

## Three modified activated carbons by different ligands for the solid phase extraction of copper and lead

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### Abstract

In the presented work, 5,5-diphenylimidazolidine-2,4-dione (phenytoin) (DFTD), 5,5-diphenylimidazolidine-2-thione-,4-one (thiophenytin) (DFID) and 2-(4'-methoxy-benzylideneimine) thiophenole (MBIP) modified activated carbons have been used for the solid phase extraction of copper and lead ions prior to their flame atomic absorption spectrometric determinations. The influences of the various analytical parameters including pH, amounts of reagent, sample volume and eluent type, etc. on the recovery efficiencies of copper and lead ions were investigated. The influences of alkaline, earth alkaline and some transition metals on the adsorption of the analytes were also examined. The detection limits by three sigma for analyte ions were 0.65 and 0.42  $\mu\text{g L}^{-1}$  using activated carbon modified with DFID; 0.52 and 0.37  $\mu\text{g L}^{-1}$  using activated carbon modified with DFTD and 0.46 and 0.31  $\mu\text{g L}^{-1}$  using activated carbon modified with MBIP for Pb(II) and Cu(II), respectively. The procedure was applied to the determination of analytes in natural waters, soil, and blood samples with satisfactory results (recoveries greater than 95%, R.S.D.'s lower than 4%).

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**Keywords:** Copper; Lead; Preconcentration; Solid phase extraction; Modified activated carbon

### 1. Introduction

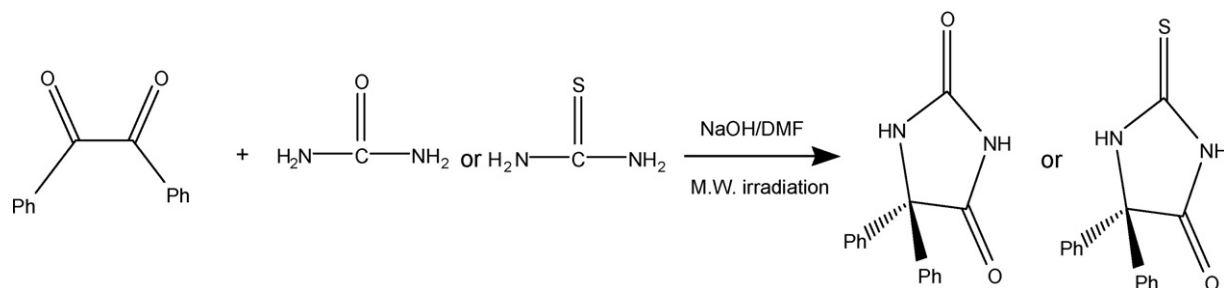
The atomic absorption spectrometry (AAS) offers fast elemental analysis and suffers from poor sensitivity in the determination of heavy metals in environmental samples. This draw back can be overcome by the combination of a suitable preconcentration-separation technique with subsequent AAS determination. Solid phase extraction (SPE) has been widely used for preconcentration of heavy metals due to advantages such as, high efficiency, simplicity, rapidity, low consumption of material especially toxic organic solvents [1–5], freedom from contamination and the possibility of combination with AAS.

The modified solid sorbents are then used and applied in the fields of normal or selective solid phase extraction of

the target species [6,7]. The selectivity of the modified solid phases toward certain metal ions is attributed to several well known factors such as the size of the organic modifier [8], the activity of the loaded surface groups [9,10], the type of the interacting donor atom and metal ion and the reported well known phenomenon of hard–soft acid–bases [11]. The reasons for widespread application of the activated carbon as a trace collector for metal ion preconcentration [12–26] are strong interaction and quantitative adsorption of molecules with  $\pi$  electrons on activated carbon. Activated carbon is a hydrophobic adsorbent which adsorbs non-polar or slightly polar substances in aqueous solutions, metal ions to be preconcentrated need to be transformed to corresponding metal chelates. Metal chelates could provide higher selectivity and high enrichment factor for such a separation and preconcentration techniques [26–34].

In the presented work, 5,5-diphenylimidazolidine-2,4-dione (phenytoin) (DFTD), 5,5-diphenylimidazolidine-2-thione-,

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Scheme 1. Preparation of phenytoin and thiophenytoin.

4-one (thiophenytoin) (DFID) and 2-(4'-methoxybenzylidene-imine) thiophenole (MBIP) modified activated carbons have been used as solid phase materials for the preconcentration and separation of copper and lead ions in environmental samples. The analytical parameters for quantitative recoveries of analytes were investigated.

## 2. Experimental

### 2.1. Reagents and solutions

Acid and bases were of the highest purity obtained from Merck, Darmstadt and were used as received. Doubly distilled deionized water was used throughout. Stock solutions of copper(II) (1000 mg L<sup>-1</sup>) and lead(II) was prepared by Cu(NO<sub>3</sub>)<sub>2</sub> and Pb(NO<sub>3</sub>)<sub>2</sub> (E. Merck, Darmstadt, Germany) in deionised doubly distilled water, respectively. Analytical grade nitrate salts of lead, cadmium, mercury, cobalt, nickel, copper, zinc, magnesium, calcium, strontium, barium, silver, sodium and potassium were of the highest purity purchased from Merck, Darmstadt and used without any further purification.

### 2.2. Instruments

The lead and copper determinations were carried out on a Shimadzu 680 A atomic absorption spectrometer with a hollow cathode lamp and a deuterium background corrector, at wavelengths of 217.0 and 324.8 nm for lead and copper, respectively, using an air-acetylene flame. The pH was determined with a Metrohm 691 pH/ion meter with a combined glass-calomel electrode.

### 2.3. Synthesis of 5,5-diphenylimidazolidine-2,4-dione (phenytoin) and 5,5-diphenylimidazolidine-2-thione-4-one (thiophenytoin) under microwave irradiation

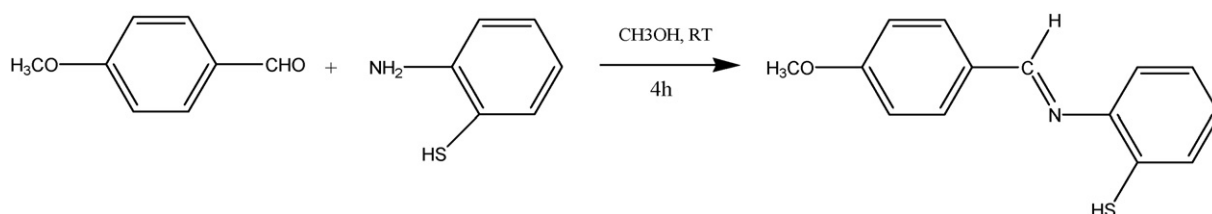
To a mixture of 5 mmol urea (0.3 g) or thiourea (0.38 g) and 5 mmol (1.261 g) 1,2-diphenylethanedione (bezil) in a beaker of 50 mL, 0.3 g sodium hydroxide and four drops dimethylformamide were added. The reaction mixture unsealed was irradiated under 20% microwave with alternatively of 1 min for 3–5 min. The progress of the reaction was monitored by TLC (*n*-hexane:ethyl acetate). After completion of the reaction, the mixture was poured into cold water and filtered. Then the filtrate was acidified by concentrated HCl to precipitate phenytoin or thiophenytoin as products rapidly (Scheme 1). The precipitate was filtered and washed twice with cold water. The pure products were obtained by recrystallization of them from the water/ethanol mixture solvent in 60–67% yield.

#### 2.3.1. Spectral data of DFID

IR (KBr, cm<sup>-1</sup>): 3201 (s), 3283 (s), 3080 (w), 1768 (s), 1736 (s), 1712 (vs), 1682 (sh), 1494 (m), 1442 (m), 1396 (m), 1224 (m), 1190 (m), 1070 (w), 1012 (m), 780 (s), 764 (s), 740 (s), 698 (s), 658 (m), 638 (s), 597 (w), 520 (w), 434 (w), 420 (s). <sup>1</sup>H NMR (DMSO, ppm): 11.12 (s, 1H), 9.32 (s, 1H), 7.3–7.42 (m, 10H). mp (290–296 °C).

#### 2.3.2. Spectral data of DFTD

IR (KBr, cm<sup>-1</sup>): 250 (s), 3160 (s), 3020 (w), 2890 (w), 2760 (w), 1740 (vs), 1720 (vs), 1580 (s), 1524 (vs), 1488 (m), 1440 (s), 1378 (vs), 1310 (m), 1278 (m), 1222 (m), 1194 (w), 1152 (vs), 1100 (w), 1024 (m), 996 (vs), 940 (s), 902 (s), 844 (w), 826 (w), 766 (s), 720 (vs), 692 (vs), 658 (vs), 645 (s), 622 (m), 584



Scheme 2. Preparation of 2-(4'-methoxybenzylidene-imine) thiophenole.

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