

ORIGINAL PAPER

Ultra-trace determination of Pb(II) and Cd(II) in drinking water and alcoholic beverages using homogeneous liquid–liquid extraction followed by flame atomic absorption spectrometry

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The determination of Pb(II) and Cd(II) in different sample matrices, including drinking water, distilled spirits and fruit wine, was carried out by flame atomic absorption spectrometry (FAAS) after pre-concentration using homogeneous liquid–liquid extraction (HLL). First, the HLL method was optimised with lead diethyldithiocarbamate (Pb-DDTC) complex which was extracted with a perfluorooctanoate anion (PFOA[−]) dissolved in lithium hydroxide under acidic conditions. The optimum extraction conditions, using 0.01 M DDTC, 0.05 M PFOA[−], 3 M HCl and 1 mL of 30 vol. % acetone, were obtained. The Pb-DDTC complex in the nitric acid digest of the samples (50–150 mL) was extracted quantitatively into a drop of 100 μ L of sediment phase. The sediment phase dissolved in 1 vol. % HNO₃ with at least 3–5 mL of the final volume was then determined by FAAS, affording a pre-concentration factor of 10–50. Hence, the HLL method afforded an increase in both sensitivity and selectivity for the metal determination by conventional FAAS, resulting in ultra-trace level detection of Pb(II) in all samples analysed (drinking water, 9.2–23 ng mL^{−1}; distilled spirits, 23–50 ng mL^{−1}; fruit wine, 24–53 ng mL^{−1}). In addition, the proposed method could successfully be applied to Cd(II) determination in these samples.

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Introduction

Lead and cadmium are known to be toxic heavy metals that accumulate in the human body throughout the lifetime. Its cumulative poisoning effects are serious haematological damage, brain damage, anaemia and kidney malfunctions. The concentration of these metals permitted in a water sample is as low as ng g^{−1} or ng mL^{−1} (Liang & Sang, 2008). Therefore, highly sensitive methods for the determination either of trace or ultra-trace levels in water and other alcoholic samples need to be established. Flame atomic absorption spectrometry (FAAS) is commonly used for the determination of trace metals in various matrices such as drinking water, liquors or wines, even though these samples require pre-treatment (Daorat-

tanachai et al., 2005; Saracoglu et al., 2006). However, the direct determination by FAAS of trace amounts of Pb or Cd in samples is difficult due to its sensitivity limit. The trace amounts of some metals are somewhat incompatible with the detection limit of such instruments, and major constituents such as organic compounds and inorganic salts may cause the main matrix effects.

Consequently, separation and pre-concentration are frequently required prior to FAAS application. The techniques most extensively used for these procedures for trace metals include liquid–liquid extraction (LLE) (Wang & Hansen, 2002), solid-phase extraction (SPE) (Soylak et al., 2005) and cloud point extraction (CPE) (Chen et al., 2005), but disadvantages such as time demands, unsatisfactory enrichment factor, large

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