

Method of Test for Polysilicone-15 and Tris-biphenyl triazine in Cosmetics

1. Scope

This method is applicable to the determination of polysilicone-15 and tris-biphenyl triazine in cosmetics.

2. Method

After extraction, analytes are determined by high performance liquid chromatography (HPLC).

2.1. Equipment

2.1.1. High performance liquid chromatograph.

2.1.1.1. Detector: photodiode array detector.

2.1.1.2. Column: Eclipse plus C18, 5 μm , 4.6 mm i.d. \times 15 cm, or an equivalent product.

2.1.2. Ultrasonicator.

2.2. Chemicals

Isopropanol, HPLC grade;

Tetrahydrofuran, HPLC grade;

Acetonitrile, HPLC grade;

Deionized water, resistivity $\geq 18 \text{ M}\Omega \cdot \text{cm}$ (at 25°C);

Polysilicone-15 and tris-biphenyl triazine, reference standards.

2.3. Apparatus

2.3.1. Volumetric flask: 10 mL.

2.3.2. Membrane filter: 0.22 μm , PVDF.

2.4. Extraction solution

Mix acetonitrile and tetrahydrofuran at the ratio of 1:4 (v/v).

2.5. Standard solution preparation

Transfer about 50 mg of polysilicone-15 and 10 mg of tris-biphenyl triazine reference standards accurately weighed into each 10-mL volumetric flask, dissolve and dilute with the extraction solution to volume as the standard stock solutions. When to use, mix appropriate amount of each standard stock solution, and dilute with the extraction solution to 25-1000 $\mu\text{g/mL}$ for polysilicone-15, and 1-50 $\mu\text{g/mL}$ for tris-biphenyl triazine as the standard solutions.

2.6. Sample solution preparation

Transfer about 1 g of the well-mixed sample accurately weighed into a 10-mL volumetric flask, add 5 mL of the extraction solution, and ultrasonicate for 30

mins. Dilute to volume with the extraction solution, and filter with a membrane filter. Take the filtrate as the sample solution.

2.7. Identification and quantitation

Accurately inject 5 µL of the sample solution and the standard solutions into HPLC separately, and operate according to the following conditions. Identify each sunscreen agent based on the retention time and the UV absorption spectrum. Calculate the amount of each sunscreen agent in the sample by the following formula:

$$\text{The amount of each sunscreen agent in the sample (\%)} = \frac{C \times V}{M} \times 10^{-4}$$

where,

C: the concentration of each sunscreen agent in the sample solution calculated by the standard curve (µg /mL)

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

M: the weight of sample (g)

HPLC operating conditions ^(note):

Photodiode array detector: the quantitative wavelength 312 nm.

Column: Eclipse plus C18, 5 µm, 4.6 mm i.d. × 15 cm.

Mobile phase: a gradient program of solvent A (deionized water), solvent B (isopropanol), and solvent C (acetonitrile) is as follows.

Time (min)	A (%)	B (%)	C (%)
0 → 5	35 → 0	60 → 100	5 → 0
5 → 10	0 → 0	100 → 100	0 → 0
10 → 16	0 → 15	100 → 80	0 → 5
16 → 21	15 → 35	80 → 60	5 → 5
21 → 26	35 → 35	60 → 60	5 → 5

Flow rate: 0.8 mL/min.

Injection volume: 5 µL.

Column temperature: 25°C.

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

Remark

1. Limits of quantification (LOQs) are 0.025% for polysilicone-15, and 0.001% for tris-biphenyl triazine.

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

Reference

1. Scientific Committee on Consumer Safety. 2010. Opinion on polysilicone-15. [http://ec.europa.eu/health/scientific_committees/consumer_safety/index_en.htm.]
2. Hüglin, D. 2016. Advanced UV absorbers for the protection of human skin. SCS Laureates and Awards & SCS Fall Meeting. Chimia 70: 496-501.

Reference chromatogram

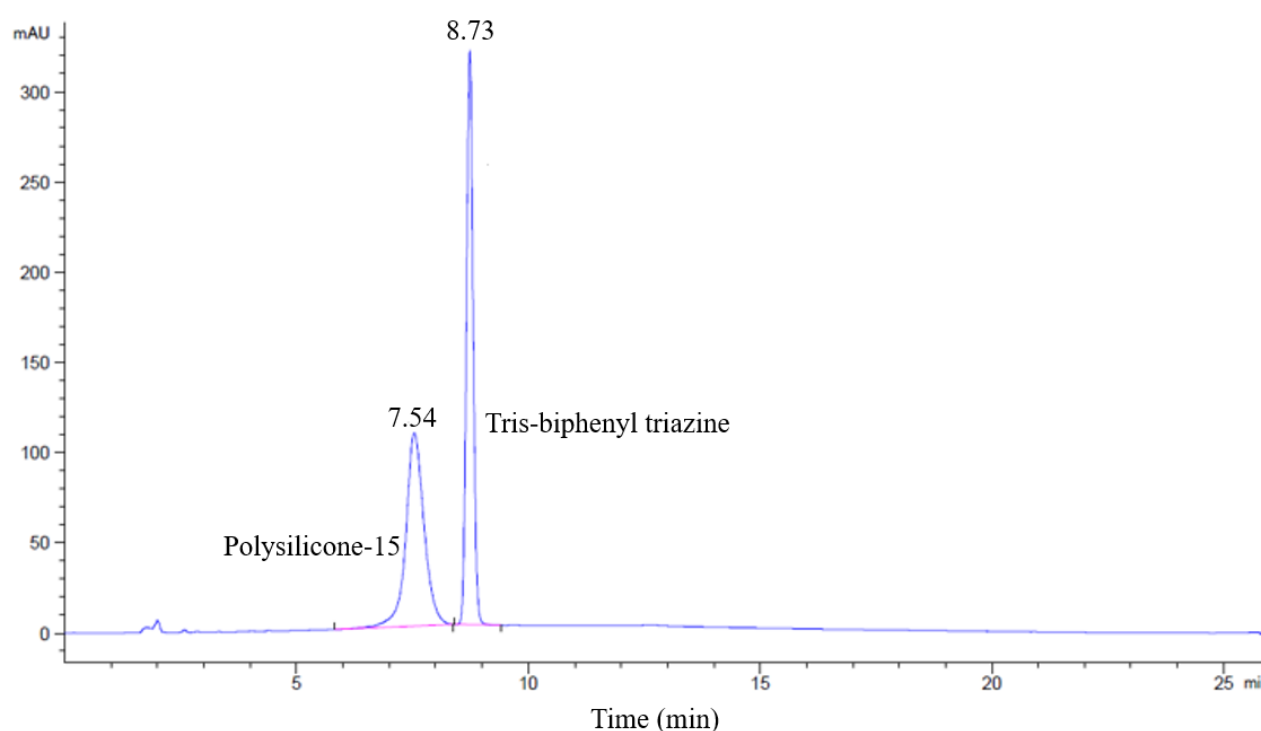


Figure. HPLC chromatogram of polysilicone-15 and tris-biphenyl triazine reference standards.