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Method of Identification for Asbestos Fibers in Cosmetics

1. Scope

This method is applicable to the identification of asbestos fibers including actinolite, amosite, anthophyllite, chrysotile, crocidolite, and tremolite in cosmetics.

2. Method

After cleaning with solvent and dry ashing, asbestos fibers are identified by transmission electron microscopy (TEM) or scanning electron microscope (SEM) coupled with energy dispersive X-ray spectroscopy (EDS).

2.1. Equipment

2.1.1. Transmission electron microscope：JEOL JEM-1400F, or an equivalent product.

2.1.2. Scanning electron microscope：JEOL JSM-7000F, or an equivalent product.

2.1.3. Energy dispersive X-ray spectroscope：Oxford X-max^N 80T, or an equivalent product.

2.1.4. Shaker.

2.1.5. Furnace.

2.1.6. Centrifuge：centrifuge force ≥ 3000 ×g.

2.1.7. Oven.

2.2. Chemicals

Acetone, reagent grade;

Formic acid, reagent grade;

Deionized water, resistivity ≥ 18 MΩ·cm (at 25°C);

Actinolite, amosite, anthophyllite, chrysotile, crocidolite and tremolite, reference standards.

2.3. Apparatus

2.3.1. Centrifuge tubes：20 mL, glass.

2.3.2. Crucibels：20 mL, ceramic.

2.3.3. Gold Grids：200 mesh.

2.3.4. Antimagnetic tweezers.

2.3.5. Lens-wiping paper.

2.4. 20% formic acid

Mix 20 mL of formic acid with deionized water to 100 mL.

2.5. Standard grids preparation

Transfer about 10 mg of actinolite, amosite, anthophyllite, chrysotile,

crocidolite and tremolite reference standards into each centrifuge tube separately, add 2 mL of 20% formic acid and 4 mL of deionized water, and mix well. Transfer 3 μL of the dispersion solution from each centrifuge tube onto a gold grid. After standing still, remove excess liquid with lens-wiping paper, and dry at 50°C for 1-3 days as the standard grids for identification.

2.6. Sample preparation

2.6.1. Transfer 2 g of the well-mixed sample into a 20 mL centrifuge tube. Add 8 mL of acetone, shake for 5 min, and then centrifuge at 3000 ×g for 5 min. Discard the supernatant, and add 8 mL of acetone into the residue, and repeat the above steps 3 times. Collect and air dry the residue as the sample for ashing.

2.6.2. Transfer the sample for ashing from section 2.6.1. into a crucible, and ash at 450°C for 2 hrs. After cooling to room temperature, weigh the crucible, and record the weight.

2.6.3. Transfer 10 mg of the ashed sample from section 2.6.2. to a centrifuge tube. Add 2 mL of 20% formic acid and 4 mL of deionized water, and mix well. Transfer 3 μL of the dispersion solution on a gold grid. After standing still, remove excess liquid with lens-wiping paper, and dry at 50°C for 1-3 days as the sample grid. Prepare 2 grids for each sample for identification.

2.7. Identificaiton

Analyze the sample and the standard grids by the transmission electron microscope or the scanning electron microscope separately. Operate according to the following conditions. Look for a fiber that has the length greater than 5 μm and the aspect ratio greater than or equal to 3:1 on the grid. Observe 20 grid openings for each grid, and investigate totally 40 grid openings for each sample grid. The fibers conforming to the length and aspect ratio are further analyzed by the energy dispersive X-ray spectroscope for elemental analysis. Identify asbestos fibers based on the elemental peaks and the abundance ratios comparing with those of the reference standards listed in the attached table.

Transmission electron microscope operating conditions(note)：

Voltage: 80-120 KV.

Magnification: 1000-5000 X.

Scanning electron microscope operating conditions(note)：

Voltage: 10 KV.

Magnification: 500-1500 X.

Energy dispersive X-ray spectroscope operating conditions(note)：

Voltage: 80-120 KV.

Spot size: 0.7 nm.

Detection time：30 sec.

Note：All the parameters can be adjusted depending on the instruments used if the above conditions are not applicable.

Remark

Further validation should be performed when interference compounds appear in the samples.

Reference

1. Ministry of Labor, Republic of China (Taiwan). 2018. Standards of Permissible Exposure Limits at Job Site. 2018.03.14 modified.
2. Japan industrial standards research council. 2016. Method of asbestos content in building materials-Part 1: The method of sample selection and qualitative determination of commercially available bark materials. JIS A1481-1.
3. International Organization for Standardization. 2014. Air quality - Bulk materials -Part 1: Sampling and qualitative determination of asbestos in commercial bulk materials. ISO 22262-1.

Table. Chemical formulas and elemental composition of asbestos fibers

Asbestos fiber	Identification of elemental composition(note)
Actinolite formula：$Ca_2(Mg,Fe^{2+})_5Si_8O_{22}(OH)_2$ (Mg/(Mg+Fe) = 0.5-0.9)	1. The ratio of Mg, Si, Fe is consistent with the reference standard. 2. The peak of Na or Al is not obvious. 3. The peak of Mn is small or absence.
Amosite formula：$(Fe^{2+},Mg)_7Si_8O_{22}(OH)_2$	1. The ratio of Mg, Si, Fe is consistent with the reference standard. 2. The peak of Na or Al is not obvious. 3. The peak of Mn is small or absence.
Anthophyllite formula：$(Mg,Fe^{2+})_7Si_8O_{22}(OH)_2$	1. The ratio of Mg, Si is consistent with the reference standard. 2. The peak of Fe is present or absence. 3. The peak of Na or Al is not obvious. 4. The peak of Mn is small or absence.
Chrysotile formula：$Mg_3Si_2O_5(OH)_4$	1. The peaks of Mg, Si are clear. 2. The ratio of Mg, Si is consistent with the reference standard. 3.The peaks of Fe, Mn, Al are small.
Crocidolite formula：$Na_2(Fe^{2+},Mg)_3Fe^{3+}{}_2Si_8O_{22}(OH)_2$	1. The ratio of Mg, Si, Fe is consistent with the reference standard. 2. The peak of Al is not obvious 3. The peak of Mg is small, Mn is absence.
Tremolite formula：$Ca_2(Mg,Fe^{2+})_5Si_8O_{22}(OH)_2$ (Mg/(Mg+Fe) = 1.0-0.9)	1. The ratio of Mg, Si, Ca, Fe is consistent with the reference standard. 2. The peak of Al is not obvious . 3. The peak of Na or K is small.

Note：1. The EDS spectrum of the asbestos fibers in the sample may show other elemental peaks when they are attached with other substances.

2. The EDS spectra of asbestos fiber standards can refer to ISO 22262-1.