
Kozmetika - Analiza kozmetičnih izdelkov - Določevanje 3-jodo-2-propinilbutilkarbamata (IPBC) v kozmetičnih pripravkih s tekočinsko kromatografijo z masno spektrometrijsko detekcijo (LC-MS)

Cosmetics - Analysis of cosmetic products - Determination of 3-iodo-2-propynyl butylcarbamate (IPBC) in cosmetic preparations, LC-MS methods

Kosmetische Mittel - Untersuchung von kosmetischen Mitteln - Bestimmung von 3-Iod-2-propinylbutylcarbamate (IPBC) in kosmetischen Mitteln, LC-MS-Verfahren

Cosmétiques - Analyse de produits cosmétiques - Détermination quantitative du carbamate 3-iodo-2-propynylbutyle (IPBC), méthodes CL-SM

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Cosmetics - Analysis of cosmetic products - Determination of 3-iodo-2-propynyl butylcarbamate (IPBC) in cosmetic preparations, LC-MS methods

Cosmétiques - Analyse des produits cosmétiques -
Détermination quantitative du carbamate 3-iodo-2-
propynylbutyle (IPBC), méthodes CL-SM

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Mitteln - Bestimmung von 3-Iod-2-propynylbutylcarbamat
(IPBC) in kosmetischen Mitteln, LC-MS-Verfahren

This European Standard was approved by CEN on 25 April 2013.

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Foreword

This document (EN 16343:2013) has been prepared by Technical Committee CEN/TC 392 “Cosmetics”, the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by November 2013, and conflicting national standards shall be withdrawn at the latest by November 2013.

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1 Scope

This European Standard specifies a method for the quantitative determination of 3-iodo-2-propynyl butylcarbamate (IPBC) in the concentration range from 0,005 g/100 g to 0,1 g/100 g - Annex V No. 56 in Regulation (EC) No 1223/2009 on cosmetic products.

2 Principle

IPBC is extracted from the cosmetic preparation using methanol. IPBC present in the sample extract is separated using reverse phase HPLC with mass specific detection (LC-MS or LC-MS/MS). Quantitative determination of IPBC is made using the external standard method of calibration or standard addition.

3 Reagents

3.1 General

If not otherwise specified, analytical-grade chemicals shall be used; water shall be distilled or of a suitable purity. "Solution" shall be understood as an aqueous solution unless otherwise specified.

3.2 Iodopropynyl butylcarbamate, CAS number: 55406-53-6 (Supplier : Sigma-Aldrich¹⁾ (521949), Dr. Ehrenstorfer GmbH¹⁾ (C 14335000)).

3.3 Methanol, HPLC grade, CAS number: 67-56-1.

3.4 Formic acid, CAS number: 64-18-6.

3.5 Tetrahydrofuran (THF), CAS number: 109-99-9.

3.6 Propan-2-ol, CAS number: 67-63-0.

3.7 Eluents

3.7.1 Eluent A, 1 ml of formic acid (3.4) is mixed with 1 000 ml of water.

3.7.2 Eluent B, Methanol (3.3).

3.8 IPBC stock solution, $\rho = 1$ mg/ml.

Weigh approximately 0,05 g of IPBC (3.2) into a 50-ml-volumetric flask. Firstly, dissolve in a small amount of methanol (3.3) and then fill up to the calibration mark with methanol. This solution has a shelf life of 8 weeks if it is stored in a refrigerator.

3.9 Calibration solutions (standard solutions)

5,0 ml of the stock solution (3.8) is transferred into a 50-ml-volumetric flask and filled to the calibration mark with methanol (3.3), ($\rho = 0,1$ mg/ml or 100 μ g/ml). From this solution, at least 5 solutions are prepared by dilution to obtain IPBC concentrations of $\rho = 0,05$ μ g/ml to $\rho = 1,0$ μ g/ml. These solutions have a shelf life of 8 weeks if they are stored in a cool place. Examples of dilutions are given in Table 1.

¹⁾ This is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by CEN of this product. Equivalent products may be used if they can be shown to lead to the same results.

Table 1 — Calibration solutions

| No. | Preparation | Dilution | Concentration µg/ml |
|------|--------------------------------|----------|------------------------|
| 3.9 | 5 ml 3.8 filled up to 50 ml | 1:10 | 100 |
| 3.10 | 10 ml 3.9 filled up to 100 ml | 1:100 | 10 |
| 3.11 | 10 ml 3.10 filled up to 100 ml | 1:1000 | 1 |
| 3.12 | 4 ml 3.10 filled up to 50 ml | 1:1250 | 0,8 |
| 3.13 | 5 ml 3.10 filled up to 100 ml | 1:2000 | 0,5 |
| 3.14 | 10 ml 3.11 filled up to 50 ml | 1:5000 | 0,2 |
| 3.15 | 5 ml 3.11 filled up to 50 ml | 1:10000 | 0,1 |

4 Apparatus and equipment

4.1 Standard laboratory equipment

4.2 Membrane filter, in the form of a disposable syringe filter, pore width: 0,2 µm²⁾.

4.3 High-performance liquid chromatograph, suitable for gradient elution with mass-specific detector.

4.4 Analytical separation column, the following parameters have proved useful:

RP 18 Phase, 5 µm, 150 mm × 2 mm, e.g. Zorbax¹⁾, Spherisorb¹⁾, Phenomenex-Luna¹⁾ or equivalent. If a precolumn is used, it shall have the same analytical properties as the separation column.

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5 Procedure <https://standards.iteh.ai/catalog/standards/sist/f630c2bf-424a-424f-9beb-a6c3da5037b0/sist-en-16343-2013>

5.1 Sample preparation

Weigh approximately 200 mg of the sample to the nearest 0,1 mg into a 20-ml-volumetric flask (alternatively a 50-ml-volumetric flask). Add 1,5 ml of THF (3.5) and shake. Add 10 ml of methanol (3.3), and allow to dissolve or suspend for 5 min in the ultrasonic bath at room temperature. Allow to cool to room temperature and fill to the calibration mark with methanol (3.3). The sample solution is diluted with methanol (3.3) to a ratio of 1:10, filtered through a membrane filter (4.2) and then analysed using LC-MS or LC-MS/MS.

For poorly soluble or suspendable matrices it is recommended to partially dissolve the sample by adding 2 ml of 2-propanol (3.6) instead of THF (3.5) or to stir it with a magnetic stirrer for 30 min prior to the treatment in the ultrasonic bath.

5.2 Liquid chromatography (LC) conditions

When using the apparatus (4.3) and column (4.4), the following conditions have shown to be useful (see Table 2):

2) The ring test was performed using 0,2 µm filter.

Table 2 — Gradient programme

| Time min | Fraction eluent A % | Fraction eluent B % |
|-------------|------------------------|------------------------|
| 0 | 85 | 15 |
| 8 | 10 | 90 |
| 12 | 10 | 90 |
| 13 | 85 | 15 |
| 25 | 85 | 15 |

Column: RP 18 Phase, 5 µm, 150 mm × 2 mm

Injection volume: 1 µl to 10 µl

Flow rate: 0,2 ml/min

Column oven temperature: 25 °C

5.3 Detection

5.3.1 General

The detection and quantitative determination can be performed by evaluating the mass traces of IPBC or by evaluating the fragment ions. To avoid false low results by adduct formation occurring in the MRM mode of the Electrospray (ESI) method, the APCI (Atmospheric Pressure Chemical Ionisation) method should be used for ionisation.

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5.3.2 MS detection in Selected Ion Monitoring (SIM) mode

Mass traces: m/z 282 $[M+H]^+$ and m/z 304 $[M+Na]^+$.

Evaluation is based on the total ion current (from the sum of the two masses).

5.3.3 MS detection in Multiple Reaction Monitoring (MRM) mode

Protonated molecule ion: 282 $[M+H]^+$

Fragment ion 1: 57

Fragment ion 2: 165

Evaluation is based on the most sensitive fragment ion.

Since the analyte may form adducts with sodium ions, false low results can be obtained if larger amounts of sodium ions are present in the sample. If ESI ionisation is used, standard addition should therefore be performed using the MRM mode so as to ensure reliable quantification results.

When performing the standard addition, the content of the added IPBC should not exceed the content expected in the sample.

6 Evaluation

6.1 Identification and quantitative determination

The IPBC is identified by comparing the retention times of the sample with those of calibration solutions.

The quantitative determination of the analyte is performed based on a calibration function or the standard addition. The calibration solutions are chromatographed in accordance with the conditions given in 5.2. The IPBC concentration is calculated from the calibration by linear regression on the basis of the obtained peak area.

6.2 Calculation

The preservative content w in g/100 g, with respect to the sample, is calculated using the following formula:

$$w = \frac{c \cdot V \cdot 100 \cdot F}{m \cdot 1000 \cdot 1000} \quad (1)$$

where

w is the IPBC content, in g/100 g;

c is the IPBC concentration in the sample solution, in µg/ml, determined from the calibration function;

m is the initial weight of the sample, in g;

V is the volume of the sample measurement solution, in ml;

F is the dilution factor, if required.

The result is given in g/100 g, rounded to three decimal places.

7 Test report

The test report shall contain the following data:

- information necessary for the identification of the sample (type, origin and designation of the sample);
- a reference to this European Standard;
- name of the laboratory performing the test;
- the date and type of sampling procedure (if known);
- the date of receipt and date of analysis;
- the date of test;
- the test results and the units in which they have been expressed;
- justification of any deviations from this official method;
- operations not specified in the method or regarded as optional, which might have affected the results.