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Method of Test for Iodopropynyl Butylcarbamate in Cosmetics

1. Scope

This method is applicable to the determination of Iodopropynyl butylcarbamate in cosmetics.

2. Method

After extraction, Iodopropynyl butylcarbamate is determined by gas chromatograph/tandem mass spectrometer (GC-MS/MS).

2.1. Equipment

2.1.1. Gas chromatograph/tandem mass spectrometer.

2.1.1.1. Ion source: electron ionization, EI.

2.1.1.2. Column: HP-5MS UI capillary column, 0.25 μm, 0.25 mm i.d. × 30 m, or an equivalent product.

2.1.2. Ultrasonicator.

2.2. Chemicals

Acetone, HPLC grade;

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

Iodopropynyl butylcarbamate, reference standard.

2.3. Apparatus

2.3.1. Volumetric flask: 20 mL.

2.3.2. Membrane filter: 0.22 μm, Nylon.

2.4. Standard solution preparation

Transfer about 20 mg of iodopropynyl butylcarbamate reference standard accurately weighed into a 20-mL volumetric flask, dissolve and dilute to volume with acetone as the standard stock solution. When to use, transfer appropriate volume of the standard stock solution, and dilute with acetone to 10 -100 ng/mL as the standard solutions.

2.5. Sample solution preparation

Transfer about 1 g of the well-mixed sample accurately weighed into a 20-mL volumetric flask. Add 10 mL of acetone, ultrasonicate for 30 min, and then dilute to volume with acetone. Filter with a membrane filter, take the filtrate as the sample solution.

2.6. Identification and quantification

Accurately inject 2 μL of the sample solution and standard solutions into GC-MS/MS separately, and operate according to the following conditions. Identify iodopropynyl butylcarbamate based on the retention time and the relative ion

intensities(note 1). Calculate the amount of iodopropynyl butylcarbamate in the sample by the following formula:

The amount of iodopropynyl butylcarbamate in the sample (%) = $\frac{C \times V}{M} \times 10^{-7}$

where,

C: the concentration of iodopropynyl butylcarbamate in the sample solution calculated by the standard curve (ng/mL)

V: the final make-up volume of the sample (20 mL)

M: the weight of the sample (g)

GC-MS/MS operating conditions(note 2):

Column: HP-5MS UI capillary column, 0.25 μm, 0.25 mm i.d.× 30 m.

Column temperature:

Initial temperature: 50℃, 1 min;

Temperature rising rate: 25℃/min;

Middle temperature 1: 150℃, 1 min;

Temperature rising rate: 25℃/min;

Middle temperature 2: 200℃, 2 min;

Temperature rising rate: 30℃/min;

Final temperature: 290℃, 2 min.

Injector temperature: 300℃.

Carrier gas and flow rate: helium, 2 mL/min.

Injection volume: 2 μL.

Ion source temperature: 300℃.

Ionization mode: EI, 70 eV.

Injection mode: splitless.

Detection mode: multiple reaction monitoring (MRM). Detection ion pair and collision energy are as follows.

Analyte	Ion pair	Collision energy (eV)
	Precursor ion (*m/z*) > Product ion (*m/z*)	
Iodopropynyl butylcarbamate	165 > 38*	10
	182 > 154	10

*Quantitative ion pair.

Note: 1. Relative ion intensities are calculated by peak areas of qualitative

ions divided by peak areas of quantitative ions. Maximum permitted tolerances of relative ion intensities are as follows:

Relative ion intensity (%)	Tolerance (%)
> 50	± 10
> 20-50	± 15
> 10-20	± 20
≤ 10	± 50

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

Remark

1. Limit of quantitation is 0.00002%.
2. Further validation should be performed when interference compounds appear in samples.

Reference

Celeiro, M., Lamas, J. P., Llompart, M. and Garcia-Jares, C. 2014. In-vial micro-matrix-solid phase dispersion for the analysis of fragrance allergens, preservatives, plasticizers, and musks in cosmetics. Cosmetics 1: 171-201.

Reference chromatogram

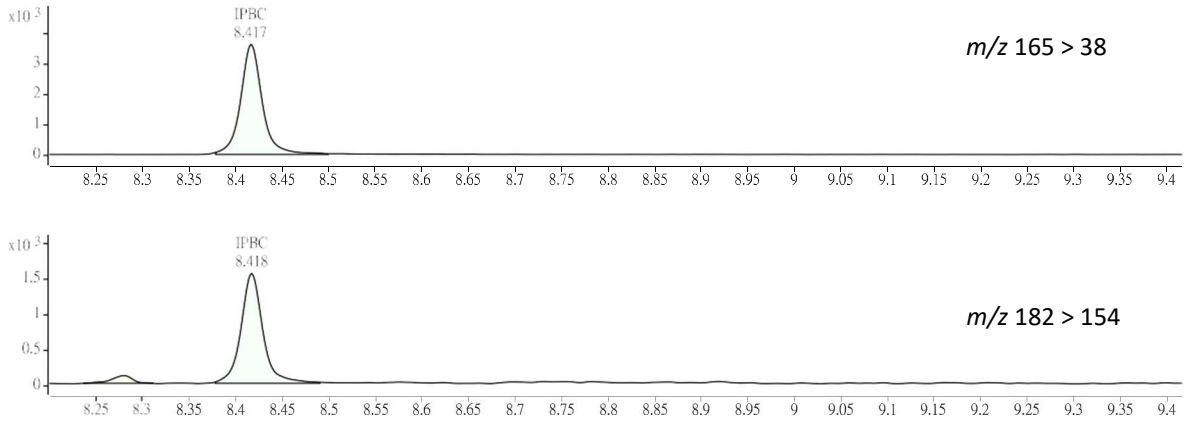

Figure. MRM chromatogram of iodopropynyl butylcarbamate analyzed by GC-MS/MS.