[1] EU-Patent, EP-B 0 927 574; US-Patent: US 6,210,538 B1.
[2] S. Haiber, H. Berndt, *Fresenius J. Anal. Chem.* **368**, 52 (2000).
[3] P. Jacob, H. Berndt, *J. Anal. At. Spectrom.* **17**, 1615, (2002).
[4] U. Pichler, A. Haase, G. Knapp, M. Michaelis, *Anal. Chem.* **71** 4050 (1999).
[5] G. Knapp, US Patent 5,672,316.

Th6 COMBINATION OF A NEW HIGH-THROUGHPUT ROTOR AND DILUTED ACID FOR CLOSED-VESSEL MICROWAVE-ASSISTED DIGESTION OF BIOLOGICAL MATERIALS.

Joaquim A. Nóbrega, Clésia C. Nascentes, Geórgia C.L. Araújo, Departamento de Química, Universidade Federal de São Carlos, Caixa Postal 676, São Carlos, SP, Brazil - 13560-970, djan@terra.com.br; Ana Rita A. Nogueira, Embrapa Pecuária Sudeste, São Carlos, SP, Brazil, and Camillo Pirola, Milestone, Sorisole, BG, Italy

The state of the art for sample preparation for metal analysis is the use of closed-vessel microwave-assisted procedures. This strategy is gradually becoming the best alternative for

obtaining a digestate suitable for measurements by spectroanalytical techniques. However, the sample throughput still remains as a critical problem that requires the development of reaction vessels and rotors able to work with a large number of samples simultaneously. This is a logical step toward the proposal of procedures that take better advantage of the fast heating speed generated by microwave radiation. Taking into account this aspect, it was recently proposed a 36-vessel rotor with PTFE-coated glass reactors and a self-resealing / auto-venting safety valve that can support up to 13.8 bar (Multiprep-36, Milestone, Italy).

In this work a microwave-assisted procedure was developed for digesting biological materials based on combination of this new rotor and diluted nitric acid plus hydrogen peroxide as digesting mixture. The nitric acid concentrations tested varied from 2 to 7 mol L^{-1} (volume of 2 mL). In all experiments, 1 mL of concentrated H_2O_2 was added to the acid solution. Bovine liver (SRM 1577b, NIST, Gaithersburg, USA) spinach leaves (SRM 1570a, NIST), and *Paspalum notatum*, a forage used for animal feeding, were used as samples. All them were successfully digested using a 5-step microwave heating program (total time: 19 min and maximum temperature: 170°C).

The contents of metals in digestates were determined using an inductively coupled plasma optical emission spectrometer with axial configuration (Varian, Vista axial view, Australia). Depending on the analyte, even 2.0 mol L^{-1} HNO_3 was enough for attaining quantitative recoveries. Barium, Ca, K, P, and Zn were quantitatively recovered even when using 2 mol L^{-1} HNO_3. Aluminum and Fe required higher amounts of acids for quantitative recoveries.

FAPESP, CNPq, CAPES