

Validation method of organochlorine pesticides residues in water using gas chromatography–quadruple mass

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Abstract

In analytical chemistry laboratories, it is critical to have a sensitive method to provide applicable qualitative and quantitative data. This research aimed to determine validated optimized method and acceptable Profession Test (PT) results of 18 organochlorine pesticides (OCPs) using gas chromatography operating with quadruple mass spectrophotometer (GC–QMS) and liquid–liquid extraction (LLE) technique. This will be used for acquiring accreditation and checking alternative instruments such as gas chromatography operational with flame ionization detector (GC–FID). Ismailia canal were selected to investigate organochlorine pesticides content in freshwater used for irrigation and drinking purposes.

The obtained data for limit of quantification (LOQ) varied from 0.159 to 0.254 with Relative Standard Deviation (RSD%) below 20% (4.18–6.12). method detection limit (MDL) ranged from 0.04 to 0.08 $\mu\text{g}/\text{ml}$ and squared correlation coefficient (R^2) ranged from 0.9992 to 0.9999. The study concluded that application to real samples has been successfully assessed by optimized method. © 2016 National Water Research Center. Production and hosting by Elsevier B.V. This is an open access article under the CC BY-NC-ND license (<http://creativecommons.org/licenses/by-nc-nd/4.0/>).

Keywords: Quadruple mass; Organochlorine pesticides; Validation method; Liquid–liquid extraction

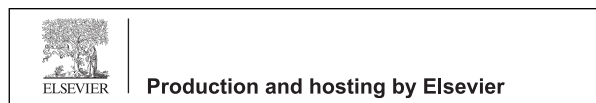
1. Introduction

Organochlorine pesticides (OCPs) are class of the Persistence Organic Pesticides (POPs) based on their persistence, toxicity, bioaccumulation nature, high lipid solubility and long range transport potential (Luzardo et al., 2009). These factors tend to bio-concentration and bio-magnification in food chain (SEI, 2010; Neves Dias et al., 2015). These chemicals reach to aquatic environment generally through surface runoff, discharge from surface pesticides' applications, careless disposal of empty containers, and equipment washings that is unsafe for human health protection and ecological managing (Wang et al., 2008; Lu et al., 2011).

Ecological monitoring of organochlorine pesticide residues is one of the most essential aspects in assessing negative environmental impacts, potential hazards to human health and guide of pesticides legal use confirming public health

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provisions (Hrouzková et al., 2013; Omwenga et al., 2016). The maximum possible limits are 0.1 µg/l for each pesticide and 0.5 µg/l for total concentration of all pesticides (UNEP, 2011) based on toxicological considerations (WHO, 2004; MHR, 2008; EC, 2015a,b).

Recent studies stated that the concentration of OCPs that are still being used in Egypt and other countries coupled with a lack of law enforcement (Xue et al., 2006; Okoknkwó et al., 2007; Nasr et al., 2009; El Bouraie et al., 2011; Abd El-Gawad and Abou El Ella, 2014; Abo El-Maali and Wahman, 2015; Miclean et al., 2015; Abdel-Rahman et al., 2016). Organochlorines were the most frequently detected contaminants in surface water (Nesser et al., 2016) so OCPs analysis is highly common in routine ecological testing laboratories for varied matrix environment (Sultana et al., 2014).

1.1. Accreditation of laboratories for pesticides

Environmental laboratories accredited to International Organization for Standardization (ISO)/International Electro-technical Commission (IEC)—(ISO/IEC 17025) in accordance to article 12 of regulation 882/2004 and article 28 of regulation 396/2005 that must be have acceptable scientific rules for pesticide residues analysis at low level for OCPs accreditation (EC, 2013). Analytical quality control (AQC) requirements support the validity of data to check compliance with method detection limit (MDL) and assess pesticides pollution (EC, 2015a,b). To ensure quality results, analytical methods should be standardized to fulfill validation criteria. Method of validation is a set of practices that are performed to ensure the potential of the analytical method meet global standards. In this respect, different parameters e.g. accuracy, precision, linearity, limit of detection (LOD), limit of quantification (LOQ), specificity are to be investigated.

Numerous applications based on gas chromatography coupled with quadruple mass spectrometry (GC–QMS) determine trace OCPs concentrations with high confirmation of the results, limited optimization of sample preparation procedures, good specificity for organochlorine and increase selectivity for OCPs analysis in water over that of traditional detectors such as flame ionization detector (FID) (Postigo et al., 2010; Abd El-Gawad & Abou El Ella, 2014).

The research aimed to validate optimized analytical method for the determination of 18 organochlorine pesticides using gas chromatography operating with quadruple mass detector (GC–QMS) and liquid/liquid extraction technique (LLE). This provide the laboratory with acceptable proficiency testing (PT) results of 18 organochlorine pesticides for acquiring accreditation, check alternative instruments such as gas chromatography operating with flame ionization detector (GC–FID) and quantify organochlorine pesticides residues in water samples.

2. Material and methods

2.1. Chemicals and reagents

Organic solvents were dichloromethane, *n*-hexane and acetone (HPLC grade, Fisher Chemicals UK) which are toxic and handled with care examining safety protection using well-organized fume hoods and wearing protective gloves. The reagents used during the experimental procedure, such as sodium chloride—Sigma, sodium thiosulfate—Merck, anhydrous sodium sulfate—USA, filter paper, silica gel (60–100 mesh) and glass wool were purchased from Fisher chemicals. Ultra-pure organic-free water used was from Milli-Q system; model Nanopure UV-Barnstead, USA.

Multi-standard M-508P-A of 18 organochlorine pesticides acquired from AccuStandards[®] all company with purity higher than 96.0% that they were α -BHC, β -BHC, γ -BHC-lindane, δ -BHC, Heptachlor, Heptachlor Epoxide, DDTs (*p,p'*-DDE, *p,p'*-DDD, *p,p'*-DDT), Aldrin, Dieldrin, Endrin, Endrin Aldehyde, Endrin ketone, Endosulfan I, Endosulfan II, Endosulfan sulfate, and Methoxychlor and stored at 4 °C.

2.2. Liquid/liquid extraction procedure

Liquid–liquid extraction (LLE) or solvent extraction (SE) is a basic technique in analytical laboratories for OCPs tests that refer to principal chemical and physical processes with different comparative solubility in water and high-purity organic solvent. This technique was used due to its ease, strength and broad approval by international standard methods (Zuloagaa et al., 2012).

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