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Determination of restricted dichloromethane and 1,1,1-trichloroethane in cosmetics - Headspace gas chromatography

化妆品中限用物质二氯甲烷和 1,1,1-三氯乙烷的测定

顶空气相色谱法

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Determination of restricted dichloromethane and 1,1,1-trichloroethane in cosmetics - Headspace gas chromatography

1 Scope

This Standard specifies reagents and materials, instruments and equipment, analytical procedures, calculation of results, recovery and precision, allowable difference, etc. of headspace gas chromatography for dichloromethane and 1,1,1-trichloroethane in cosmetics.

This Standard is applicable to the determinations of dichloromethane and 1,1,1-trichloroethane in cream, milk, liquid, water cosmetics.

The detection limits of this Standard for dichloromethane and 1,1,1-trichloroethane are 0.070mg/kg and 0.0010mg/kg, respectively. The limits of quantification are 0.25mg/kg and 0.0050mg/kg, respectively.

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 6682, *Water for analytical laboratory use - Specification and test methods*

3 Principle

The specimen is placed in a sealed headspace vial. At a certain temperature and time equilibrium, the target compound in the specimen reaches dynamic equilibrium in the gas-liquid two phases. At this time, the concentration of the target compound in the gas phase is proportional to its concentration in the liquid phase. By measuring the concentration of the target compound in the gas phase, the concentration of the target compound in the specimen can be calculated.

Weigh 10.00g of (to the nearest of 0.01g) sample directly into a 50mL volumetric flask. Add methanol to the scale. Cover to seal. Vortex on a vortex shaker until the sample is completely dissolved or evenly mixed. Pipette 10.0mL of sample solution into the headspace vial. Take 1mL of upper gas after gas-liquid equilibrium into gas chromatography for detection.

6.2 Headspace sampler conditions

The headspace sampler conditions are as follows:

a) Vaporization chamber temperature: 85°C; dosing tube temperature: 90°C; transmission line temperature: 100°C;

b) Gas-liquid equilibrium time: 40min; injection time: 1min.

6.3 Gas chromatographic reference conditions

The gas chromatographic reference conditions are as follows:

a) Chromatographic column: HP-VOC, 30m × 0.2mm × 1.1μm, or the equivalent;

b) Column temperature: keep at 40°C for 2 min, ramp to 120°C at a rate of 20°C/min (keep for 0min), and then ramp to 180°C at a rate of 10°C/min (for 5min);

c) Inlet temperature: 250°C;

d) Split ratio: 1:5;

e) Detector temperature: 300°C;

f) Carrier gas: nitrogen;

g) Flow rate: 1.0mL/min.

6.4 Standard working curve drawing

Respectively pipette 10mL of standard working solution at each level in the headspace bottle. Detect by 6.3 chromatographic conditions. Take the concentrations of working solutions at various levels (dichloromethane concentrations are 0.05μg/mL, 0.10μg/mL, 0.50μg/mL, 1.0μg/mL, 5.0μg/mL, 10μg/mL, the concentrations of 1,1,1-trichloroethane are 0.0010μg/mL, 0.0020μg/mL, 0.010μg/mL, 0.020μg/mL, 0.20μg/mL, 0.50μg/mL) as the abscissa, corresponding peak areas as ordinate. Perform linear regression to obtain standard curve equation.

For gas chromatograms of dichloromethane and 1,1,1-trichloroethane standard products, see Figure B.1 in Annex B.

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