

Review

Separation and preconcentration procedures for the determination of lead using spectrometric techniques: A review

Maria das Graças A. Korn^a, Jailson B. de Andrade^a, Djane S. de Jesus^{a,b}, Valfredo A. Lemos^c, Marcus L.S.F. Bandeira^{a,d}, Walter N.L. dos Santos^a, Marcos A. Bezerra^{a,c}, Fabio A.C. Amorim^a, Anderson S. Souza^{a,c}, Sergio L.C. Ferreira^{a,*}

^a Universidade Federal da Bahia, Instituto de Química, Campus Universitário de Ondina, 40170-290 Salvador, BA, Brazil

^b Centro Federal de Educação Tecnológica da Bahia, Rua Emídio Santos, s/n-Barbalho, Salvador, BA, Brazil

^c Universidade Estadual do Sudoeste da Bahia, Departamento de Química e Exatas, Campus de Jequié, 45200-190 Jequié, BA, Brazil

^d Faculdade de Tecnologia e Ciências, Av. Luís Viana Filho, 8812 Paralela Salvador, BA, Brazil

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Abstract

Lead is recognized worldwide as a poisonous metal. Thus, the determination of this element is often required in environmental, biological, food and geological samples. However, these analyses are difficult because such samples contain relatively low concentrations of lead, which fall below the detection limit of conventional analytical techniques such as flame atomic absorption spectrometry and inductively coupled plasma optical emission spectrometry. Several preconcentration procedures to determine lead have therefore been devised, involving separation techniques such as liquid–liquid extraction, solid phase extraction, coprecipitation and cloud point extraction. Citing 160 references, this paper offers a critical review of preconcentration procedures for determining lead using spectroanalytical techniques.

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* Corresponding author. Fax: +55 71 3235 5166.

E-mail addresses: slcf@ufba.br, sergio1057@hotmail.com (S.L.C. Ferreira).

1. Introduction

The word lead derives from the Anglo-Saxon *leaden*, and the symbol Pb, from the Latin word for lead, *Plumbum*. Lead is by far the most abundant (13 ppm) heavy element in the earth's crust. It is one of the few metals that can be found in metallic form in nature (rather than in compounds that must be reduced to extract metal). Lead, a lustrous bluish-white metal, is very soft, highly malleable, ductile and a relatively poor conductor of electricity. It is highly corrosion resistant but tarnishes under exposure to air [1–4].

Lead is an ancient metal, having been known since before recorded history. It is one of the oldest metals known to man and is mentioned in the Old Testament. In Ancient Egypt (7000–5000 B.C.), lead was used in pottery glazing and the Hanging Gardens of Babylon were floored with lead slabs to guard against moisture. The Roman Empire used lead extensively for water pipes and plumbing, some of which is still in use today. The Romans also used lead to manufacture cooking utensils. Studies suggest that the Roman civilization fell largely as a result of widespread lead poisoning. This theory is supported by data showing high lead concentrations in the bones of ancient Romans. In the Middle Ages, the practice of “sweetening” wine with lead or lead acetate became a serious toxicological problem. In those days, the alchemists believed lead was the oldest metal and made innumerable attempts to transform it into gold [3–5].

Our environment contains countless sources of lead, which is a natural component of soil, water, vegetation, animal life and air [6–10]. In the modern world, it is ubiquitous in food, water and air. Lead is now known to be a poison [5,11] absorbed into the bloodstream, from where, especially in the case of inorganic lead, it is distributed to soft tissue, bones and teeth (95% in bones and teeth). Organic forms of lead are fat soluble and therefore have a particular tendency to concentrate in brain [12].

The mechanism of absorption is not yet completely clear, but lead is believed to bind to oxo-groups in enzymes, affecting virtually every step in heme synthesis and porphyrin metabolism. It also inhibits acetylcholinesterase, acid phosphatase, ATPase, etc. Typical symptoms of lead poisoning are abdominal pain, anaemia, headaches and convulsions, chronic nephritis of the kidney, brain damage and central nervous-system disorders. Safe lead stripping from the body remains a major challenge. Due to the poisoning properties of lead, the industrialized countries have banned the use of lead in two practically universal forms—tetraethyl lead and lead-based paints for home use. Until recently, lead poisoning was diagnosed based on its symptoms; however, today it is diagnosed by analyzing its presence in blood using atomic absorption methods [1,5,6].

2. Lead determination

2.1. Atomic absorption spectrometry (AAS)

The determination of lead by flame atomic absorption spectrometry (FAAS) is practically free of interference and requires an air–acetylene flame [13]. The interference caused by alu-

minum and iron can be overcome by the addition of ascorbic acid, citric acid and EDTA [13]. The threshold of sensitivity of this technique is very low (LOD 0.01 mg L⁻¹) and is often unsuitable for trace analysis [13]. In this sense, many preconcentration procedures must be performed to determine trace amounts of lead, as indicated in the tables shown here. The most important analytical lines of lead are 217.00 and 283.31 nm. The 217.00 nm line is more sensitive, notwithstanding the greater amount of background absorption effects [13].

Electrothermal atomic absorption spectrometry (ETAAS) is a good alternative for determining trace amounts of lead in several types of samples in view of its good sensitivity [13]. However, in some cases, previous preconcentration and separation steps are carried out before analytical measurements by ETAAS.

The use of a modifier stabilizes lead, allowing for its determination without causing matrix effects. The Pd–Mg modifier is the one most commonly used, since it produces the best results. This modifier allows for the application of pyrolysis temperatures ranging from 1200 to 1400 °C, which enables the separation of most interfering elements [13–17]. The stabilizing effect of this modifier also raises the atomization temperature to 2000 °C, which allows a characteristic mass of about 16 pg [13]. Ammonium phosphate is another modifier frequently used for determining lead by ETAAS, allowing for an atomization temperature of 1600 °C and enabling a low characteristic mass of 12 pg [13,18].

2.2. Inductively coupled plasma optical emission spectrometry (ICP OES)

Inductively coupled plasma optical emission spectrometry is an analytical technique often employed to determine lead in various types of samples [19]. The main emission lines are: Pb II 220.353 nm, Pb I 216.999 nm and Pb I 283.306 nm, with 220.353 nm being the most sensitive. However, the low level of lead in many samples lies below the detection limit of this technique. Moreover, several types of spectral interference have been reported in the determination of lead by ICP OES. Virtually all photomultiplier-based ICP spectrometers use the Pb 220.353 nm analytical line, despite its severe background continuum and inter-element interference from Al 220.4 nm and background shift due to iron (Fe). Direct spectral overlap interference due to iron has also been found in the 216.9 and 283.9 nm analytical lines [20,21]. Thus, preconcentration and separation procedures have been devised to allow trace amounts of lead to be determined in complex matrices using ICP OES. Several tables shown in this paper summarize the use of separation techniques such as liquid–liquid extraction, solid phase extraction, cloud point extraction and others, as pre-steps in determining lead using ICP OES.

3. Lead separation and preconcentration

Separation techniques such as coprecipitation [23,50], liquid–liquid extraction [57–60], solid phase extraction [69–134] and more recently, cloud point extraction [139–146,150] and on-line coprecipitation using a knotted reac-

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