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**SOLID PHASE MICROEXTRACTION AND STIR BAR SORPTIVE  
EXTRACTION FOR ORGANOTIN  
COMPOUNDS – A COMPARISON (P 9)**

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Organotin compounds have been largely used in agricultural and industrial applications. Hyphenated techniques were developed for the sensitive and selective determination of such species. For this task GC has been coupled with atomic emission detection.

Derivatization to transform the Sn-compounds into sufficiently volatile compounds was necessary and carried out using sodium tetrapropylborate.

For sample preparation the application of solid phase microextraction (SPME) give recent advances in comparison to classical liquid-liquid extraction (LEE). A problem in the usage of SPME exists however in the small volume of the PDMS coating for enrichment the analytes. For improvement of both sample enrichment and extraction of the organotin compounds Stir Bar Sorptive Extraction (SBSE) [1] was applied. It base on the application of stir bars coated with PDMS. Here the extraction yield is substantially higher.

Aim of this study was to compare the capabilities of GC-AED in combination with SPME and SBSE. After optimisation of the experimental parameters it was possible to reach detection limits in the pg / l – level.

A comparison of both methods shows the expected results. By application of SBSE it was possible to increase the detection limits one order of magnitude.

With SPME the reproducibility of the analytical results (in the 1 ng / l concentration range) was found to be between 10 and 15%, it could be enhanced to 5-8 % by application of SBSE.

These low limits of detection and the good reproducibility allowed the determination of organotin compounds according required regulations.

[1] E. Baltussen, P. Sandra, F. David, C. Cramers, J. Microcolumn Sep. 1999, 11 (10), 737

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**DETERMINATION OF POLYCYCLIC AROMATIC HYDROCARBONS  
FROM PUMPKIN SEEDS AND PUMPKIN SEED OILS BY  
MICROWAVE-ASSISTED SAPONIFICATION (P 10)**

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Polycyclic aromatic hydrocarbons (PAHs) are widespread environmental contaminants of both anthropogenic and natural origin. Human health hazards associated with these compounds are owing to their persistence, their hydrophobic character, their bioaccumulation and the carcinogenic properties of several individual polyarenes. Human exposure to PAH occurs principally by (1) direct inhalation of polluted air and tobacco smoke; (2) dietary intake of smoked and other foodstuffs and polluted water; and (3) dermal contact with soot, tars and polluted soils.

The production of styrian pumpkin seed oil requires a roasting of the pumpkin seeds, generally higher than 60 °C. The roasting at elevated temperatures produces the typical taste of the pumpkin seed oil but may also be the source for a contamination with PAHs. Since norms and legal limits for hot-pressed oils are missing the legal limits of 25 µg/kg for the sum of 16 PAHs and 5 µg/kg for the heavy fraction for refined and cold-pressed oils, such as olive oil and others, have to be applied.

Separation of hydrophobic analytes from oily matrices can be performed by GPC, dialysis, caffeine-complex formation or saponification with alcoholic potassium hydroxide. Traditional saponification methods need at least 40 min, microwave-assisted saponification is completed within 10 min. Therefore, sample preparation was performed by microwave-assisted saponification with 1.5 M methanolic potassium hydroxide using a Multiwave 3000 from Anton Paar GmbH (Graz, Austria) followed by a liquid-liquid extraction. Further purification was achieved by extraction with sulfuric acid (76 %) and a final clean up procedure by adsorption chromatography on activated silica gel combined with a preparative Bondesil-cyano phase. For a selective measurement of the individual analytes, gas chromatography combined with mass spectroscopy was used in single ion monitoring mode [1].

The whole analytical procedure was validated by systematic recovery experiments.

[1] Gfrerer M, Serschen M, Wenzl T, Gawlik BM, Lankmayr E. *Chromatographia* 55 7/8 (2002) 467-473

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**MEMBRANE EXTRACTION WITH A SORBENT INTERFACE-GAS-  
CHROMATOGRAPHY AS EFFECTIVE AND FAST MEANS FOR  
CONTINUOUS MONITORING OF THERMAL DEGRADATION  
PRODUCTS OF POLYMERS**

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