

M6 WATER-SOLUBLE TERTIARY AMINES FOR SAMPLE PREPARATION IN ATOMIC SPECTROSCOPY: AN OVERVIEW. Joaquim A. Nóbrega, Daniele M. Santos, *Departamento de Química, Universidade Federal de São Carlos, São Carlos, SP, Brasil; Ramon M. Barnes, University Research Institute for Analytical Chemistry, 85 N. Whitney Street, Amherst, MA 01002; Poliana C. Aleixo*, Ana Rita A. Nogueira, Embrapa Pecuária Sudeste, São Carlos, SP, Brasil, and Mark Tatro, Spectrasol Inc., Warwick, NY

Despite the predominance of wet acid digestion methods, would it not be more appropriate to carry out sample preparations in alkaline medium? Alkaline digestion procedures, however, will not become as general as acid digestions owing to their chemical properties, such as the cation hydrolysis at high pH's or for some easily hydrolysable cations even in less acidic solutions. Nevertheless, for some elements and samples, the use of an alkaline medium should be preferred for sample preparation, owing to solubility, matrix and volatility characteristics. This work focuses on the use of water-soluble tertiary amines for sample preparation for atomic spectroscopy. A commercial mixture of tertiary amines called CFA-C (Spectrosol, Warwick, NY) was initially proposed to neutralize hydrofluoric acid. Subsequently, its application has been extended in atomic spectroscopy. Beneficial effects on sample nebulization, isobaric interferences, and sensitivities are observed for inductively coupled plasma mass spectrometry (ICP-MS) and optical emission spectrometry (ICP-OES). Efficient extraction of elements in animal and vegetable tissues, dilution of milk samples, and improvement of automatic sampler action in graphite furnace atomic absorption spectrophotometry also are achieved. These spectroscopic applications are discussed and the main effects are critically reviewed.



M7 CRITICAL EVALUATION OF PRESSURIZED MICROWAVE-ASSISTED DIGESTION EFFICIENCY USING NITRIC ACID OXIDIZING SYSTEMS. Henryk Matusiewicz, Politechnika Poznanska, Department of Analytical Chemistry, 60-965 Poznan, Poland

The possibilities of enhancement of a medium-pressure microwave-assisted digestion system for sample preparation in trace element analysis of biological material were investigated. Based on optimal digestion conditions for oxidizing systems with nitric acid, different digestion procedures were examined to minimize residual carbon. The substitution of nitric acid and the addition of hydrogen peroxide and ozone to nitric acid were evaluated. The residual carbon content of the digestate was determined coulometrically. Addition of hydrogen peroxide during organic oxidation reactions does not lower the dissolved carbon in the solution. Ozone was tested as an additional, potentially non-contaminating, digestion/oxidation system to the nitric acid used in the sample preparation method.



M8 FOCUSED-MICROWAVE-ASSISTED SAMPLE PREPARATION. Joaquim A. Nóbrega, Daniele M. Santos, Lilian C. Trevizan, Letícia M. Costa, Departamento de Química, Universidade Federal de São Carlos, São Carlos, SP, Brasil, and Ana Rita A. Nogueira, Embrapa Pecuária Sudeste, São Carlos, SP, Brasil

Focused-microwave-assisted sample preparation is a suitable strategy when dealing with high masses of organic samples. However, the final acid concentration of the digestate can make difficult routine analytical measurements using spectroscopic techniques. Acids could be evaporated, but this step could be slow even when using microwave-assisted heating and requires a scrubber system for acid vapor collection and neutralization. We are investigating two procedures to decrease the acid concentration of digestates. The first one is based on acid vapor phase digestion of samples contained in PTFE devices [1] inserted into the microwave flask. The acid solution is heated by absorption of microwave radiation, then the acid vapor partially condenses in the upper part of the reaction flask and it is partially collected in each sample container. Calcium, Fe, Mg, Mn, and Zn were quantitatively recovered in samples of animal and vegetable tissues. Better recoveries were attained when adding a small volume of sodium hypochlorite to the sample. This effect is probably related to the generation of chlorine in the sample container after collecting condensed acid. The second procedure developed is based on the gradual addition of liquid samples to a previously heated acid digestion mixture. This procedure was successfully applied for digestion of milk, fruit juices, and red wine. The main advantage is the possibility of digesting up to four-fold more sample using up to ten-fold lower amounts of concentrated acids. Results obtained using both digestion procedures and measurements by ICP-OES with axial view will be presented.

[1] Patent pending, INPI Brazil.

M9 STATE-OF-THE-ART IN SOLID-PHASE EXTRACTION OF BIOFLUIDS. Karl-Siegfried Boos, and C.T. Fleischer, Institute of Clinical Chemistry, University Hospital Grosshadern, D-81366 Munich, Germany

A major drawback of many SPE protocols in bioanalytical LC is a rather unselective and/or time-consuming clean-up procedure. In order to optimise SPE based sample processing with regard to selectivity and speed we investigated tailor-made SPE adsorbents, distinct SPE column hardware as well as a novel software (ChromSword® ADS, Merck KGaA) for LC(MS)-integrated SPE using column-switching.

Restricted Access Materials (RAM) is one family of such special SPE column packings. RAMs are characterized in that they have a defined size-selective exclusion barrier and a non-adsorptive outer particle surface towards macromolecular matrix components (e.g., proteins, nucleic acids, polysaccharides). Low-molecular compounds (e.g., drugs) have free access to the binding centres and thus can be extracted and enriched prior to their analytical separation and MS detection. A RAM-SPE column (e.g., LiChrospher® ADS,

