

Formaldehyde and formaldehyde-releasing agents are commonly used in personal care products, such as cosmetics, as preservatives and disinfectants. Due to high toxicity and suspected carcinogenicity of formaldehyde the products are tested to determine free formaldehyde content and ensure safety of the consumers.

HPLC method with post-column derivatization allows for quantifying free formaldehyde in a wide range of products and materials and is the basis of the analytical method listed in *Cosmetic Safety Technical Specification 2015*. The method is simple, selective and very sensitive. This method is suitable for the determination of free formaldehyde content in water-based cosmetic products, cream emulsions and gel cosmetics. Potential interferences are either separated from formaldehyde on HPLC column or do not react with post-column reagent and are not detected.

Method

Analytical Conditions

Column: Reversed-phase C₁₈ column,
5 µm, 4.6 x 250 mm

Column Temperature: 30 °C

Flow Rate: 1.0 mL/min

Mobile Phase: 2 mL/L of Phosphoric acid in DI water

Separation of Formaldehyde is isocratic but column washing step with 50 / 50 mobile phase /acetonitrile is recommended.

Injection Volume: 10 µL

Post-Column Conditions

Post-Column System: Onyx PCX or Vector PCX

Reactor Volume: 0.5 mL

Reactor Temperature: 100 °C

Reagent: DI Water

62.5 g/L Ammonium Acetate

7.5 mL/L Glacial Acetic Acid

5 mL/L Acetylacetone

Flow Rate: 0.5 mL/min

Detection: UV/Vis 420 nm

Sample Preparation

Accurately weigh out 0.2 g of sample into a centrifuge tube. Add 10 mL of phosphoric acid solution (2 mL/L in DI water). If needed, 10 mL of Methylene Chloride could be added to remove non-polar compounds. Shake the mixture for 1 min and centrifuge at 5000 rpm for 5 min. Filter the water portion through 0.45 µm filter before injecting

Table 1. Recoveries of free formaldehyde

Sample Type	Hand Cream	Hair Gel	Hair Color
Formaldehyde in the blank	0.0002 %	0.0001 %	0.0001 %
RSD, N=3	0.3 %	5.7 %	2.3 %
Spike level 1	0.001 %	0.001 %	0.001 %
Recoveries, N=3	97.4 %	96.9 %	92.5 %
RSD, N=3	0.7 %	2.4 %	1.8 %
Spike level 2	0.005 %	0.005 %	0.005 %
Recoveries, N=3	98.3 %	95.9 %	93.9 %
RSD, N=3	0.6 %	0.9 %	0.7 %

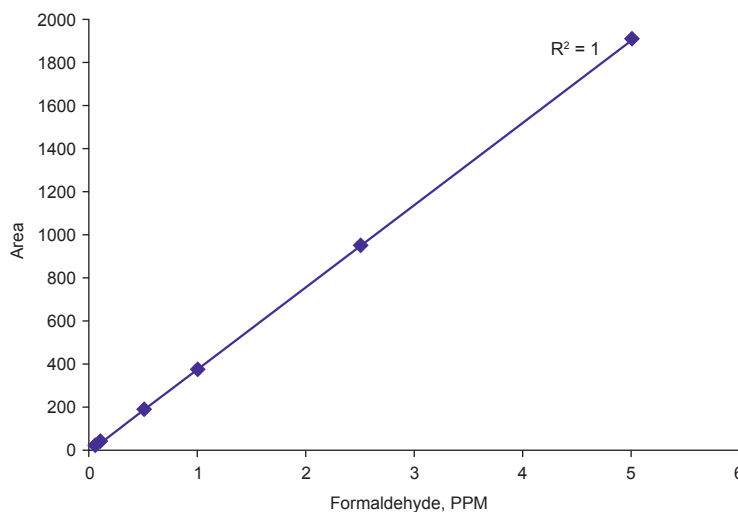


Fig 1. Calibration curve for Formaldehyde (0.05 – 5 ppm)

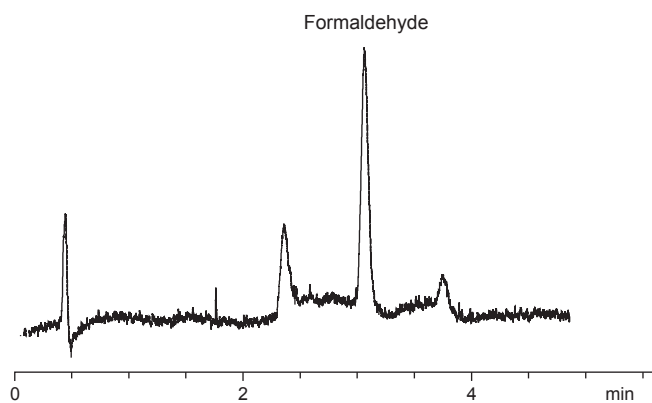


Fig 2. Chromatogram of hand cream blank, containing 0.0002 % of free formaldehyde

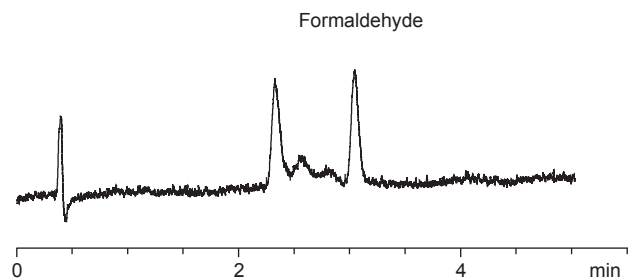


Fig 3. Chromatogram of hair color blank, containing 0.0001 % of free formaldehyde

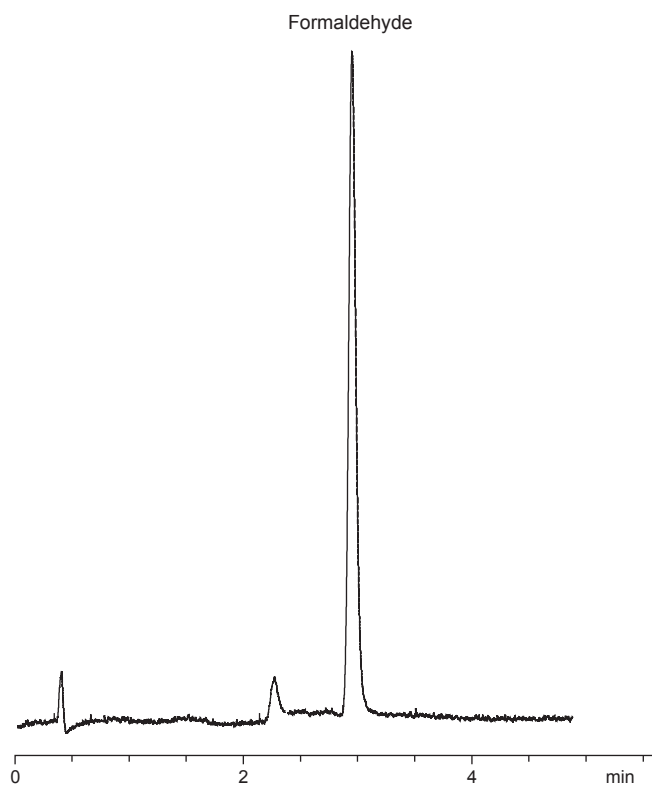


Fig 4. Chromatogram of hair gel sample spiked at 0.001 % of free formaldehyde

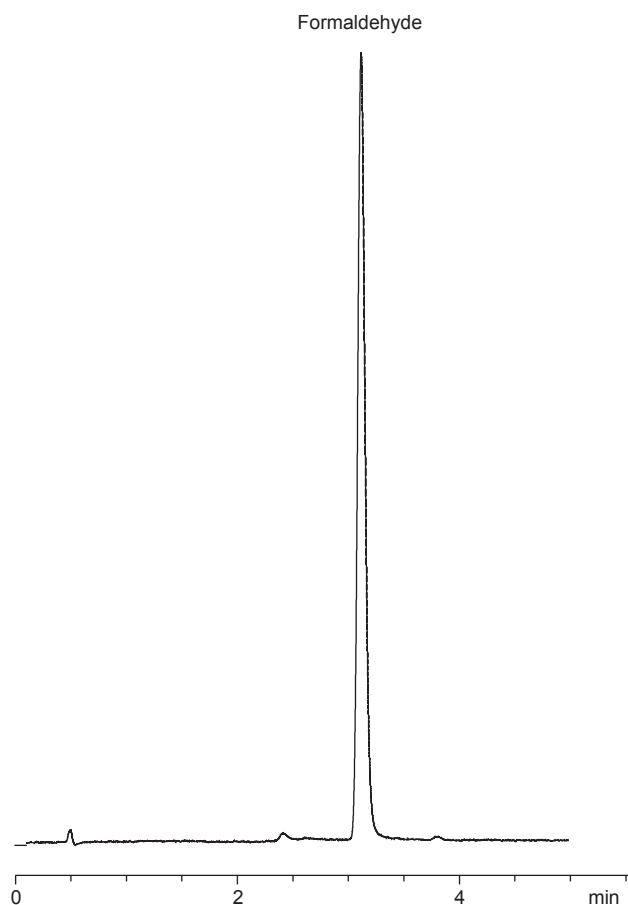


Fig 5. Chromatogram of hand cream sample spiked at 0.005 % of free formaldehyde