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Determination of free formaldehyde in cosmetics containing formaldehyde-releasing preservatives by reversed-phase dispersive liquid-liquid microextraction and liquid chromatography with post-column derivatization

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Abstract

An analytical method for the determination of traces of formaldehyde in cosmetic products containing formaldehyde-releasing preservatives has been developed. The method is based on reversed-phase dispersive liquid-liquid microextraction (RP-DLLME), that allows the extraction of highly polar compounds, followed by liquid chromatography–ultraviolet/visible (LC-UV/Vis) determination with post-column derivatization. The variables involved in the RP-DLLME process were studied to provide the best enrichment factors. Under the selected conditions, a mixture of 500 µL of acetonitrile (disperser solvent) and 50 µL of water (extraction solvent) was rapidly injected into 5 mL of toluene sample solution. The extracts were injected into the LC-UV/Vis system using phosphate buffer 6 mmol L^{-1} at pH 2 as mobile phase. After chromatographic separation, the eluate merged with a flow stream of pentane-2,4-dione in ammonium acetate solution as derivatizing reagent and passed throughout a post-column reactor at 85 ºC in order to derivatize formaldehyde into 3,5-diacetyl-1,4-dihydrolutidine, according to Hantzsch reaction, which was finally measured spectrophotometrically at 407 nm. The method was successfully validated showing good linearity, an enrichment factor of 86 ± 2, limits of detection and quantification of 0.7 and 2.3 ng mL^{-1}, respectively, and good repeatability (RSD < 9.2 %). Finally, the proposed analytical method was applied to the determination of formaldehyde in different commercial cosmetic samples containing formaldehyde-releasing preservatives, such as bronopol, diazolidinyl urea, imidazolidinyl urea, and DMDM hydantoin, with good relative recovery values (91 – 113 %) thus showing that matrix effects were negligible. The good analytical features of the proposed method besides