Analysis of isothiazolinone biocides in cosmetic products and detergents by HPLC

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INTRODUCTION

Despite their sensitizing effect isothiazolinones biocides are used in several cosmetics and cleaning products as antimicrobial agents. The most important isothiazolinones biocides are methylisothiazolinone (MIT), chloromethylisothiazolinone (CMI), benzoisothiazolinone (BIT), octylisothiazolinone (OIT), dichloroisothiazolinone (DCOIT), and butyldichloroisothiazolinone (BBIT). The use of these heterocyclic compounds is restricted by law [1, 2, 3]. If manufacturers use these preservatives, these substances must be declared on the packaging.

Due to very different properties of these substances sample preparation and chromatographic separation are often not suitable for all target compounds. In the first part of this work we present a methodology for sample preparation of isothiazolinones analysis which includes a solid phase extraction (SPE) method for cosmetics and cleaning products. The second part of this work points out the chromatographic conditions on core-shell columns with perfluoroalkylpropyl (PF) modification with UV and MS detection and illustrates limits of detection (LOD) and quantification (LOQ).

RESULTS

SPE recoveries determined for standard solutions

The recovery of all analytes is about 80 % or more. However, significant losses can occur at the exchange of the solvent if no keeper is used. In particular, the losses are greatest for the volatile analytes, MIT and CMI.

CHROMATOGRAPHIC METHOD

Column: NUCLEOSHELL® HR-X pentafluorophenylpropyl modification, multi-endcapping, particle size 3.7 μm, 100 x 2 mm ID

Flow rate: 0.27 mL/min

Curtain gas: 10.00 psig

Pressure: 80 %

Ion source: Turbo Spray (ESI)

Scan type: MRM

UV detection:

1.0 μg BBIT/g, 0.7 μg OIT/g and 1.8 μg DCOIT/g are possible for detergents and cleaning agents. There 0.2 μg OIT/g and 0.5 μg DCOIT/g and the quantification of 2.0 μg MIT/g, 7.2 μg CMI/g, 0.4 μg BIT/g, 0.7 μg CMIT/g, 0.3 μg BIT/g, 0.1 μg BBIT/g, 0.2 μg OIT/g and 0.1 μg DCOIT/g and a quantification of 2.1

Mass spectrometry and UV detection results

With the developed methodology the detection of 0.6 μg MIT/g, 1.9 μg CMIT/g, 0.3 μg BBIT/g, 0.2 μg OIT/g and 0.5 μg DCOIT/g and the quantification of 2.0 μg MIT/g, 7.2 μg CMI/g, 0.4 μg BIT/g, 0.7 μg CMIT/g, 0.3 μg BIT/g, 0.1 μg BBIT/g, 0.2 μg OIT/g and 0.1 μg DCOIT/g.

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REFERENCES


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