

Acid Vapor Partial Digestion of Biological Samples in a Focused Microwave:
Multielement Determination by ICP OES with Axially-Viewed Configuration

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75

Sample preparation is an important step before instrumental measurements. Emphasis has been placed on the development of fast and contamination-free methods for determination of trace elements. Vapor-phase partial digestion is an alternative procedure for sample preparation, which allows the simultaneous purification of the acid, reduces the risk of contamination and improves detection limits by reducing analytical blanks. In this work, an acid vapor partial digestion method was proposed using a focused microwave oven (STAR-System 6, CEM, NC, USA) operating at atmospheric pressure. A laboratory-made PTFE support vessel equipped with four sample cups was adapted to fit on the microwave glass vessel. Method accuracy was checked by using bovine liver certified reference material (NIST 1577b, MD, USA). Samples were directly weighed (50 mg) into PTFE cups followed by addition of 150 μ l H₂O₂. The mixture was exposed during 25 min to acid vapor steaming from 15 ml of concentrated HNO₃ placed in the bottom of the glass vessel. The acid vapor was generated at 115 °C. The resulting suspension was diluted with 0.14 mol l⁻¹ sub-boiled HNO₃ to a final volume of 3 ml. Aluminum, Cu, Fe, Mg, Mn, Zn, and the residual carbon content (RCC) were determined by inductively coupled plasma optical emission spectrometry (VISTA AX, Varian, Australia) with axially-viewed configuration. Table 1 shows the results obtained with the proposed method.

Table 1. Determination of Al, Cu, Fe, Mg, Mn and Zn (mg kg⁻¹) in bovine liver (n = 3)

Element	Certified	Determined
Al	3*	1.45 ± 0.28
Cu	160 ± 8	140 ± 12
Fe	184 ± 15	117 ± 26
Mg	601 ± 28	540 ± 7
Mn	10.5 ± 1.7	8.48 ± 0.11
Zn	127 ± 16	117 ± 6

* non-certified value

Except for Al, element recoveries were between 64% and 111% and RCC's in the digestates were 25 ± 5% (n = 3). As an additional advantage of the proposed method, up to 24 samples can be simultaneously prepared when using a 6-cavity focused microwave.

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