



Tetradecyl(trihexyl)phosphonium chloride ionic liquid single-drop microextraction for electrothermal atomic absorption spectrometric determination of lead in water samples

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ABSTRACT

A highly efficient single-drop microextraction (SDME) procedure using a low-cost room temperature ionic liquid (RTIL), i.e., tetradecyl(trihexyl)phosphonium chloride (CYPHOS® IL 101), for Pb determination at trace levels in real water samples was developed. Lead was chelated with 2-(5-bromo-2-pyridylazo)-5-diethylaminophenol (5-Br-PADAP) reagent and extracted into a 4 µL microdrop of CYPHOS® IL 101. The RTIL drop was directly injected into the graphite furnace of the electrothermal atomic absorption spectrometer (ETAAS). Under optimal microextraction conditions, a preconcentration factor of 32 was achieved with only 1.5 mL of sample resulting in a phase-volume ratio of 375. The limit of detection (LOD) obtained was 3.2 ng L⁻¹ and the relative standard deviation (RSD) for 10 replicates at 0.5 µg L⁻¹ Pb²⁺ concentration level was 4.9%, calculated at peak heights. The calibration graph was linear from concentration levels near the detection limits up to at least 4.5 µg L⁻¹ with a correlation coefficient of 0.9996. The accuracy of the methodology was evaluated by analysis of a certified reference material (CRM). The method was successfully applied to the determination of Pb in tap, mineral, well and river water samples.

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1. Introduction

Lead is one of the metals considered as a major threat to human health. This heavy metal is characterized by its toxic effects on the gastrointestinal tract, renal system and central or peripheral nervous system [1]. The main sources of lead in humans are inhaled air, diet and drinking water [2]. Lead could be present in water due to general environmental contamination. Polluting sources are quite varied, ranging from industrial and traffic emissions to the use of purification mud and agricultural excipients [3]. The World Health Organization (WHO) has established a maximum permissible limit of 10 µg L⁻¹ Pb in drinking water [4]. Therefore, evaluation of Pb at levels close to this limit and in

presence of relatively high amounts of other ions and organic matter makes straight determination of Pb a difficult task for quality control of waters.

In general, the most widely used techniques for trace determination of Pb are electrothermal atomic absorption spectrometry (ETAAS) [5,6], inductively coupled plasma optical emission spectrometry (ICP-OES) [7,8] and inductively coupled plasma-mass spectrometry (ICP-MS) [9,10]. Among these, ETAAS and ICP-MS are the most sensitive techniques. Despite the great potential of ICP-MS for trace Pb determination, this technique often involves a greater cost, higher sample volume requirements and increased instrumentation complexity, limiting its widespread application to routine analytical work. ETAAS is still being used as it shows several advantages including, fast analysis time, relative simplicity, reduced cost, low sample volume requirements and low detection limits. All of these features have been responsible for its broad application to trace and ultratrace elemental analysis of different samples. However, the low levels of lead in some types of water samples are not compatible with ETAAS detection limits, hence a preconcentration step is needed prior to ETAAS determination [11].

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Table 1
Instrumental and experimental conditions for Pb determination.

Instrumental conditions				
Wavelength (nm)	283.3			
Spectral band width (nm)	0.7			
Lamp current (mA)	360			
Modifier volume (μL)	20			
Modifier amount (μg)	50 μg $(\text{NH}_4\text{H}_2\text{PO}_4)$ 3 μg $\text{Mg}(\text{NO}_3)_2$			
Graphite furnace temperature program				
Step	Temperature ($^{\circ}\text{C}$)	Ramp time (s)	Hold time (s)	Argon flow rate (mL min^{-1})
Drying	110	1	30	300
	150	10	40	300
Pyrolysis	600	10	10	300
	800	5	10	300
Atomization	1600	1	5	0
Cleaning	2400	1	2	300
Extraction conditions				
Working pH	9.0			
Sample volume	1.5 mL			
Pb^{2+} concentration	$0.5 \mu\text{g L}^{-1}$			
5-Br-PADAP concentration	$2.7 \times 10^{-7} \text{ mol L}^{-1}$			
Ethanol concentration	2% (v/v)			
Buffer concentration	$4 \times 10^{-2} \text{ mol L}^{-1}$			
RTIL drop volume	4 μL			
Extraction time	15 min			
MeOH volume (washing solution)	20 μL			

Several sample preparation methods such as solid phase extraction [12], liquid–liquid extraction [13], co-precipitation [14] and knotted reactor separation [15] have been employed for traces lead preconcentration. Conventional liquid–liquid extraction (L–L) is a very effective analytical technique to extract and preconcentrate metallic and organometallic species as well as organic compounds from liquid samples [16]. Recently, considerable interest has been appeared in solvent microextraction procedures based on room temperature ionic liquids (RTILs) [17–19], in which the volume of solvent required is significantly reduced while achieving high extraction efficiency. Single-drop microextraction (SDME) is a simple procedure where analytes are extracted into a single-drop of solvent suspended from the tip of a microsyringe needle [20] that can be directly immersed in the aqueous sample (DI-SDME) or in its headspace (HS-SDME). Due to their high viscosity, thermal stability and undetectable volatility in comparison with conventional solvents, ILs are very suitable for single SDME and HS-SDME. Moreover, larger and more reproducible extracting volumes can be used as well as higher stability of the drop and better precision levels are obtained. ILs have been proposed in SDME and HSME for organic compounds determination [21,22]. Very recently, Manzoori et al. have proposed the use of SDME-ETAAS for determining trace amounts of Mn and Pb in water and food samples [23,24].

Ionic liquids (ILs) have been recognized as alternative solvents for a diversity of chemical applications, such as analytical and separation chemistry [25–28]. Among the various IL families, most of the current research has been focused on 1-alkyl-3-methylimidazolium, tetraalkylammonium and alkylpyridinium based ionic liquids [29,30]. Tetraalkylphosphonium-based ILs are also known for their unique solvent properties, and consequently, they have been considered as alternative solvents for separation chemistry in some publications [31–34].

In this work, a phosphonium-containing RTIL tetradecyl(trihexyl)phosphonium chloride (CYPHOS[®] IL 101) (Fig. 1) is proposed for extraction and preconcentration of Pb with SDME technique. A suitable chelating reagent, such as 2-(5-bromo-

2-pyridylazo)-5-diethylaminophenol (5-Br-PADAP), was used to increase extraction efficiency of the metal ion [35]. To our knowledge, the combination of CYPHOS[®] IL 101 and 5-Br-PADAP chelating agent for developing a SDME and preconcentration procedure to determine Pb has not been reported before. Furthermore, the determination of metals in phosphonium-based ionic media by ETAAS has not been so far studied. The method was applied for evaluating trace levels of Pb in tap, mineral, well and river water samples.

2. Experimental

2.1. Instrumentation

Elemental detection was performed using a Perkin-Elmer 5100 atomic absorption spectrometer (Perkin-Elmer, Norwalk, CT, USA) equipped with a pyrolytic graphite tube (Perkin-Elmer) and a transversely heated graphite atomizer Zeeman-effect background

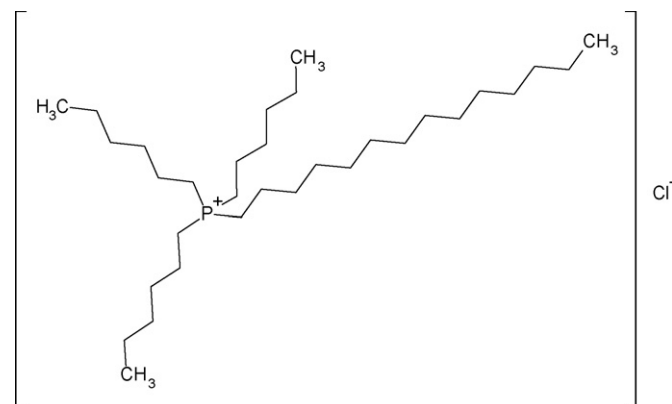


Fig. 1. Molecular structure of the RTIL CYPHOS[®] IL 101 used in this work.

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