Trends in Sample Preparation ´07

Book of Abstracts

Seggau Castle / Austria

26th - 30th June 2007

organized by

TU Graz

Institute of Analytical Chemistry and Radiochemistry

and

Österreichische Gesellschaft für Analytische Chemie

ASAC

Austrian Society of Analytical Chemistry

SPiol 30742

Poster 04

Investigation of reaction products resulted from biological materials after pressure digestion with diluted nitric acid

A.R.A. Nogueira[1], M.H. Gonzalez[1,2], J.A. Nóbrega[3], L. Forato[4], R.V. Oliveira[3]

1) Embrapa Pecuária Sudeste, São Carlos SP, Brazil
2) Instituto de Química de São Carlos, Universidade de São Paulo, São Carlos SP, Brazil
3) Departamento de Química, Universidade Federal de São Carlos, São Carlos SP, Brazil
4) Embrapa Instrumentação Agropecuária, São Carlos SP, Brazil
anarita@cppse.embrapa.br

Microwave-assisted with diluted acids is an alternative to digest organic samples owing inorganic elements solubilization for spectroscopic techniques determination [1,2]. Nitric acid cconcentrates solutions, when warm in closed systems, have NO_2 as main decomposition product. Otherwise when HNO_3 diluted solutions are using, NO is the main reaction product. The digestion efficiency in diluted nitric acid probably is related to the oxidation of the NO with the oxidant atmosphere producing NO_2, reabsorption of this oxide in solution, followed by a disproportion reaction, regenerating to nitric acid. Along the existence of gaseous oxygen in the atmosphere of reaction vial, this cycle remain:

$2\ NO(g) + O_2 \rightarrow 2\ NO_2(g)$

$2\ NO_2(g) + H_2O(l) \rightarrow HNO_3 + HNO_2$

$HNO_2 \rightarrow H_2O + NO_2 + NO$

Moreover, the efficiency of this procedure depends on the original characteristics of the samples. In this way, the decomposition efficiency was evaluated considering the final products present in the solutions after decomposition of plant and animal samples. Grains of soybean and samples of forage, bovine blood, and bovine viscera were digested in cavity-microwave oven using oxidants mixtures in different acid concentrations. The decomposition efficiency was evaluated from residual organic carbon determination and mineral recovery by inductively coupled plasma optical emission spectrometry. The original sample characterization was performed from crude protein amount, fatty, and original carbon. The residual solutions were firstly characterized by spectroscopy technique (1H NMR) to identify main remaining organic compounds [2]. After the first results, separation studies were performed by high performance liquid chromatography with UV detector. The reaction products were correlated with the sample's initial chemical composition. High concentration of acid (14 mol L^{-1} HNO_3) resulted in higher variety of organic compounds, such as nitro-, aliphatic- and aromatic- compounds when compared to less concentrate solutions (2 mol L^{-1} HNO_3). It could be explain by the high oxidant power provide by the high pressure and high temperature condition, generating more organic residues. Comparing the digested solutions with the original sample composition, biological matrix with structural amino acids, proteins and lipids produced nitrobenzoic acid isomers and other organic compounds provided from the cleavage in chemical bonds. The m-NBA was obtained in the majority of the digestion solutions.

[1] Y.Z. Chao; M.K. Wong; L.L. Koh; Y.C. Wee, J. Anal. Atom. Spectrom., 11,1996, 585-590.
[2] J.C.L. Araújo, M.H. Gonzalez, A.G. Ferreira, A.R.A. Nogueira, J.A. Nobrega, Spect. Acta B, 57, 2002, 2121-2132.