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Method of Test for Dichlorophen, Bithionol and Hexachlorophene in Cosmetics

1. Scope

This method is applicable to the determination of dichlorophen, bithionol and hexachlorophene 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: Lichrospher 100 RP-18, 5 μm, 4.0 mm i.d. × 25 cm, or an equivalent product.

2.1.2. Ultrasonicator.

2.2. Chemicals

Methanol, HPLC grade;

Acetic acid, reagent grade;

Deionized water, resistivity ≧ 18 MΩ • cm (at 25°C);

Dichlorophen, bithionol and hexachlorophene, reference standards.

2.3. Apparatus

2.3.1. Volumetric flask: 10 mL, 20 mL.

2.3.2. Membrane filter: 0.45 μm, Nylon.

2.4. 1%(v/v) Acetic acid solution

Dilute 1 mL of acetic acid with deionized water to 100 mL.

2.5. Mobile phase

Mix methanol and 1%(v/v) acetic acid solution at the ratio of 9:1 (v/v), and filter with a membrane filter.

2.6. Standard solution preparation

Transfer about 10 mg of dichlorophen, bithionol, and hexachlorophene reference standards accurately weighed into each 10-mL volumetric flask, dissolve and dilute with methanol to volume as the standard stock solutions. When to use, mix appropriate amount of each standard stock solution, and dilute with methanol to 0.1-50 μg/mL as the standard solutions.

2.7. Sample solution preparation

Transfer about 1 g of the well-mixed sample accurately weighed into a 20-mL

volumetric flask, add 10 mL of methanol, and ultrasonicate for 30 mins. Dilute to volume with methanol and filter with a membrane filter. Take the filtrate as the sample solution.

2.8. Identification and quantification

Accurately inject 20 μL of the sample solution and the standard solutions into HPLC separately, and operate according to the following conditions. Identify each analyte based on the retention time and the UV absorption spectrum. Calculate the amount of dichlorophen, bithionol, and hexachlorophene in the sample by the following formula (ppm):

The amount of dichlorophen, bithionol, and hexachlorophene in the sample (ppm) $= \frac{C \times V}{M}$

where,

C: the concentration of dichlorophen, bithionol, and hexachlorophene 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：quantitative wavelength 300 nm

Column: Lichrospher 100 RP-18, 5 μm, 4.0 mm i.d. × 25 cm.

Mobile phase: as section 2.5.

Flow rate: 0.8 mL/min.

Injection volume: 20 μL.

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) for dichlorophen, bithionol, and hexachlorophene are 2 ppm.
2. Further validation should be performed when interference compounds appear in samples.

Reference

1. Cheng, S. S., Su, S. C., Lin, Y. Y., Liao, C. H. 1999. Investigation of hexachlorophene in cosmetics. Annual report of Bureau of Drug and Food Analysis, 17, 193-197.

2. Tokunaga, H., Ko, R., Uchino, T. and Ando, M. 2002. Studies for identifying prohibited ingredients such as bithionol and dichlorophen in cosmetics. J. Soc. Cosmet. Chem. Jpn. 36: 111-118.

Reference chromatogram

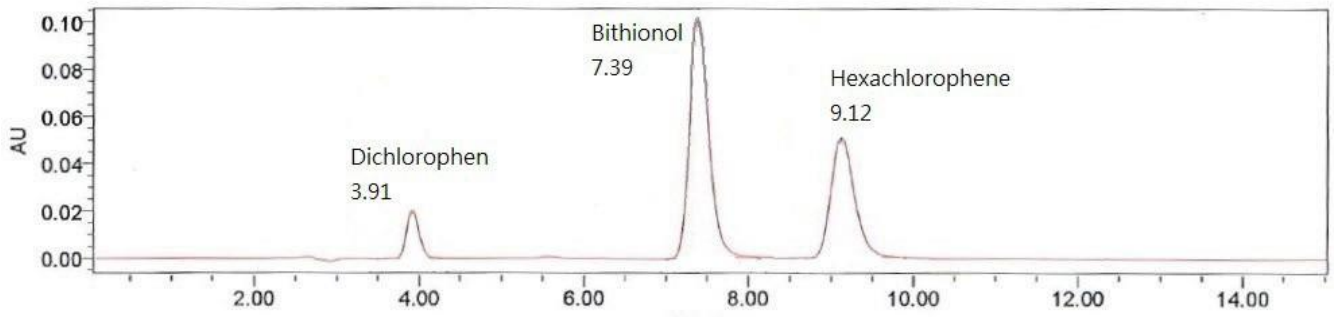

Figure. HPLC chromatogram of dichlorophen, bithionol and hexachlorophene standards.