



Analysis of hexachlorocyclohexanes in aquatic samples by one-step microwave-assisted headspace controlled-temperature liquid-phase microextraction and gas chromatography with electron capture detection

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ABSTRACT

A microwave-assisted headspace controlled-temperature liquid-phase microextraction (HS-CT-LPME) technique was applied for the one-step sample extraction of hexachlorocyclohexanes (HCHs) from aqueous samples with complicate matrices, followed by gas chromatographic (GC) analysis with electron capture detector (ECD). Microwave heating was applied to accelerate the evaporation of HCHs into the headspace and an external-cooling system was used to control the temperature in the sampling zone for HS-LPME. Parameters affecting extraction efficiency, such as LPME solvent, sampling position and temperature, microwave power and irradiation time (the same as sampling time), sample pH, and salt addition were thoroughly investigated. From experimental results, the following conditions were selected for the extraction of HCHs from 10-mL water sample (pH 2.0) by using 1-octanol as the LPME solvent, with sampling done at 38 °C for 6 min under 167 W of microwave irradiation. The detections were linear in the concentration of 0.1–10 µg/L for α-HCH and γ-HCH, and 1–100 µg/L for β-HCH and δ-HCH. Detection limits were 0.05, 0.4, 0.03 and 0.1 µg/L for α-, β-, γ- and δ-HCH, respectively. Environmental water samples were analyzed with recovery between 86.4% and 102.4% for farm-field water, and between 92.2% and 98.6% for river water. The proposed method proved to serve as a simple, rapid, sensitive, inexpensive, and eco-friendly procedure for the determination of HCHs in aqueous samples.

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1. Introduction

Lindane (γ-Hexachlorocyclohexane) is an organochlorine pesticide and a persistent organic pollutant (POP) that has been widely used for the control of agricultural and medical pests [1–2]. Contamination of water occurs by the use of HCH in these aspects. Because of environment-persistence, bioaccumulation and human-toxicity, lindane is listed as a pollutant of concern in EPA's Great Waters Program [3]. HCH is a mixture of alpha, beta, gamma and delta isomers. Since these isomers differ qualitatively and quantitatively in biological activity, they are required to be monitored in aquatic samples in order to assess their health risk.

Simplification, rapidity, miniaturization and eco-friendly procedures have been of interest in the development of sample pre-concentration techniques. In the past decade, solid phase microextraction (SPME) technique has been developed [4] and is widely applied as an alternative to conventional extraction methods [5–6], which are laborious, time- and solvent-consuming

procedures. Meanwhile, headspace solid-phase microextraction (HS-SPME) sampling method had been introduced to eliminate interference problems commonly found in the direct immersion (DI) approach; and has been successfully applied to the determination of organochlorine pesticides through sample heating to accelerate the evaporation of analytes into headspace for sampling [7–11].

During the development of SPME techniques, miniaturization of extraction solvent was also studied in sample preparation. He and Lee [12] developed a liquid–liquid microextraction technique by using a microsyringe, called liquid-phase microextraction (LPME). Later, a porous polypropylene hollow fiber had been introduced in order to protect the solvent drop in the immersed LPME procedure [13–14]. The hollow fiber based LPME has been successfully applied to the determination of organochlorine pesticides in aquatic samples [15–17]. Similar to SPME, HS sampling is applied to substitute immersed sampling of LPME to avoid interference from complicate matrices [18–20].

Microwave heating is applied to accelerate analyte vaporization in order to shorten the sampling time of HS-SPME [11,21]. However, when microwave heating hyphenates to HS-LPME, it results in significant evaporation of the extraction solvent, subsequently

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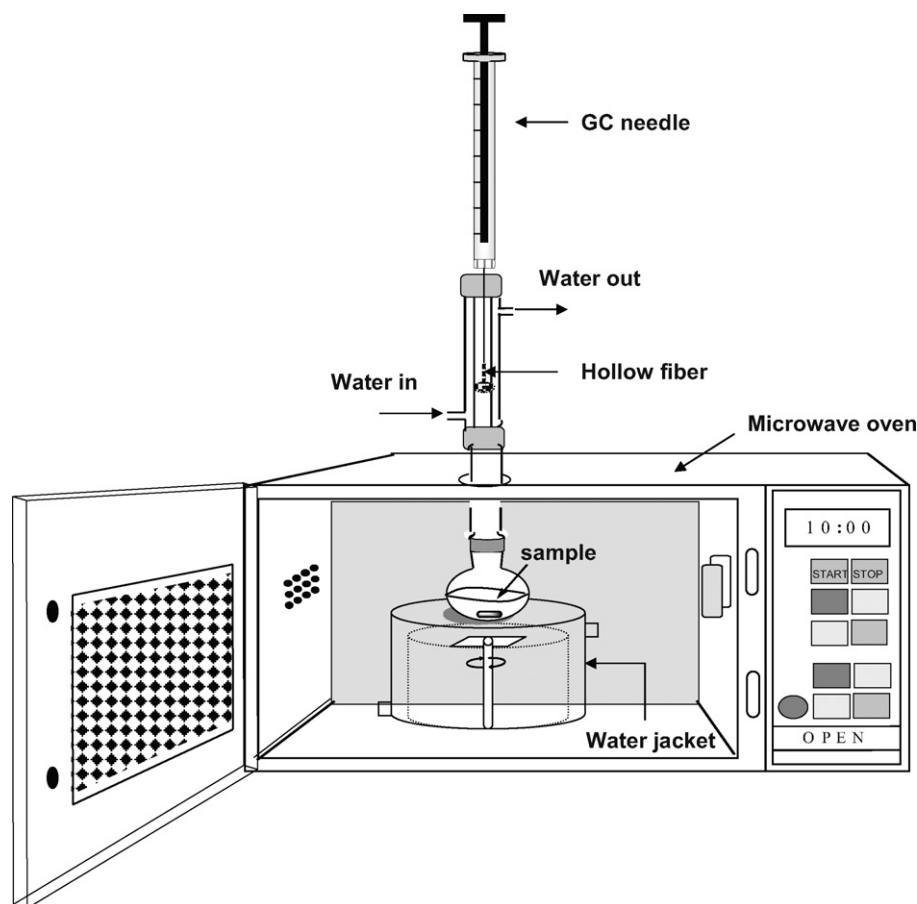


Fig. 1. Assembly of MA-HS-CT-LPME sampling system.

affecting the extraction reproducibility. In our previous study, we designed an external-cooling system to keep the temperature of the sampling point constant, which prevents the vaporization of the LPME extraction solvent [22–23]. With this external-cooling system, a dense cloud (mist) of analyte-water vapor is formed in the headspace due to the sudden cooling of vapors. In the present approach, we report here a HS-LPME sampling technique, which combines the fast microwave-assisted (MA) heating and controlled-temperature sampling (CT) for the determination of HCHs in complicated aquatic samples for effective GC determination.

2. Experimental

2.1. Reagents and solutions

Deionized water for all aqueous solutions was produced in the laboratory using the Barnstead Nanopure water system (Barnstead, NY, USA). All chemicals used in the study were of ACS reagent grade. Four isomers of HCH, α -HCH, β -HCH, γ -HCH and δ -HCH (analytical-standards grade) purchased from Dr Ehrenstorfer (Augsburg, Germany), were used for preparing standard stock solutions that were used without further purification. Standard stock solutions of 1000 mg/L HCHs were prepared individually in acetone (LC grade, Merck, Darmstadt, Germany), and diluted to 100 mg/L and 10 mg/L by using acetone. Fresh working solutions of standards were prepared by appropriate dilution with acetone. All standards and working solutions were stored at 4 °C in silanized brown glass bottles with Teflon-lined caps. Methanol, acetone, hexane, 1-octanol and n-decane of HPLC grade were obtained

from Merck (Darmstadt, Germany). Ethylene Glycol and sodium hydroxide were obtained from Riedel-de Haën (Seelze, Germany). Citric acid and potassium chloride were purchased from Showa (Tokyo, Japan), hydrochloric acid (36.5%) was obtained from J.T. Baker (Phillipsburg, NJ, USA), and toluene was from Tedia (Fairfield, OH, USA). Dichlorodimethylsilane (99%) was obtained from Supelco (Bellefonte, PA, USA). High purity nitrogen (99.9995%) that was used as the carrier gas was obtained from a local supplier (Lien-Hwa, Taichung, Taiwan). River and farm-field water samples were collected from the agriculture district of Dali city (Taichung County, Taiwan).

2.2. Microwave-assisted HS-CT-LPME

The microwave oven used in this work was a NE-V32A inverter system (2450 MHz, National, Taiwan) with a maximum power of 1400 W. The arrangement of the MA-HS-CT-LPME sampling system is shown in Fig. 1. In the sampling system, cooling water system from a water circulating machine (Yih Der BL-720, Taiwan) was connected to the sampling chamber to control the sampling zone temperature and a circulating water flow in the microwave oven to reduce the effective power of microwave irradiation. After modification, the effective powers of microwave irradiation of 47, 70, 116, 167 and 279 W were used in this study. To prevent leakage of irradiation, aluminum foils were attached to the inner and outer wall of the microwave oven at the interface between the microwave body and the headspace sampling apparatus. A microwave leak detector (MD-2000, Less EMF, NY, USA) was used to check the safety aspects of the equipment during the experiments.

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