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Determination of asbestos in products

制品中石棉含量测定方法

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Determination of asbestos in products

Warning - Users of this document shall have practical experience in formal laboratory work. This document does not address all possible safety issues. Users are responsible for implementing appropriate safety and health measures and ensuring compliance with relevant national regulations.

1 Scope

This document specifies the method overview and principles, reagents and materials, instruments and equipment, sample collection and preparation, qualitative and quantitative methods, test result reporting for the determination of asbestos content in products.

This document applies to the determination of asbestos content in building materials, friction materials, sealing materials, thermal insulation materials, other non-metallic minerals.

2 Normative references

The contents of the following documents, through normative references, constitute essential provisions of this document. 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 6682 Water for analytical laboratory use - Specification and test methods

3 Terms and definitions

The following terms and definitions apply to this document.

3.1

Asbestos

Among the minerals that constitute rocks, fibrous silicate minerals belonging to the serpentine group (chrysotile) and fibrous silicate minerals belonging to the amphibole group (amosite, crocidolite, tremolite, actinolite, anthophyllite).

3.2

Vermiculite

Immersion liquid

The liquid used to immerse the sample in microscopic qualitative analysis.

3.10

Vermiculite standard sample

This sample, made from vermiculite and used for the analysis of vermiculite spray materials, making the pure vermiculite contain 0.5% tremolite and 0.8% chrysotile.

3.11

Residual ratio

The ratio -- of the amount of the secondary analytical sample (residue) for quantitative determination, which is obtained after a series of treatments on the primary analytical sample, such as with formic acid, TO the amount of the primary analytical sample.

3.12

Weight loss ratio

The ratio -- of the amount of the primary analytical sample after heat treatment TO the total sample.

3.13

Integral intensity

The area integral value of a specified X-ray diffraction peak.

4 Symbols and abbreviations

The following symbols and abbreviations apply to this document (see Table 1).

5 Method overview and principle

5.1 Qualitative method overview and principle

5.1.1 Qualitative method overview

5.1.1.1 The presence of asbestos in the product is determined by qualitative analysis using X-ray diffraction and polarizing microscopy.

5.1.1.2 The presence of asbestos in the vermiculite spray material is determined by

Note: When performing quantitative analysis using an X-ray diffractometer, internal standard methods and standard addition methods can be used. Although these methods are less accurate for asbestos analysis, they can still be used to determine asbestos content.

6 Reagents and materials

6.1 Asbestos standard samples: Chrysotile, tremolite, actinolite, anthophyllite, amosite, crocidolite.

6.2 Vermiculite standard sample.

6.3 Impregnation solution: Refractive indices ($n_D^{25\,℃}$) of 1.550, 1.605, 1.630, 1.680, 1.700, respectively.

6.4 Formic acid: Analytically pure.

6.5 Water: Grade III water as specified in GB/T 6682.

6.6 Isopropyl alcohol: Analytically pure.

6.7 Potassium chloride aqueous solution: Analytically pure, concentration 1 mol/L.

6.8 Slides: Colorless, transparent glass. Cleanliness shall meet the requirements of surface cleanliness and be free of visible pits, particles, stones, scratches, cracks, etc.

6.9 Cover glass: Colorless, transparent glass. Cleanliness shall meet the requirements of surface cleanliness and be free of visible pits, particles, stones, scratches, cracks, etc.

6.10 Other items: Crucible, standard sieve, conical flask, dropper, pipette, brush, conductive tape (or double-sided tape), scissors, ear bulb, etc.

7 Instruments and equipment

7.1 X-ray diffractometer (XRD): The goniometer's angle measurement accuracy shall be better than 0.02° (2θ); the instrument's resolution shall be better than 60%; the overall stability shall be better than ±1%. The technical specifications of the X-ray diffractometer for qualitative and quantitative asbestos analysis shall comply with the requirements in Appendix A.

7.2 Polarizing light microscope (PLM): The specifications of the polarizing light microscope shall comply with the requirements in Appendix B.

7.3 High-temperature furnace: Meet the temperature range of room temperature to 600 °C, with an accuracy of not less than 10 °C.

7.4 Oven: Meet the temperature range of room temperature to 300 °C, with an accuracy of not less than 5 °C.

7.5 Magnetic stirrer.

7.6 High-speed centrifuge: Speed 5000 rpm ~ 15000 rpm.

7.7 Ultrasonic cleaner.

7.8 Balance.

7.9 Analytical balance: Accuracy of not less than 0.0001 g.

7.10 Constant temperature water bath: Meet the temperature range of room temperature to 100 °C, with an accuracy of not less than 0.5 °C.

7.11 Filter: Equipped with a 25 mm diameter fluororesin-bonded glass fiber filter.

7.12 Pulverizing equipment: Meet the test requirements specified in this document. Mortars (porcelain, agate, aluminum, etc.), Violet mills, ultra-centrifugal grinders, vibrating grinders, ball mills, etc. may be used, singly or in combination.

8 Sample collection

8.1 Sample collection method

8.1.1 Samples shall be collected from factory-produced or imported building materials, friction materials, sealing materials, insulation materials, non-metallic minerals. When collecting samples, the collector shall take appropriate protective measures to protect themselves from inhaling asbestos dust (hereinafter referred to as "dust").

8.1.2 When collecting samples at the production site, care should be taken to maintain the original condition and use sharp knives to collect samples, to avoid scattering dust.

8.1.3 When collecting samples from products, they shall be collected representatively from each batch.

8.1.4 The sample size shall be representative of the product being tested. For example, for soft materials such as spray coatings and insulation, 3 sets of samples, each approximately 10 cm^3 in volume, shall be collected from three different locations on the same batch of materials. These samples shall be placed in separate sealed containers. All samples shall then be placed in a larger sealed container, to serve as representative samples for measurement. For harder, plate-like materials, 3 sets of samples, totaling approximately 10 cm^2, shall be collected from 3 different locations on the same batch of materials. These samples shall then be placed in separate sealed containers. All samples shall then be placed in a larger sealed container, to serve as representative samples for analysis.

8.1.5 Depending on the length of construction, the building material product being tested may contain both asbestos-containing and non-asbestos-containing portions. Furthermore, during maintenance work, some asbestos-containing products may have been replaced with non-asbestos-containing products. Taking these factors into account, the sampling location shall be selected based on construction records such as design documents.

8.1.6 When collecting samples with low asbestos content or a wide range of content variation (such as spray coatings), sampling shall encompass the entire depth of the product, avoiding sampling only the surface. This method is also used for sampling insulation materials exposed to high temperatures for extended periods.

8.1.7 Paste or liquid products used for joints or repairs may contain asbestos. Samples shall be collected only from the asbestos-containing areas (usually joints).

8.2 Sample delivery and custody

When transporting samples from the collection site to the analysis site, they shall be placed in sealed containers.

8.3 Sample collection records

To identify the collected samples, the following information shall be recorded:

a) Product name;

b) Collection location and address;

c) Sample information: Shape, material, sample area, sample volume, collection method, collection time, etc.;

d) Collector.

9 Sample preparation

9.1 Preparation for primary analytical sample

9.1.1 Preparation for primary analytical sample of inorganic component

9.1.1.1 From the 3 collected inorganic component samples, equal amounts of analytical samples shall be extracted and pulverized in a pulverizer. If the sample is hard, scrape the sides with a knife, planer, or other similar tool; place the scraped portion into a pulverizer. This pulverizer can be a mortar (porcelain, agate, or aluminum mortar), a Vierli mill, an ultra-centrifugal grinder, a vibrating grinder, or a ball mill, either singly or in combination.

9.1.1.2 Sieve the pulverized sample through a standard sieve with a pore size of 425

9.2.1.2 Accurately weigh 100 mg (m_1: weight of the primary analytical sample) of the primary analytical sample into a conical flask. Add 20 mL of 20% formic acid and 40 mL of water. Disperse the sample using an ultrasonic cleaner for 1 min.

9.2.1.3 Place the conical flask in a constant temperature water bath at (30 ± 1) °C; vibrate continuously for 12 min.

9.2.1.4 Perform extraction filtration and drying using an extraction filtration device equipped with a 25 mm diameter filter. Use an extraction filtration device and filter with the same diameter as the sample stage of the X-ray diffractometer.

9.2.1.5 After drying, weigh the total weight of the filter and sample; calculate the mass of the sample collected on the filter (m_2: the weight of the secondary analytical sample for quantitative determination). If the residual ratio (m_2/m_1) is ≤ 15%, use the sample as the secondary analytical sample for quantitative determination.

9.2.1.6 If the residual ratio (m_2/m_1) is > 15%, prepare a tertiary analytical sample for quantitative determination according to 9.2.2.

9.2.2 Preparation of tertiary analytical sample for quantitative determination

9.2.2.1 If the residual ratio of the sample prepared in 9.2.1 is greater than 15%, take 10 mg ~ 15 mg and dissolve it in water.

9.2.2.2 Perform extraction filtration using an extraction filtration apparatus equipped with a filter of known mass; dry it.

9.2.2.3 After drying, weigh the total weight of the filter and sample; calculate the mass of the sample collected on the filter (m_3: the weight of the tertiary analytical sample for quantitative determination). Use the tertiary analytical sample for quantitative determination.

9.2.2.4 The following precautions shall be taken, when preparing tertiary analytical samples for quantitative determination:

- For inorganic component samples whose mass decreases after high-temperature treatment as specified in 9.1.2.2, the primary analytical sample after high-temperature treatment can also be used to prepare the secondary analytical sample for quantitative determination.

- If the amount of secondary analytical sample for quantitative determination is insufficient to prepare the tertiary analytical sample for quantitative determination, the primary analytical sample shall be prepared as a secondary analytical sample under the same conditions as the secondary analytical sample preparation method, for quantitative determination; then make preparation.

10 Qualitative methods

10.1 X-ray diffraction method

10.1.1 Weigh 3 sets of the primary analytical sample as needed; place them separately into conical flasks. Add 20 mL of 20% formic acid solution and 40 mL of water per 100 mg of sample. Disperse the sample using an ultrasonic cleaner for 1 minute.

10.1.2 Place the conical flask in a constant-temperature water bath at (30 ± 1) °C; shake continuously for 12 minutes. Filter the sample using an extraction and filtration apparatus equipped with a 0.8 μm pore size white membrane filter and a 25 mm diameter filter screen; dry it. The dried sample will be analyzed by X-ray diffraction.

Note: If, based on product information and other data, the sample collected is expected to have a high asbestos content, then the primary analytical sample can be used directly for X-ray diffraction analysis.

10.1.3 Place the sample prepared in 10.1.2 into a sample dish, ensuring that the sample surface is uniform and flush with the surface of the dish. The sample dish is a metal or glass plate with holes or pits.

10.1.4 Perform X-ray diffraction analysis on the sample dish containing the sample, according to the qualitative analysis conditions specified in Appendix A.

10.1.5 Observe the X-ray diffraction pattern of the sample, to confirm the presence of characteristic peaks for asbestos (chrysotile, amosite, crocidolite, tremolite/actinolite, anthophyllite) as shown in Figures 4 ~ 8 or for the vermiculite group (hydrobiotite, vermiculite) as shown in Figures 9 and 10.

Note: Tremolite and actinolite are difficult to distinguish by X-ray diffraction; they can be classified as the same categories during analysis.

10.1.6 To confirm characteristic diffraction peaks, compare the X-ray diffraction pattern of the sample with that of an asbestos standard sample under the same conditions. Alternatively, use ICDD to examine all characteristic peaks.

Note: Characteristic X-ray diffraction peaks for asbestos are a peak near 10° ~ 12° or a first or second diffraction peak near 24° ~ 30°.

10.1.7 If the asbestos type is determined through qualitative analysis, or if similar X-ray diffraction peaks of asbestos appear, subsequent analysis shall be conducted in accordance with the provisions of 10.2.

10.1.8 If similar X-ray diffraction peaks of vermiculite appear, subsequent analysis shall be conducted in accordance with the provisions of 10.3.

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