DETERMINATION OF FREE FORMALDEHYDE

in cosmetic products

"Knowledge is of no value unless you put it into practice."

Anton Chekhov

European Network of Official Cosmetics Control Laboratories (OCCLs)

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High performance liquid chromatography method for the determination of free formaldehyde in cosmetic products using 2,4-DNPH for derivatisation

European Directorate for the Quality of Medicines & HealthCare

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High performance liquid chromatography method for the determination of free formaldehyde in cosmetic products using 2,4-DNPH for derivatisation

Users should verify the performance of the method in their laboratory for each different matrix.

1. Scope

This procedure describes a method for the determination of free formaldehyde in cosmetic products for concentration ranging from 75 to 1200 mg/kg.

2. Principle

Free formaldehyde is extracted using THF:water (9:1) as a solvent. The sample extract and the standard solutions are derivatised at pH ≈ 1 using 2,4-dinitrophenylhydrazine (2,4-DNPH). The derivative is stabilised at pH 1.5–2.5 by using phosphate buffer. The yellow derivative is analysed using HPLC with a reversed phase stationary phase (RP8) and UV detection at 354 nm. The identity of formaldehyde is confirmed by comparing UV spectra with reference samples.

3. Limitation of the method

Using this procedure (THF extraction) only the free formaldehyde is determined.

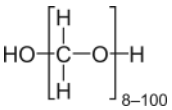
Total formaldehyde, i.e. free formaldehyde and formaldehyde bound to the parent preservative, can be determined using an HPLC method, based on derivatisation with acetylacetone (see *References,* c).

4. Terms, definitions and abbreviations

Formaldehyde is a gas at room temperature with a sharp pungent odour.

Formalin is a saturated aqueous solution of formaldehyde (37 % by mass).

Paraformaldehyde is polymerised formaldehyde, a white powder with a sharp pungent odour. Dissolution in hot water results in a formaldehyde solution.



Free formaldehyde as determined in this method is defined as:

a. Formaldehyde (or methylene glycol) formed by hydrolysis of a preservative,

b. Excess formaldehyde used to synthesise the preservative,

c. Formaldehyde from other formaldehyde-based raw materials used to prepare the cosmetic product,

d. Formalin used as a preservative for the raw materials themselves.

Examples of formaldehyde-releasing preservatives, formaldehyde donors, are: bronopol, quaternium 15, DMDM hydantoin, imidazolidinyl urea, sodium hydroxymethyl glycinate, bronidox, diazolidinyl urea and benzylhemiformal. These preservatives are known to hydrolyse in aqueous solution.

5. Health, safety and environment

It is the user's responsibility to use safe and proper techniques in handling materials in the methods of analysis specified in this document.

- Consult manufacturers for specific details such as material safety data sheets and other recommendations.
- Wear protective goggles, gloves and coats in all laboratory areas.
- Take great care with substances which are toxic and/or human carcinogens.
- Use a fume cupboard during the preparation of organic solvent solutions.

- Dispose of solvents in accordance with environmental requirements.

6. Apparatus, reagents and solutions

6.1. Apparatus

HPLC system equipped with a Diode Array Detector (DAD)

Column	Lichrosorb RP8 (250 x 4 mm, 5 µm) or an equivalent stationary phase
Column temperature	25.0 °C
Injection volume	10 µL
Detection	Wavelength 354 nm (bandwidth 8 nm, no ref)
	Spectral range: 210–400 nm
Flow rate	1 mL/min
Runtime	15 min

Make sure that the mobile phase (gradient) tubes correspond to the solvents.

6.2. Accessories

Use standard laboratory glassware and equipment, with the addition of:

6.2.1. *Adjustable pipettes for organic solvents*

Ranges 20–200 µL, 200–1 000 µL and 1–5 mL.

6.2.2. *Glass tubes*

10 mL with stopper NS14.

6.3. Reagents

Reagents of at least analytical grade are used. Water is to be double distilled or of equal quality.

6.3.1. *Formaldehyde, 37 % (m/m)*

Stabilised with 10–15 % methanol, CAS [50-00-0].

6.3.2. *Triton-X 100 detergent*

CAS [9002-93-1].

6.3.3. *2,4-Dinitrophenylhydrazine (2,4-DNPH)*

Approximately 50 % in water, CAS [119-26-6].

6.3.4. *Tetrahydrofuran (THF)*

Stabilised with BHT, CAS [109-99-9].

6.3.5. *Acetonitrile (ACN)*

CAS [75-05-8].

6.3.6. *Potassium dihydrogen phosphate (KH_2PO_4)*

CAS [7778-77-0].

6.3.7. *Disodium hydrogen phosphate dihydrate ($Na_2HPO_4 \times 2\,H_2O$)*

CAS [10028-24-7].

6.3.8. *Butylhydroxytoluene (BHT)*

CAS [128-37-0].

6.3.9. *Sodium hydroxide (NaOH)*

Caustic soda, CAS [1310-73-2].

6.3.10. *Hydrochloric acid (HCl)*

37 %, CAS [7647-01-0].

6.3.11. *Potassium iodide (KI)*

CAS [7681-11-0].

6.3.12. *Potassium iodate (KIO_3)*

CAS [7758-05-6], dried before use for 2 to 3 h at 180 °C and stored in a desiccator.

6.3.13. *Iodine (I_2)*

CAS [7553-56-2].

6.3.14. *Soluble starch*

CAS [9005-84-9].

6.3.15. *Sodium thiosulfate ($Na_2S_2O_3 \times 5H_2O$) solution*

0.1 N (0.1 mol/L), CAS [10102-17-7].

6.4. Solutions

6.4.1. *Sodium hydroxide (NaOH), 4 N*

Weigh 160 g of NaOH (6.3.9) and dissolve in some water, add water up to 800 mL and cool to room temperature. Transfer to a 1 000 mL volumetric flask and make up to the mark with water.

6.4.2. *Sodium hydroxide (NaOH), 1 N*

Dilute 250 mL of 4 N NaOH (6.4.1) to 1 000 mL with water.

6.4.3. *Hydrochloric acid (HCl), 4 N*

Dilute 330 mL of HCl (6.3.10) to 1 000 mL with water in a volumetric flask.

6.4.4. *Iodine solution, 1 N (0.5 mol/L)*

Dissolve 240 g of KI (6.3.11) in approximately 25 mL of water. Add 127 g of I_2 (6.3.13) and dissolve in a little water. Transfer to a 1 000 mL volumetric flask and make up to the mark with water. Store in an amber coloured flask.

6.4.5. *Iodine solution, 0.1 N*

Dilute 100 mL of 1 N iodine solution (6.4.4) to 1 000 mL with water in a volumetric flask.

6.4.6. *Starch solution, 0.5 %*

Weigh 0.5 g of soluble starch (6.3.14) and dissolve in 80 mL of boiling water. Allow the solution to cool to room temperature. Transfer to a 100 mL volumetric flask and

make up to the mark with water. Store the solution for a maximum period of 3 months.

6.4.7. *Sodium thiosulfate ($Na_2S_2O_3 \times 5\ H_2O$) solution, 0.1 N (0.1 mol/L)*

Determine the strength of this solution as follows:

Weigh to the nearest 0.1 mg about 70 mg of dried potassium iodate (6.3.12) and 2 g of potassium iodide (6.3.11) and dissolve in 50 mL of water. Add 5 mL of 4 N HCl (6.4.3) and titrate the excess of iodine with 0.1 N sodium thiosulfate solution using 3 mL of starch solution (6.4.6) as indicator. Calculate the concentration according to 8.1.

Repeat the procedure to obtain a duplicate.

6.4.8. *2,4-DNPH solution, 0.1% in 2 N HCl*

Weigh approximately 200 mg of 2,4-DNPH (6.3.3) and dissolve in 50 mL of 4 N HCl (6.4.3), transfer to a 100 mL volumetric flask and make up to the mark with water. Protect the solution against light by using amber coloured glassware.

6.4.9. *0.1 M Phosphate buffer pH 6.8*

Weigh 1.1348 g of KH_2PO_4 (6.3.6) and 1.4845 g of $Na_2HPO_4 \times 2\ H_2O$ (6.3.7) in a 250 mL volumetric flask and dissolve in water. Check the pH and correct if necessary using 1 N NaOH (6.4.2). Fill up to the mark with water.

6.4.10. *THF, extra stabilised*

Dissolve approximately 1 g of BHT (6.3.8) in 2.5 L THF (6.3.4).

Note: The use of stabilised THF is necessary to stabilise the derivative. Commercially available stabilised THF contains the stabiliser BHT in concentrations that may vary from batch to batch (about 200 to 500 mg/L). The degree of stability of the derivative depends on the stability of the THF (availability of BHT). For this reason, BHT is added to the commercially available stabilised THF.

6.4.11. *THF solution 9:1 (V/V)*

Mix 900 mL of THF (6.4.10) and 100 mL of water and homogenise.

6.4.12. *Mobile phase, ACN:H_2O (45:55)*

When using an isocratic pump: mix 450 mL of ACN (6.3.5) with 550 mL of water for HPLC.

Homogenise and degas before use.

6.4.13. *Formaldehyde stock solution (10 mg/mL)*

Weigh (WS) formaldehyde (6.3.1) (≈ 2.5 mL), with an accuracy of 0.1 mg, in a 100 mL volumetric flask and make up to the mark with water. Prepare the solution immediately before use. Calculate the concentration according to 8.2.2.

6.4.14. *Diluted formaldehyde stock solution (100 mg/L)*

Pipette accurately 1.0 mL of stock solution (6.4.13) into a 100 mL volumetric flask and make up to the mark with water. Prepare the solution immediately before use.

6.4.15. *Formaldehyde standard solutions (1.5–24 mg/L)*

Pipette accurately the volume of diluted formaldehyde stock solution (6.4.14) into a volumetric flask as indicated in Table 1. Make up to the mark with THF solution 9:1 (*V/V*) (6.4.11).

Table 1 – *Preparation of standard solutions in concentration 1.5–24 mg/L*

Calibration range	St_1	St_2	St_3	St_4	St_5	St_6
Volume [mL]	0.375	0.75	1.50	3.00	4.50	6.00
Volumetric flask [mL]	25	25	25	25	25	25
Concentration [≈ mg/L]	1.5	3	6	12	18	24

6.4.16. *Blank*

A blank sample and a blank solvent [St_0 = THF solution 9:1 (*V/V*) (6.4.11)] must be analysed.

For liquid and viscous materials (7.2.1), prepare a blank sample using 0.1 mL of Triton X-100 in 10 mL of THF solution 9:1 (*V/V*) (6.4.11).

For gels (7.2.2), prepare a blank sample by mixing 10 mL of water with 40 mL of THF solution 9:1 (*V/V*) (6.4.11).

If an extract is further diluted with THF solution 9:1 (*V/V*) (6.4.11), the blank sample must be diluted in the same manner. Different blank samples could be included in a sequence. The blank sample may contain some formaldehyde due to the natural presence of low concentrations of formaldehyde in water. Correct the results for the blank if the concentration of formaldehyde in the blank is more than 10 % of the area of the peak corresponding to the lowest concentration in the calibration range. Inject several blank solvents (St_0) in a sequence, preferably at least every 20th injection.

7. Procedure

Clean all glassware that has been in contact with 2,4-DNPH before final cleaning in a dishwasher. Dispose of the chemical waste in an appropriate manner.

7.1. Formaldehyde titration

Check the concentration of the formaldehyde reagent (6.3.1) in duplicate at least twice a year.

For the titration, the amount of formaldehyde must be in the range of 10–20 mg.

To determine the concentration of formaldehyde an excess of iodine is added. In alkaline conditions, formaldehyde is oxidised to formic acid according to:

$$CH_2O + 2\,OH^- \rightarrow HCOOH + H_2O + 2e^-$$
$$I_2 + 2e^- \rightarrow 2\,I^-$$
$$CH_2O + 2\,OH^- + I_2 \rightarrow HCOOH + H_2O + 2\,I^-$$

The excess of iodine is titrated using sodium thiosulfate according to:

$$I_2 + 2e^- \rightarrow 2\,I^-$$
$$2\,S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2e^-$$
$$I_2 + 2S_2O_3 \rightarrow 2\,I^- + S_4O_6^{2-}$$

7.1.1. *Step 1*

Pipette accurately 1.5 mL (*V*) of freshly prepared formaldehyde stock solution (6.4.13) into a flask with a stopper. Add 25.0 mL, accurately measured, of 0.1 N iodine solution (6.4.5). Add 2 mL of 4 N NaOH (6.4.1) and store the solution for 10 to 15 min in the dark. Shake occasionally.

7.1.2. *Step 2*

Add 5 mL of 4 N HCl (6.4.3) and immediately titrate the excess of iodine with 0.1 N sodium thiosulfate (6.4.7) until the colour changes from brown to light yellow. Add 3 mL of a 0.5 % starch solution (6.4.6) and titrate until the solution is colourless (V_1).

Perform a blank analysis in duplicate using water instead of formaldehyde stock solution (V_0). Calculate the concentration of formaldehyde according to 8.2.1.

7.2. Sample extraction

Homogenise the sample if possible. The initial dilution factor of the sample is 50. If the concentration of formaldehyde in the sample solution is higher than that of the highest standard solution an additional dilution is performed: add 200 µL of extract + 800 µL of THF solution 9:1 (*V*/*V*) (6.4.11) in a glass tube with a stopper. The factor for additional dilution of the sample solution is f = 5.

Perform a blank determination for each extraction procedure used (see 6.4.16).

Warning: This method may generate a high systematic error. For this reason a recovery experiment must be performed to ensure the precision and trueness.

7.2.1. *Liquid and viscous materials, e.g. cream, lotion, shampoo, oil*

Weigh 1 g (W) of sample, to the nearest 0.01 g, in a 50 mL volumetric flask (V). Dissolve in about 40 mL of THF solution 9:1 (*V*/*V*) (6.4.11) with vigorous shaking. If the emulsion does not break up, add about 0.5 mL of Triton-X-100 (6.3.6.3.2). Make up to the mark and homogenise. The solution may be stable for at least 24 h under optimal conditions. In the case of an out-of-specification result, repeat the analysis and analyse the sample solution immediately after preparation.

7.2.2. *Gels, e.g. toothpaste*

Weigh 1 g (W) of sample, to the nearest 0.01 g, in a glass beaker. Add 2.5 mL of water and make a slurry by stirring thoroughly with a spatula. Immediately transfer the slurry to a 50 mL volumetric flask, rinse the glass beaker and the spatula with 3 quantities, each of 2.5 mL, of water, and finally with THF solution 9:1 (*V*/*V*) (6.4.11). Make up to the mark with THF solution 9:1 (*V*/*V*) (6.4.11) and homogenise. Minimise the contact time with water alone to minimise hydrolysis of the formaldehyde donor. The solution may be stable for at least 24 h under optimal conditions. In the case of an out-of-specification result, repeat the analysis and analyse the sample solution immediately after preparation.

7.3. Derivatisation

Pipette (see remark) accurately 1.00 mL of solution (samples, standards and blanks) into test tubes (6.2.2) with stoppers. Add 2,4-DNPH reagent (6.4.8) according to Table 2 and derivatise for between 2 and 2.5 min. Add 1 N NaOH (6.4.2) and 0.1 M phosphate buffer (6.4.9) to stabilise the extract at pH 2–2.5.

Table 2 – *Addition of 2,4-DNPH reagent*

t (mm:ss)	Addition	Vibramix Time	Vibramix Setting
00:00	0.45 mL of 2,4-DNPH (6.4.8)	1 sec.	6 (1 tube)
00:10 ≤ t ≤ 01:00	–	≥ 20 sec.	10 (rack with tubes)
02:00 ≤ t ≤ 02:30	0.75 mL of 1 N NaOH (6.4.2)	1 sec.	6 (1 tube)
02:30 ≤ t ≤ 03:00	0.40 mL of 0.1 M phosphate buffer (6.4.9)	1 sec.	6 (1 tube)
02:35 ≤ t ≤ 03:05	–	10 sec.	10 (rack with tubes)

Up to 6 solutions can be derivatised in parallel within the time schedule. After each addition, the test tube must be closed with a stopper.

Remark: Solutions with organic solvents must be added using a pipette suitable for volatile solvents.

7.4. Analysis

Inject 10 µL of each derivatised solution (7.3) into the HPLC system and analyse using the conditions in 6.1. The solution may be stable for at least 24 h under optimal conditions. In the case of an out-of-specification result, repeat the analysis and analyse the derivatised sample solution immediately after preparation.

7.5. Peak identification

The approximate retention times of the analytes are:

Compound	RT (min)	Comment
2,4-DNPH	5.90	A peak due to excess of 2,4-DNPH
Formaldehyde 2,4-DNPH derivative	10.25	–

Retention times are given for information and they need to be confirmed in every individual HPLC system. An example of a chromatogram obtained with the 9 mg/L formaldehyde solution is shown in Annex 1.

8. Calculation

8.1. Calculation of the normality of the 0.1 N sodium thiosulfate solution (6.4.7)

$$t_{Na_2S_2O_3}\,[N] = \frac{m}{35.667 \times V}$$

Where:

t concentration of sodium thiosulfate solution in normality/litre [N]

m mass of dried potassium iodate [mg]

V volume of sodium thiosulfate used [mL]

35.667 equivalent mass of iodate = 214.001 (g/mol)/6 (eq/mol) [g/eq].

8.2. Calculation of the concentration of formaldehyde in the reagent

8.2.1. *Calculate the concentration of formaldehyde in the reagent (6.3.1) from the following expression:*

$$CM_{HCHO}\,[\%m/m] = \frac{15.013 \times (V_0 - V_1) \times t \times 10}{WS \times V}$$

Where:

CM_{HCHO} concentration of formaldehyde in the reagent [%*m/m*]

V_1 volume of sodium thiosulfate used to titrate the formaldehyde stock solution (7.1.2) [mL]

V_0 volume of sodium thiosulfate used to titrate the blank (7.1.2) [mL]

V volume of stock solution used for titration (7.1.1) [1.5 mL]

WS concentration of formaldehyde in the stock solution (6.4.13) [g/100 mL]

t normality of sodium thiosulfate solution [N]

15.013 equivalent mass of formaldehyde [30.026/2 g/eq] (10 mL of titrated 0.1 N iodine solution = 15.013 mg of formaldehyde).

Check if the amount of formaldehyde in the reagent is within 37.0 ± 0.5 % *m/m*. When the amount is within this range set the concentration of formaldehyde to 37.0 % *m/m*, otherwise use the determined amount.

8.2.2. *Calculate the concentration of formaldehyde in the standard stock solution (6.4.13) from the following expression:*

$$CS_{HCHO}\,[mg/mL] = \frac{WS \times CM_{HCHO} \times 10}{V}$$

Where:

CS_{HCHO} concentration of formaldehyde in the stock solution [mg/mL]

CM_{HCHO} 37 % *m/m*, see 8.2.1 [%]

WS mass of reagent (6.3.1) in the stock solution ≈ 2.7 [g]

V volume of stock solution = 100 [mL].

8.3. Calibration

Prepare a calibration curve (non-forced through the origin) by plotting the peak areas obtained with the standard solutions against the concentration of formaldehyde; use weighted linear regression [1/X = Linear (Amnt)].

$$Y = A + B \times X$$

Where:

A y-intercept

B slope

Y response (area)

X concentration [mg/L] of the standard solutions (before derivatisation).

Calculate the residual bias of each standard solution.

$$Bias_{residual}\,[\%] = \frac{X_{measured} - X_{prepared}}{X_{prepared}} \times 100 = \left(\frac{X_{measured}}{X_{prepared}} - 1\right) \times 100$$

Test the correlation coefficient and the residuals according to the criteria:

$r > 0.995$ or $r^2 > 0.990$

$bias_{residual} < 10$ %

8.4. Drift

Check the drift by repeated injections of a standard in the middle of the calibration range (St_3, Table 1), approximately every 10th injection. The drift must be ≤ 10 %.

$$drift[\%] = \frac{|X_{highest} - X_{lowest}|}{X_{mean}} \times 100\%$$

Where:

X_{mean} mean of the measured concentrations including the value used in the calibration curve

$X_{highest}$ highest measured concentration of the standard in the middle of the calibration range

X_{lowest} lowest measured concentration of the standard in the middle of the calibration range.

Instead of concentration X, response Y is also acceptable to check pure detector drift.

8.5. Confirmation

Compare the spectrum (210–400 nm) of the sample with the spectrum of the standard solution with the nearest concentration. Criteria: match > 99 %.

8.6. Calculate the concentration of formaldehyde in the product from the following expression:

$$concentration[mg/kg] = X \times \frac{V}{W} \times \frac{f}{cf}$$

Where:

X concentrations of formaldehyde in the extracts [mg/L] interpolated from the calibration curve and corrected for the blank when the blank area > 10 % of the area of the peak corresponding to the lowest concentration in the calibration range

W mass of the sample [g]

V volume of the volumetric flask [mL]

f factor for additional dilution

cf correction factor for systematic errors, normally cf = 1.

9. Reporting

The test report should contain the following data:

a. Information necessary for the identification of the sample (type, origin and designation of the sample;

b. The date of receipt and date of analysis;

c. The test results (including the measurement uncertainty) and the units in which they have been expressed;

d. Justification of any deviation from the method;

e. Operations not specified in the method or regarded as optional, which might have affected the results.

10. Validation

Interlaboratory validation was performed using body milk and night cream. Reproducibility and repeatability results obtained are reported in Annex 2.

10.1. Linearity

Using 0.1 % 2,4-DNPH the working range is linear from 0.7 to approximately 40 mg/L.

10.2. Selectivity

The literature [*References,* a and b] shows that RP8 columns of different suppliers can influence the selectivity.

10.3. Weighting of calibration curve

For standard solutions with a wide calibration range, even a correlation coefficient r ≥ 0.995 is no guarantee of a low bias for the determination. To correct for this, the linear regression could be calculated with the weighting 1/X [Linear (Amnt)].

11. References

a. Pai-Wen Wu *et al;* Determination of Formaldehyde in Cosmetics by HPLC Method and Acetylacetone Method, *Journal of Food and Drug Analysis*, Vol. II, No. 1 (2003) p. 8-15

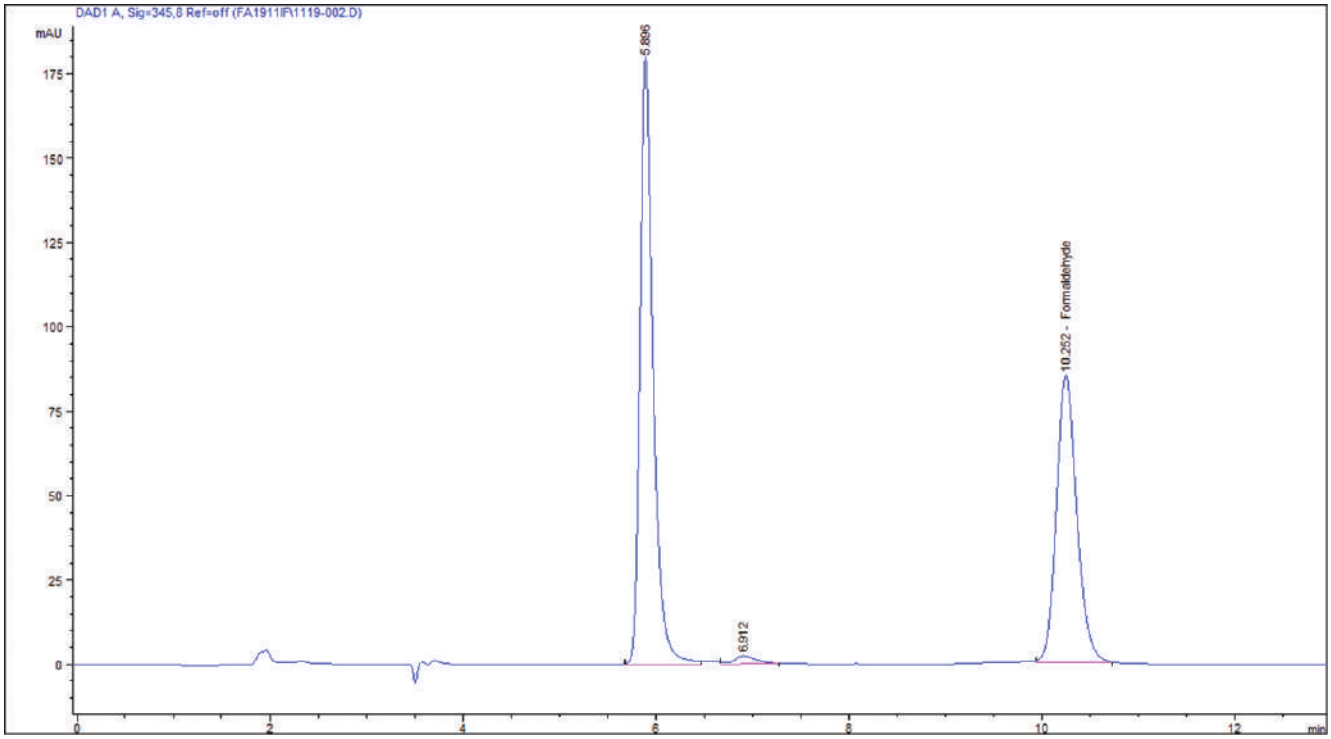
b. Vander Heyden, Y. *et al;* Simultaneous determination of ketoconazole and formaldehyde in shampoo: Liquid chromatography method development and validation, *Journal of Chromatography A*, 958 (2002) p. 191-201

c. Food and Commodities Act, Formaldehyde in textile (water extraction method), http://wetten.overheid.nl/BWBR0012348/ (retrieved on 08-01-2016)

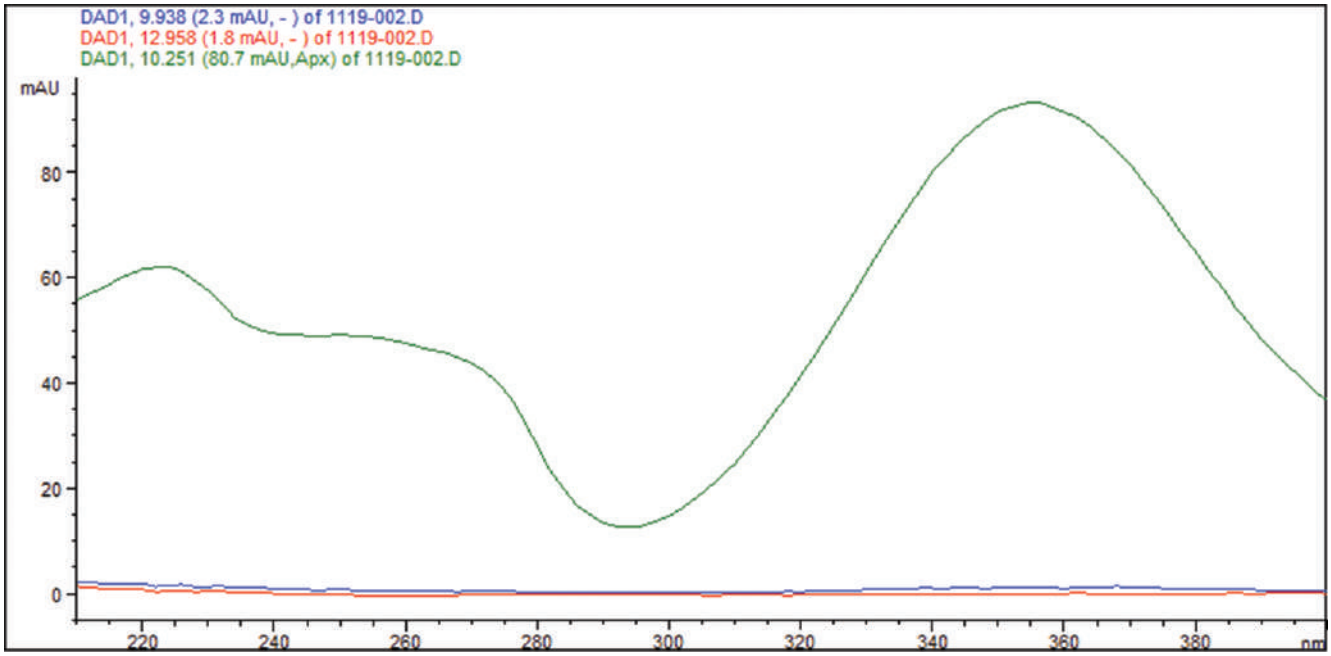
d. Determination of free formaldehyde in lotion, shampoo, toothpaste and finger-paint using 2,4-DNPH for derivatisation and HPLC-PDA, CHE01-WV419, Netherlands Food and Consumer Product Safety Authority (NVWA)

Annex 1. Example chromatogram

9 mg/L Formaldehyde (3rd peak) 2,4-DNPH derivative.
The highest peak (1st peak) is due to excess of 2,4-DNPH.
The small peak (2nd peak) is 2,4-DNPH due to unstable THF (low BHT).



Spectrum



Annex 2. Results of interlaboratory trial[1] for body milk and night cream

Night cream

	Replicates				
Lab	1	2	3	s	s_2
1	0.009	0.009	0.008	5.77350E-04	3.33333E-07
2	0.008	0.008	0.009	5.77350E-04	3.33333E-07
3	0.01	0.009	0.009	5.77350E-04	3.33333E-07
4	0.012	0.012	0.012	2.12459E-18	4.51390E-36
5	0.0093	0.01	0.0104	5.56776E-04	3.10000E-07
6	0.01	0.011	0.009	1.00000E-03	1.00000E-06
7	0.00885	0.0094	0.00915	2.75379E-04	7.58333E-08

Number of labs	7			
Mean (%m/m)	0.0096			
Reproducibility st. dev. (sR) (%m/m)	0.0012			
Reproducibility rel. st. dev. (RSDR) (%)	13.0			
Sum			3.56421E-03	2.38583E-06
Repeatability st. dev. (sr) (%m/m)	0.0006			
Repeatability rel. st. dev. (RSDr) (%)	6.1			
Reproducibility (%m/m)	0.0035			
Repeatability (%m/m)	0.0016			

Body milk

	Replicates				
Lab	1	2	3	s	s_2
1	0.019	0.02	0.019	5.77350E-04	3.33333E-07
2	0.019	0.019	0.02	5.77350E-04	3.33333E-07
3	0.02	0.019	0.02	5.77350E-04	3.33333E-07
4	0.021	0.021	0.021	0.00000E+00	0.00000E+00
5	0.0159	0.0162	0.0169	5.13160E-04	2.63333E-07
6	0.025	0.025	0.022	1.73205E-03	3.00000E-06
7	0.0192	0.0193	0.0181	6.65833E-04	4.43333E-07

Number of labs	7			
Mean (%m/m)	0.0096			
Reproducibility st. dev. (sR) (%m/m)	0.0012			
Reproducibility rel. st. dev. (RSDR) (%)	13.0			
Sum			3.56421E-03	2.38583E-06
Repeatability st. dev. (sr) (%m/m)	0.0006			
Repeatability rel. st. dev. (RSDr) (%)	6.1			
Reproducibility (%m/m)	0.0035			
Repeatability (%m/m)	0.0016			

1 EDQM interlaboratory study, Free formaldehyde released from precursors in different cosmetic products, March 2014.

ENG

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