

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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Table of Contents Quantitative Determination Of Formaldehyde In Cosmetics

1. Understanding the eBook Quantitative Determination Of Formaldehyde In Cosmetics
 - The Rise of Digital Reading Quantitative Determination Of Formaldehyde In Cosmetics
 - Advantages of eBooks Over Traditional Books
2. Identifying Quantitative Determination Of Formaldehyde In Cosmetics
 - Exploring Different Genres
 - Considering Fiction vs. Non-Fiction
 - Determining Your Reading Goals
3. Choosing the Right eBook Platform
 - Popular eBook Platforms
 - Features to Look for in an Quantitative Determination Of Formaldehyde In Cosmetics
 - User-Friendly Interface
4. Exploring eBook Recommendations from Quantitative Determination Of Formaldehyde In Cosmetics
 - Personalized Recommendations

- Quantitative Determination Of Formaldehyde In Cosmetics User Reviews and Ratings
- Quantitative Determination Of Formaldehyde In Cosmetics and Bestseller Lists
- 5. Accessing Quantitative Determination Of Formaldehyde In Cosmetics Free and Paid eBooks
 - Quantitative Determination Of Formaldehyde In Cosmetics Public Domain eBooks
 - Quantitative Determination Of Formaldehyde In Cosmetics eBook Subscription Services
 - Quantitative Determination Of Formaldehyde In Cosmetics Budget-Friendly Options
- 6. Navigating Quantitative Determination Of Formaldehyde In Cosmetics eBook Formats
 - ePub, PDF, MOBI, and More
 - Quantitative Determination Of Formaldehyde In Cosmetics Compatibility with Devices
 - Quantitative Determination Of Formaldehyde In Cosmetics Enhanced eBook Features
- 7. Enhancing Your Reading Experience
 - Adjustable Fonts and Text Sizes of Quantitative Determination Of Formaldehyde In Cosmetics
 - Highlighting and Note-Taking Quantitative Determination Of Formaldehyde In Cosmetics
 - Interactive Elements Quantitative Determination Of Formaldehyde In Cosmetics
- 8. Staying Engaged with Quantitative Determination Of Formaldehyde In Cosmetics
 - Joining Online Reading Communities
 - Participating in Virtual Book Clubs
 - Following Authors and Publishers Quantitative Determination Of Formaldehyde In Cosmetics
- 9. Balancing eBooks and Physical Books Quantitative Determination Of Formaldehyde In Cosmetics
 - Benefits of a Digital Library
 - Creating a Diverse Reading Collection Quantitative Determination Of Formaldehyde In Cosmetics
- 10. Overcoming Reading Challenges
 - Dealing with Digital Eye Strain
 - Minimizing Distractions
 - Managing Screen Time
- 11. Cultivating a Reading Routine Quantitative Determination Of Formaldehyde In Cosmetics
 - Setting Reading Goals Quantitative Determination Of Formaldehyde In Cosmetics
 - Carving Out Dedicated Reading Time
- 12. Sourcing Reliable Information of Quantitative Determination Of Formaldehyde In Cosmetics
 - Fact-Checking eBook Content of Quantitative Determination Of Formaldehyde In Cosmetics

- Distinguishing Credible Sources
- 13. Promoting Lifelong Learning
 - Utilizing eBooks for Skill Development
 - Exploring Educational eBooks
- 14. Embracing eBook Trends
 - Integration of Multimedia Elements
 - Interactive and Gamified eBooks

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