

**AAS - Determination of Mercury in marine reference materials after wet ashing by means of microwaves**

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The application of microwaves (2450 MHz) to the mineralization of environmental samples for the purpose of the determination of mercury has been investigated.

The method was tested on 5 reference materials produced by the IAEA : MA-M-2/TM (mussel homogenate), MA-A-3/TM (shrimp homogenate), MA-B-3/TM (fish homogenate), IAEA-350 (tunny-fish, lyophilized) and SD-M-2/TM (deep-sea sediment). The concentration of mercury in these materials is either certified or known with an acceptable degree of accuracy.

The digester "Microdigest" - 300 (PROLABO) was used for the mineralization of these samples. Mineralization tests were done under various operating conditions : microwave energy, duration of operation, oxidizing reagents (HNO<sub>3</sub> - H<sub>2</sub>O<sub>2</sub> and HNO<sub>3</sub> - H<sub>2</sub>SO<sub>4</sub> - H<sub>2</sub>O<sub>2</sub>), condenser type (Vigreux column or Liebig column). Resulting solutions were analysed by cold-vapour atomic absorption spectrometry (HATCH 1 OTT, 1968). Results are given in Table 1. For each material the mean value of the individual determinations is compared with the consensus value (certified) or the expected value (non-certified). The agreement between these values is good (less than  $\pm 10\%$  deviation) in the case of shrimp homogenate MA-A-3/TM and lyophilized tunny-fish IAEA - 350 (the expected value for this sample was obtained in our laboratory by applying a conventional wet-ashing method). The mean value is lower than the certified value for the mussel homogenate MA-M-2/TM (-23%) and higher than the certified value in the case of the sediment SD-M-2/TM (+22%).

The applications of WILCOXON's non-parametric test showed that results of "strong attacks" (high microwave energy and long duration) are significantly higher than results of "weak attacks" for all analysed materials ( $p < 0.01$ ). Results obtained by use of the Liebig condenser seem also to be higher than results obtained with the Vigreux column but the difference is less significant ( $p < 0.10$ ). The effect of the addition of sulphuric acid is not systematic and depends on the material analysed.

Microwave mineralization coupled with cold-vapour atomic absorption spectrometry is a rapid and efficient analytical method for the determination of mercury in environmental materials. It takes about 15-30 minutes to solubilize mercury from the samples instead of several hours with the classical wet-ashing method.

**Table 1. Results of Microwave mineralization**

Sample N°	Oxidizing reagents (1)	Type of attack (2)	Condenser used
1	A	weak	Vigreux
2	A	strong	Vigreux
3	A	strong	Liebig
4	B	weak	Vigreux
5	B	strong	Vigreux
6	B	strong	Liebig

(1) A : 10 ml HNO<sub>3</sub> 65% followed by 2 ml H<sub>2</sub>O<sub>2</sub> 35%

B : 7 ml HNO<sub>3</sub> 65% + 3 ml H<sub>2</sub>SO<sub>4</sub> 98% followed by 2 ml H<sub>2</sub>O<sub>2</sub> 35%

(2) weak attack : A : max energy 10%, duration 15 min.  
B : " " 5%, duration 20 min.

strong attack : A : max energy 15%, duration 20 min.  
B : " " 15%, duration 35 min.

**Results of mercury determinations ( $\mu\text{g g}^{-1}$ )**

Sample N°	MA-M-2/TM	MA-A-3/TM	MA-B-3/TM	IAEA-350	SD-M-2/TM
1	0.70	1.53	0.45	3.11	0.065
2	0.75	1.69	0.43	3.94	0.080
3	0.75	1.82	0.53	4.31	0.076
4	0.68	1.64	0.41	4.24	0.057
5	0.75	1.82	0.47	4.39	0.064
6	0.73	1.96	0.47	4.65	0.057
Mean value	0.73	1.74	0.46	4.11	0.066
Expected value	0.95(1)	1.79(2)	0.51(1)	4.42(3)	0.054(1)
Rel. deviation	-23.2%	-2.8%	-9.8%	-7.0%	+22.2%

(1) certified (2) non-certified (3) conventional wet-ashing

Correlation : mean value (y) - expected value (x) :

$$y = 0.938x - 0.028$$

$$r = 0.9987 (p < 0.001)$$