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Determination of plastic microbead in cosmetics

化妆品中塑料微珠的测定

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Determination of plastic microbead in cosmetics

1 Scope

This Standard specifies the principle, reagents and materials, instruments and equipment, specimen processing, spectrum testing, spectrum analysis and result reporting for Fourier transform infrared spectroscopy, Fourier transform micro-infrared spectroscopy that are used to determine the plastic microbead in cosmetics.

This Standard is applicable to the determination of plastic microbead in cosmetics. Toothpaste, soap, laundry detergent and other daily chemical products can refer to it as reference.

This Standard is inapplicable to the determination of plastic microbead in make-up cosmetics.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

GB/T 1914, *Chemical analytical filter paper*

GB/T 6003.1, *Test sieves - Technical requirements and testing - Part 1: Test sieves of metal wire cloth*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1 plastic microbead

Solid plastic particles less than or equal to 5mm in size and insoluble in water.

4 Principle

Use an appropriate solvent to disperse the sample. Filter it through a stainless-steel test sieve with a mesh size of 5mm. The filtrate is filtered through filter

6.5 Agate mortar.

6.6 Suction filtration device.

7 Specimen processing

7.1 General

7.1.1 Selection of sample processing method

Weigh about 5g representative sample into a 250mL beaker. Add 100mL of hot water and stir. Observe the dispersion state of the sample in water. In the case of complete dispersion, process according to 7.2.1. When dispersing in the case of flocculation in water, process according to 7.2.2. When dispersing in the case of water producing a lot of foam, process according to 7.2.3. If it is not dispersed in water, process in accordance with 7.2.4.

7.1.2 Auxiliary processing method

Due to the complexity of the cosmetic matrix, in order to take into account, the sample processing effect and efficiency, the following auxiliary processing methods can be used:

- If the sample contains wax, colloid and it is difficult to filter, it can be dissolved in three times with no less than 150mL of solvent. Directly discard liquids containing wax or colloids by standing or centrifuging. Collect the solids and proceed to sample processing;
- If there are bubbles during suction filtration, add a small amount of isopropanol (5.3) to defoam;
- If it is obvious that there are no solid particles larger than 5mm by visual inspection, the step of filtration by the stainless-steel test sieve can be omitted;
- If the obtained solid particles have matrix (such as wax, colloid) that interferes with infrared qualitative analysis, it can use an appropriate amount of ethanol aqueous solution (5.6) and n-hexane (5.2) to wash the solid particles in sequence. Conduct suction drying. Collect solid particles to be tested.

7.2 Sample processing

7.2.1 Method 1

Weigh about 5g of sample into a 250mL beaker. Add 5mL of water. Use a glass rod to keep stirring, so as to break and disperse suspended particles. Then add

the substrate is rinsed clean. Then use 30mL of hot absolute ethanol (5.1) to rinse the insoluble matter in sessions. Conduct suction filtration. If there are no solid particles on the medium-speed qualitative filter paper, the filtrate shall be filtered with the slow qualitative filter paper (5.9). Use not less than 100mL of hot 95% ethanol (5.5), not less than 100mL of hot water and 30mL of hot absolute ethanol (5.1) to rinse sequentially. After the suction filtration, place the filter paper and insoluble matter in the original beaker. Dry in an oven (6.4) at 60°C. Place in a desiccator to room temperature. Collect the solid particles to be tested.

7.2.4 Method 4

Weigh about 5g of the specimen (in the case of matrix interference in the sample, such as wax, colloid, the weighing amount can be appropriately reduced, but not less than 1g) in a 250mL beaker. Add 5mL of n-hexane (5.2). Use a glass rod to keep stirring to break and disperse the suspended particles. Then add 95mL of n-hexane (5.2). Stir well. Use the stainless-steel test sieve (5.7) to filter. Collect the filtrate in a beaker. After standing still, use the decant method to suction and filter through the medium-speed qualitative filter paper (5.8). Drain the clear liquid as much as possible. Try to keep the insoluble matter in the beaker. Use not less than 100mL of n-hexane (5.2) in sessions to repeat the pouring method to rinse the insoluble matter in the beaker. Conduct suction filtration. Then put the beaker in a 60°C water bath to heat and evaporate. Then add 100mL of water. Place it in a 60°C water bath and heat to disperse. Stand still. Conduct suction filtration while it is hot. Use no less than 100mL of hot water in sessions to rinse the insoluble matter until the substrate is rinsed clean. Then use 30mL of hot absolute ethanol (5.1) in sessions to rinse the insoluble matter. Conduct suction filtration. If there are no solid particles on the medium-speed qualitative filter paper, the n-hexane filtrate and water filtrate shall be filtered with the slow qualitative filter paper (5.9) in sequence. Use not less than 100mL of n-hexane (5.2), not less than 100mL of hot water and 30mL of hot absolute ethanol (5.1) to rinse in sequence. After the suction filtration, place the filter paper and insoluble matter in the original beaker. Dry in an oven (6.4) at 60°C. Place in a desiccator to room temperature. Collect the solid particles to be tested.

8 Spectral tests

8.1 Fourier transform infrared spectroscopy

8.1.1 Sample preparation

8.1.1.1 Potassium bromide tablet method

Take 1mg~2mg of solid particles and about 100mg of potassium bromide (5.4)

Due to the small sampling amount of micro-infrared test samples and a single test is not representative, different particles shall be selected for multiple tests according to the actual situation of the sample.

9 Spectral analysis

9.1 Computer search

Use Fourier transform infrared spectrometer computer retrieval system for spectrogram retrieval and analysis. According to the search results, use the computer retrieval system to compare the spectra with the collected spectra for qualitative analysis. It shall be noted that the search results are affected by the purity of the sample, the structure of the compound, and the spectral library.

9.2 Reference spectrum analysis

Use the test conditions same with the sample to determine based on the known plastics OR obtain the reference spectrum by consulting the data. Compare the infrared spectrum of the sample with the reference spectrum. Conduct qualitative analysis based on the position, number, relative intensity and shape of its characteristic absorption peaks. Annex A shows the infrared spectrum and absorption characteristics of some plastics.

10 Result report

According to sample processing and Fourier transform infrared spectroscopy or Fourier transform micro-infrared spectroscopy analysis, report the results:

- When the presence of plastic is confirmed according to the spectral analysis in Chapter 9, report the color and type of plastic microbeads;
- When solid particles are not collected according to Chapter 7 sample processing, or although solid particles are collected but plastics are not detected according to Chapter 9 spectrum analysis, report it as "not detected".

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