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CHEMOMETRIC CHARACTERIZATION OF ANIMAL-DERIVED SAMPLES BY INDUCTIVELY COUPLED PLASMA OPTICAL EMISSION SPECTROMETRY AND MICROWAVE-ASSISTED SAMPLE PREPARATION.

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Sample pretreatment is generally necessary in the majority of the analytical methods. In elemental analysis, most of the analytical techniques require the solid sample to be transformed into a homogeneous liquid phase [1]. Microwave-based techniques have been efficiently used in the decomposition of a variety of samples for elemental analysis [2]. A microwave-assisted acid-decomposition procedure is proposed for sample preparation of a variety of bovine-derived materials prior to multielement determination by inductively coupled plasma optical emission spectrometry (ICP-OES). Chemometric approaches such as principal component (PCA) and hierarchical cluster (HCA) analysis were applied to sample characterization.

Initially, the procedure involved sample grinding in a cryogenic mill to produce more uniform particles in the final homogenate, which improves the method precision. Samples (300 mg) were acid decomposed at high pressure and temperature employing a reagent mixture of 2 mL of concentrated HNO_3 and 1 mL of 30% v/v H_2O_2. A simultaneous Varian Vista RL ICP-OES with radial view configuration operated with a concentric nebulizer was used for determination of Al, Ba, C, Ca, Cu, Fe, K, Mg, Mn, Na, P, S and Zn in blood, hide, carcass, viscera, ribs, head and feet samples. Accuracy of the elemental analysis was indicated by the good agreement between certified and found values of the elements in bovine tissues at 95 % confidence level. The microwave assisted decomposition procedure was fast and reproducible, and proved to be efficient for element analysis by sensitive multielement techniques such as ICP-OES.

Principal component analysis and HCA were applied to the data matrix (120 ´ 12) of the element composition of each sample by using the Pirouette 3.0 version program. The data were autoscaled due to differences in the order of magnitude of the variables response. The dendrograms in the HCA were obtained using Euclidean distances and the incremental clustering technique. This analysis greatly supported the principal component analysis. The PCA and HCA supplied information on the common and discrepant characteristics among the investigated samples according to their element composition. Barium, Fe, K, Mn, Na, and S have been found to be the most discriminating variables.

[1] H.M. Kingston and S.J. Haswell, eds. *Microwave-enhanced Chemistry: Fundamentals, Sample Preparation and Applications*, ACS, Washington DC, 1997.

[2] R.C. Richter, D. Link and H.M. Kingston, *Anal. Chem.*, 2001, **73**, 30A.