

Quantitative determination of formaldehyde in cosmetics using combined headspace–solid-phase microextraction–gas chromatography

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Synopsis

The objective of this research was the application of headspace (HS)–solid-phase microextraction (SPME) for the quantitation of formaldehyde present in raw materials and cosmetic formulations. The formaldehyde was derivatized *in situ* first with pentadfluorophenylhydrazine (PPPH), to form a derivative hydrazone. The formed hydrazone was adsorbed on a SPME fiber during headspace extraction under controlled conditions (time, temperature, volume, etc.). After the adsorption step, the SPME fiber was directly transferred into the gas chromatography (GC) injection port in which the analytes were thermally desorbed. Deuterated acetone was used as an internal standard (IS) in order to quantitate the formaldehyde content. For the experiment, a gas chromatograph equipped with a flame ionization detector (GC/FID) was employed. A gas chromatograph/mass spectrometer (GC/MS) was used for the qualitative confirmation of results in this work.

INTRODUCTION

For decades, formaldehyde was one of the most widely used preservatives in personal care products due to its versatility, cost, and efficiency. In recent years, the potential for carcinogenic and respiratory sensitization from formaldehyde has become widely understood. This has led to a movement in the industry that has imposed regulations, restrictions, and formaldehyde's usage being banned.

In surfactant and cosmetic industries several analytical procedures have been developed for qualitative as well as quantitative analysis. One of the most well known procedures for qualitative analysis is the *phloroglucinol test*, in which formaldehyde reacts in an alkaline medium with phloroglucinol to produce a reddish-brown color complex. For the quantitation of formaldehyde, several methods have been employed, one of which is based on *Nash reagent*. In this determination, formaldehyde is condensed with ammonia and acetylacetone to form the lutidine derivative 3,5-diacetyl-1,4-dihydrolutidine (1,2). Nash reagent is sensitive not only to formaldehyde, but also to other aldehydes. If chromophore compounds are present in the product, they could interfere with the spectrophotometric determination. Formaldehyde can also be determined colorimetri-

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