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Method Article

Determination of short-chain chlorinated paraffins in multiple matrices of Arctic using gas chromatography-electron capture negative ion-low resolution mass spectrometry



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A B S T R A C T

Gas chromatography-electron capture negative ion-low resolution mass spectrometry (GC-ECNI-MS) was used for the quantification of short-chain chlorinated paraffins (SCCPs) in multiple matrices of Arctic. Samples were spiked with surrogate standards (¹³C₁₀-*trans*-chlordan) and extracted with dichloromethane and hexane (1:1, v/v) using accelerated solvent extraction (ASE). The extract was cleaned using a multilayer Silica-Florisil column and ε-HCH was added before instrument analysis. The SCCPs were analyzed using a gas chromatograph (GC) in electron capture negative ion (ECNI) mode coupled with a 7000B triple quadrupole mass spectrometer (MS) in single quad mode. The calibration is performed using three commercial standards (chlorine contents of 51.5%, 55.5%, and 63.0%). A reasonable linear correlation for commercial standards was found between chlorine content and total response factor ($R^2 = 0.96$). To ensure instrument sensitivity, the SCCP congeners were divided into four groups by the optimized combinations (C₁₀, C₁₁, C₁₂ and C₁₃) and subjected to analysis by four individual injections. This method is suited to analyse the total concentration of SCCPs and individual SCCP congener groups in the environmental analysis.

- Accelerated solvent extraction offers a lower cost of time for per sample and reducing solvent consumption.
- The presented quantification procedure makes the quantification of SCCPs independent from the chlorine content of the used standard mixtures.

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• The samples were subjected to analysis by four individual injections for the instrument sensitivity, and the total concentration of SCCPs and CP congener groups can be obtained at the same time.

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ARTICLE INFO

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Keywords: Accelerated solvent extraction (ASE), Multilayer silica-florisil column, GC-ECNI-MS

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Specification Table

Subject Area	Environmental Science
More specific subject area:	Environmental analytical chemistry
Method name:	Gas Chromatography-Electron Capture Negative Ion-low resolution Mass Spectrometry (GC-ECNI-LRMS)
Name and reference of original method	New quantification procedure for the analysis of chlorinated paraffins using electron capture negative ionization mass spectrometry
Resource availability	Accelerated Solvent Extraction (Dionex ASE 350)7890?A GC - 7000B MS (Agilent, USA)

Method details

Chemicals

The commercial standards of the SCCPs (chlorine contents of 51.5%, 55.5%, and 63.0%, with the congener group patterns shown in Fig. 1) at concentrations of 100 ng/μL in cyclohexane and ε-hexachlorocyclohexane (ε-HCH, solution in cyclohexane, 10 ng/μL) were purchased from Ehrenstorfer GmbH (Augsburg, Germany). The ¹³C₁₀-*trans*-chlordane (100 ng/μL solution in *n*-nonane, purity 99%) was purchased from Cambridge Isotope Laboratories (Andover, USA). Pesticide-grade dichloromethane, *n*-hexane, acetone, cyclohexane, and toluene were purchased from JT Baker (Phillipsburg, NJ). Florisil (60–100 mesh) and silica gel (180–280 mesh) were purchased from Merck (Whitehouse Station, NJ). Anhydrous sodium sulfate was purchased from Sinopharm Chemical Reagent Beijing Co., Ltd.

Silica gel was activated at 550 °C for 12 h, Florisil was activated at 140 °C for 7 h, and anhydrous sodium sulfate was activated at 660 °C for 6 h before use. A mixture of 30% (40%) acid silica gel was prepared by mixing 100 g of activated silica gel and 44 g (66 g) of concentrated sulfuric acid.

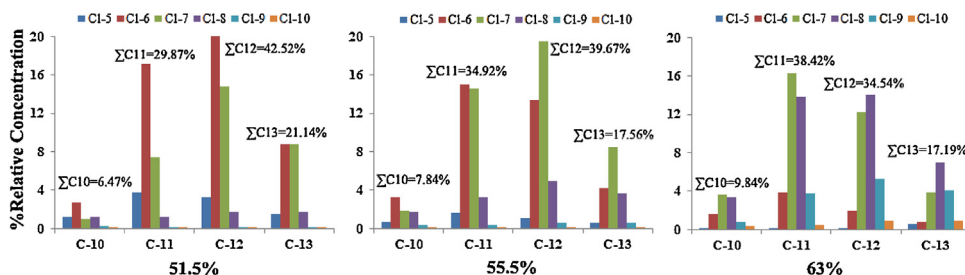


Fig. 1. Congener group pattern for commercial references of SCCPs (C₁₀₋₁₃, 51.5%, 55.5% and 63.0% chlorine content).

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