre-dried specimens, according to the provisions of GB/T 6730.1.

7 Preparation of molten samples

7.1 The preparation accuracy of the molten sample shall meet the requirements in Appendix C.

7.2 Preparation of calibration S samples, calibration samples, blank samples

Weigh the reagents, according to the amount of reagents listed in Table 3, accurate to 0.0002 g. Add 6.000 g of flux (4.14) and 0.360 g of sodium nitrate (4.11). After fully mixing, place it in a crucible (5.3). Pre-oxidize it at 700 °C, for 8 min ~ 10 min. Then heat up to 1050 °C ~ 1100 °C for 10 min to melt. Shake the melt in the crucible, during this time. After cooling, add 0.03 g of ammonium iodide (4.12) and melt for 3 min. After taking out, cool and strip the melt in the crucible (or the melt is injected into the mold for cooling and stripping).

Use the independently produced calibration samples (S), to prepare duplicate calibration S samples, calibration samples, blank samples.

m_0 - The specimen weight, in grams (g);

m_1 - The specimen weight after burning, in grams (g).

7.4 Preparation of unknown samples

Weigh 0.6000 g of the burned sample (7.3) and 0.1548 g of potassium carbonate (4.10), accurate to 0.0002 g. The rest operations follow the requirements of 7.2.

7.4.1 Visual inspection

After the molten sample is prepared, visually inspect the molten sample for defects such as unmolten substances, crystals or air bubbles. The defective molten sample shall be discarded; a qualified molten sample shall be prepared again.

7.4.2 Storage of molten samples

In order to prevent the molten sample from absorbing water or being contaminated, quickly put the molten sample into the desiccator (when the molten sample is still warm); do not touch the analysis surface with hands; do not handle it in any way, especially do not rinse with water or other solvents, grinding or polishing.

7.4.3 Cleaning of crucibles and molds

Between two melting operations, the crucible and mold need to be cleaned. Usually soak in hot hydrochloric acid (1 + 1) for about 1 h. Visually check that all residual melts have been removed. Use distilled water to rinse it clean. Use it after drying.

Conditional laboratories can also use the quick cleaning method. Put the crucible or mold into a beaker, which is filled with hydrochloric acid (about 2 mol/L). Put it in an ultrasonic bath, until all residual melt is removed. Use distilled water to rinse it clean. Use it after drying.

8 Analytical conditions and analytical procedures

8.1 Analytical conditions

This standard recommends the use of the following analysis conditions:

1) See Table 4, for analysis lines and analysis crystals;

2) The X-ray fluorescence measurement intensity of each element in the calibration S sample is not lower than the count value, which is listed in Table 4;

3) Using proportional counters and (or) scintillation counters, pulse height selectors; the specimen rotates during measurement;

9 Calculation of results

9.1 Calculation of inter-element correction factor and specimen content

According to the methods listed in B.2.1.1 ~ B.2.1.3 in Appendix B, take the average of the two sets of calibration S samples prepared independently and the X-ray fluorescence intensity of the calibration samples; input it into the computer program, to calculate the inter-element correction factor.

Note 1: This correction factor can be used for a long time, for the same wavelength dispersive X-ray fluorescence spectrometer, under the same equipment and experimental conditions.

Note 2: If the sample to be tested contains a component that seriously interferes with the element to be measured, this component needs to be used as a residue item, to synthesize a residue calibration sample. According to the principle of self-consistent correction, a parameter is added in the correction factor calculation program, to calculate the correction factor of the residue to each analyte.

Note 3: When using a different production batch of flux, the flux blank shall be re-measured.

Note 4: When using 100% Si sheet as the blank sample, the measurement of manganese element content needs to be chemically corrected.

According to the methods listed in B.2.2 ~ B.2.3 in Appendix B, input the X-ray fluorescence intensity of the unknown sample into the computer program, to calculate the specimen content.

9.2 General processing of analysis results

9.2.1 Repeatability and tolerance

The precision of this standard is determined, according to the statistics of GB/T 6379 based on the results of 8 laboratories, by measuring 6 horizontal specimens. The precision is as shown in Table 5.

Appendix B

(Normative)

Calculation of inter-element correction factors and unknown sample content

B.1 Scope

This Appendix specifies the method for calculating inter-element correction factors and unknown sample contents, using a computer program.

B.2 Operation

B.2.1 Calculation of factor

B.2.1.1 Registration of measurement components

Run c:\tk\tk.prg under MicrosoftVisualFoxPro6.0. Enter the 'Registration of measurement component' in the 'Coefficient calculation program'. Register the content of each element (oxide content) in the S sample, in the ratio column, such as: Fe: 0.6700; Si: 0.10; Ca: 0.065; Mg: 0.05; Al: 0.05; Ti: 0.015; Mn: 0.02; P: 0.02; Ba: 0.01. In the column of S amount, register the proportion of standard mixture S in each calibration sample, Fe: 0.50; Si: 0.50; Ca: 0.75; Mg: 0.75; Al: 0.75; Ti: 0.75; Mn: 0.75; P: 0.75; Ba: 0.75. Save the registered data and return it.

B.2.1.2 Registration of standard intensity

Enter 'Standard intensity registration'. Register the fluorescence intensity of each sample after subtracting the intensity of the blank sample, in the order of S sheet, Fe sheet, Si sheet, Ca sheet, Mg sheet, Al sheet, Ti sheet, Mn sheet, P sheet, Ba sheet. Close the database and return to the previous menu.

B.2.1.3 Calculation of correction factor

Return to the previous menu, after entering 'Correction factor calculation'. After the correction factor calculation is completed, click "Exit", to return to the main menu.

B.2.2 Content calculation

B.2.2.1 Registration of blank strength

Enter "Content calculation program" in the main menu. Enter 'Registration of blank intensity'. Record the X-ray fluorescence intensities of Si, Ca, Mg, Al, Ti, P in 100% Fe sheet and the X-ray fluorescence intensity of Fe, Mn, Ba in 100% Si sheet. Close the database and return to the previous menu.

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