

Simultaneous determination of degradation products of nonylphenol polyethoxylates and their halogenated derivatives by solid-phase extraction and gas chromatography-tandem mass spectrometry after trimethylsilylation

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Abstract: An efficient method for the simultaneous determination of the degradation products of nonylphenol polyethoxylates (NP_nEOs, n = number of ethoxy units), i.e., nonylphenol (NP), NP_nEOs (n=1-3), nonylphenoxy carboxylic acids (NP_nECs, n=1-2, number of ethoxy units plus an acetate) and their halogenated derivatives (XNP, XNP1EO and XNP1EC; X=Br or Cl), in water samples were developed. After trimethylsilylation with N,O-bis(trimethylsilyl)acetamide, all the analytes were determined by gas chromatography-tandem mass spectrometry (GC-MS-MS) with electron ionization (EI). The ion peaks of [M-85]⁺ of the derivatives were selected as precursor ions and their product ions showing the highest intensities were used for the quantitative analysis. The instrumental detection limits were in the range from 2.1 to 11 pg. The recoveries of the analytes from the water samples were optimized by using solid-phase extraction (SPE). The deuterated reagents of octylphenol, octylphenol monoethoxylate and octylphenoxyacetic acid were used as the surrogates. The method detection limits (500 ml water sample) using C₁₈ SPE were from 2.5 to 18 ng/l. The recoveries from spiked pure water and the environmental water samples were greater than 78%. The method was successfully applied to environmental samples. Remarkably, the concentrations of the halogenated compounds (ClNP, ClNP1EO and BrNP1EO) were detected at the hundreds of ng/l levels in the Neya river. ?? 2003 Elsevier B.V. All rights reserved.

Author Keywords: Derivatization, GC; Environmental analysis; Halogenated compounds; Nonylphenol; Nonylphenol polyethoxylates; Nonylphenoxy carboxylic acids; Solid-phase extraction; Water analysis

Index Keywords: Degradation; Electrons; Extraction; Halogenation; Ionization; Organic acids; Phenols; Solid phase extraction (SPE); Gas chromatography; acetamide derivative; carboxylic acid derivative; halide; nonylphenol; phenol derivative; reagent; analytic method; article; gas chromatography; precursor; priority journal; quantitative analysis; silylation; solid phase extraction; tandem mass spectrometry; water analysis; water sampling; Gas Chromatography-Mass Spectrometry; Phenols; Sensitivity and Specificity; Trimethylsilyl Compounds

Year: 2003

Source title: Journal of Chromatography A

Volume: 1020

Issue: 2

Page : 161-171

Cited by: 17

Link: Scopus Link

Chemicals/CAS: nonylphenol, 25154-52-3; nonylphenol, 25154-52-3; Phenols; Trimethylsilyl Compounds
Manufacturers: 3M, United States

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ISSN: 219673

CODEN: JCRAE

DOI: 10.1016/j.chroma.2003.08.064

PubMed ID: 14661741

Language of Original Document: English

Abbreviated Source Title: Journal of Chromatography A

Document Type: Article

Source: Scopus

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