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4-O1. Development of methods for trace element determination in accordance with Green chemistry principles

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This lecture presents our efforts to develop environmentally benign analytical methods for trace element analysis by FAAS, ICP-OES, and ICP-MS, reducing sample consumption, utilizing non-hazardous reagents, applying better energy efficiency for sample preparation and system miniaturization.

A microwave-assisted solid phase extraction (MW-SPE) using a microcolumn packed with thioureido propyl functionalized silica gel was developed for Pd and Pt retention followed by elution with 0.5% thiourea under microwave irradiation. In this work, microwave energy was applied for the first time for acceleration of the elution process of trace levels of Pd and Pt in environmental and biological samples.

For the first time the idea of microwave-assisted cloud point extraction (MW-CPE) was studied. MW-CPE based on the use of the non-toxic surfactant Triton X-100 was found to be a green and efficient alternative to classical liquid-liquid extraction. Examples of group preconcentration of Rh, Pd and Pt using 2-mercaptobenzothiazole, N,N'-diphenylthiourea or ammonium O,O'-diethyl dithiophosphate prior to ICP-OES and ICP-MS analysis are presented. Micro sampling (350 µl) by air segmented discrete introduction (ASDI) was performed for determination of Zn in blood serum from children suffering from zinc deficiency.

Another method for separation of Zn, Cu, Cd, and Pb after MW-CPE with ammonium pyrrolidine dithiocarbamate (APDC) was developed. By the combination MW-CPE and ASDI-FAAS the detection limits were lowered by a factor of 10 (Zn, Cu, Pb) or 9 (Cd) compared to the direct FAAS analysis.

A new area of investigation with a high 'green' potential is the use of nanoparticles in the sample preparation process. Magnetic nanoparticles-solid phase extraction (MNPs-SPE) of trace metals (V, Co, Ni, Cu, Zn, As, Se, Pb, and Cd) as complexes with APDC was developed. Due to the very high surface-to-mass ratio, quantitative extraction was achieved using only 10 mg of MNPs and 5 min for complex formation and sorption. MNPs with retained metal complexes were easily separated from the bulk solution by a permanent magnet. The combined MNPs-SPE procedure was applied to analyze urine samples by ICP-MS. The method is a 'greener' alternative to classical solid phase extraction of metal chelates due to the application of a smaller amount of sorbent phase.

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4-O2. Kinetic-spectrophotometric determination of *p*-toluidine based upon its oxidation by periodate catalysed by MnII

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The Mn(II)-catalysed periodate oxidation of *p*-toluidine in acetone-water medium is first order with respect to catalyst, substrate and oxidant each. The reaction progress was followed by monitoring the increase in the absorbance of reaction intermediate. The main reaction product, characterized on the basis of melting point and spectroscopic studies, is 4-methyl-1,2-benzoquinone. The effects of pH, dielectric constant of medium, ionic strength, and free radical scavengers were studied with a view to establish the conditions for determination of *p*-toluidine in the range 74.84 to 429.04 µg/ml. The characteristics of various calibration curves, Sandell's sensitivity, molar absorptivity, percentage recovery, effect of interference, and correlation coefficient were evaluated. An attempt was made for proposing a suitable mechanism for the reaction studied. Thermodynamic parameters are also reported.