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Determination of the content of attapulgite phase - X-ray diffraction standard curve method

凹凸棒石相含量的测定 X射线衍射-标准曲线法

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1 Scope

This document describes the principle of the method, the reagents, the instruments, equipment and materials, the environmental requirements and test conditions, the test procedure, the calculation of results and the permissible difference for the determination of the content of the attapulgite phase by the X-ray diffraction standard curve method.

This document applies to the determination of the content of the attapulgite phase.

2 Normative references

The contents of the following documents constitute indispensable provisions of this document through normative reference in the text. For dated references, only the edition corresponding to that date applies to this document; for undated references, the latest edition, including all amendments, applies to this document.

GB/T 6003.1 Test sieves - Technical requirements and testing - Part 1: Test sieves of metal wire cloth. GB/T 8170 Rules of rounding off for numerical values and expression and judgement of limiting values. JY/T 0587 General rules for polycrystalline X-ray diffraction methods.

3 Terms and definitions

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

4 Principle of the method

Different mineral crystals give different characteristic X-ray diffraction peaks, and the

intensity of those characteristic peaks is positively correlated with the content of the mineral in the sample.

A series of standard samples of known attapulgite phase content is prepared and mixed uniformly with an equal amount of the internal standard substance alpha-Al2O3. A set of intensity data can then be measured for the characteristic peaks of the attapulgite phase and of the alpha-Al2O3 internal standard phase, and a standard working curve is plotted.

By substituting the characteristic peak intensity data measured on the sample under test into the standard working curve, the attapulgite phase content of the sample under test is obtained.

The X-ray diffraction pattern of attapulgite clay is given in Figure 1.

5 Reagents, instruments, equipment and materials

Reagents. Internal standard substance alpha-Al2O3: purity greater than 99 percent. Amorphous SiO2: purity greater than 99 percent. Attapulgite certified reference material [GBW(E)070055]: certified value of the attapulgite phase content 85 percent.

Instruments, equipment and materials. X-ray diffractometer: source power not less than 1600 W, accuracy of the 2-theta angle better than 0.02 degrees. Analytical balance: precision not lower than 0.001 g. Electrically heated drying oven: temperature adjustment range from room temperature to 150 degrees Celsius, temperature control accuracy +/-3 degrees Celsius. Desiccator. Test sieve: aperture 0.075 mm, conforming to the requirements of GB/T 6003.1. Agate mortar. Weighing paper. Small spatula or glass plate.

6 Environmental requirements and test conditions

Environmental requirements. Temperature 22 degrees Celsius +/-2 degrees Celsius, relative humidity not greater than 70 percent.

Test conditions. The test conditions are given in Table 1. The X-ray tube is copper (Cu); monochromatization, that is removal of the K-beta line, is by a nickel filter; the tube voltage is 40 kV and the tube current 40 mA; the scanning range in 2-theta is 3 degrees to 50 degrees; the scanning speed is 2 degrees per minute and the step size 0.017 degrees.

7 Test procedure

Phase identification. For an unknown sample, phase analysis shall be carried out before the attapulgite phase content is analysed, in order to establish whether the sample contains an attapulgite phase. Phase analysis is carried out by the method specified in JY/T 0587.

Selection of the characteristic peak of attapulgite. The reflection at d(110) equal to 1.04 nm is selected as the characteristic peak for the attapulgite content.

Selection of the characteristic peak of the internal standard substance. The reflection at d(113) equal to 0.2086 nm is selected as the characteristic peak for the content of the internal standard substance alpha-Al2O3.

Preparation of the standard samples. Suitable amounts of the internal standard substance alpha-Al2O3, of the amorphous SiO2 and of the attapulgite certified reference material [GBW(E)070055] are placed in an electrically heated drying oven at 65 degrees Celsius +/-3 degrees Celsius and dried for 12 h, then removed and cooled to room temperature in a desiccator. Portions of 1.000 g, 0.800 g, 0.600 g, 0.400 g, 0.200 g, 0.100 g and 0.050 g of the attapulgite certified reference material [GBW(E)070055] are weighed out and mixed in turn with 0.000 g, 0.200 g, 0.400 g, 0.600 g, 0.800 g, 0.900 g and 0.950 g of amorphous SiO2. To each mixture 1.000 g of the internal standard substance alpha-Al2O3 is added, and each is ground until fully and uniformly mixed. The mixtures are wrapped in weighing paper and kept in a desiccator until measurement.

Preparation of the sample under test. The ore sample to be tested is first crushed to below 5 mm, placed in an electrically heated drying oven at 65 degrees Celsius +/-3 degrees Celsius and dried for 12 h, then removed and cooled to room temperature in a desiccator. It is ground until it all passes a test sieve of 0.075 mm aperture. 1.000 g of the sieved sample and 1.000 g of the internal standard substance alpha-Al2O3 are weighed into an agate mortar and ground until fully and uniformly mixed. The mixture is wrapped in weighing paper and kept in a desiccator until measurement.

Standard working curve. The specimens are made up by back loading. The series of standard samples prepared in 7.4 is placed in the specimen frame on a glass plate, pressed gently firm and flat with a small spatula or a glass plate, and put into the specimen holder of the X-ray diffractometer. The automatic system calibration and the setting of the test conditions are completed in turn, the pattern is scanned and saved, and a set of peak height intensities or integrated intensities is obtained for the attapulgite characteristic peak (110 reflection) and for the alpha-Al2O3 characteristic peak (113 reflection) of the standard samples. Taking as the abscissa the ratio of the peak height intensity (or integrated intensity) of the attapulgite phase characteristic peak of the series of standard samples to the peak height intensity (or integrated intensity) of the characteristic peak of the internal standard substance alpha-Al2O3, and as the ordinate the attapulgite phase content, the standard working curve for the determination of the attapulgite phase content is plotted.

Measurement. The specimen is made up by back loading. The sample under test prepared in 7.5 is placed in the specimen frame on a glass plate, pressed gently firm and flat with a small spatula or a glass plate, and put into the specimen holder of the X-ray diffractometer. The automatic system calibration and the test conditions are completed in turn, and the scan is set up so as to obtain the pattern of the sample under test.

8 Calculation of results

The attapulgite phase content y of the sample is calculated by formula (1), which is a straight-line relation of the form y equals k times x plus b.

In the formula: y is the attapulgite phase content of the sample, expressed as a percentage; k is a constant, not less than 0.99; x is the ratio of the peak height intensity (or integrated intensity) of the attapulgite phase characteristic peak of the sample to the peak height intensity (or integrated intensity) of the characteristic peak of the internal standard substance alpha-Al2O3; b is a constant.

The attapulgite phase content of the sample is the arithmetic mean of two parallel test results, rounded in accordance with GB/T 8170 to one significant figure after the decimal point.

9 Permissible difference

Where the mean of the two parallel test results is not greater than 15 percent, the absolute deviation shall be not greater than 2 percent. Where the mean of the two parallel test results is greater than 15 percent, the absolute deviation shall be not greater than 1 percent. Otherwise the determination shall be repeated.