

# Arsenic in marine fish tissue by ETAAS: study of significant difficulties

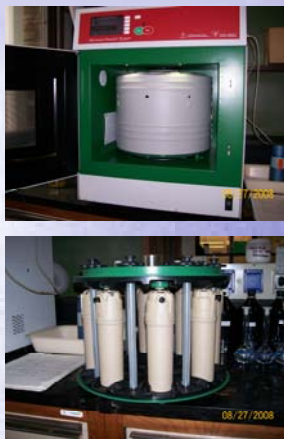
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## INTRODUCTION

It is well known the importance of arsenic determination in fish tissues of marine origin. There are presently difficulties in determining total arsenic in this matrix. It is analyzed the influence of the sample treatment, and the effects due to matrix interference, chloride and arsenobetaine, this compound is difficult to decompose and can remain intact after some digestion procedures. The method validation is carried out using certified reference materials: DORM-2, DOLT-2, TORT-2, all of them from INMS-NRC and BCR-626 (Arsenobetaine solution) from EC. The trazability to S.I. is assured by arsenic standard solution SRM3101a from NIST. A swordfish sample belonging to the CCQM-K43.1 is analyzed.

## EXPERIMENTAL



**Sample treatment:**  
With microwave reaction system  
Multiwave 3000 Perkin Elmer/Anton Paar. Using 1 or 2 power step.

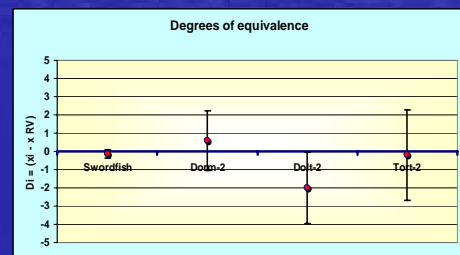
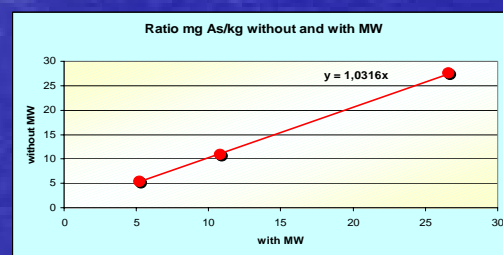
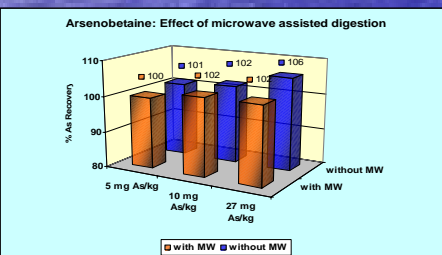
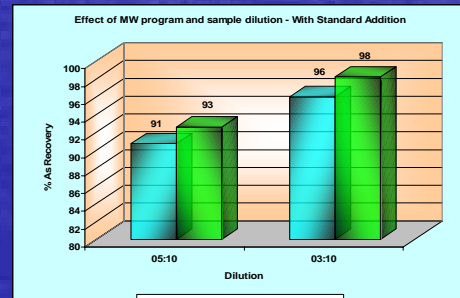
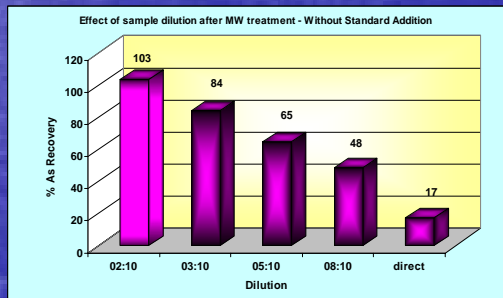
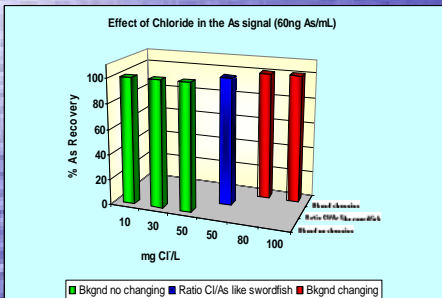
Program: Muscle (with 1 power step)		Closed vessel: PTFE-MFL		
Sample weight: 0.5 g		Reagents: 5 mL HNO <sub>3</sub> + 2 mL H <sub>2</sub> O <sub>2</sub>		
Final volume: 50mL		p: rate 0.5 bar/s 60bar		
Step	Power (w)	Ramp (min)	Hold (min)	Fan
1	1200	15:00	30:00	1
2	0	0	15:00	3

**Measurement:**  
With 5100ZL Perkin Elmer. THGA  
With Zeeman-effect background correction. Pd+Mg matrix modifier. STPF conditions.

Step #	Temperature (°C)	Ramp Time	Hold Time	Internal Flow	Gas Type	Micro Sp/L	Read
1	110	1	10	250	N	0	0
2	130	15	10	250	N	0	0
3	1800	10	10	200	N	0	0
4	2300	0	5	0	N	0	0
5	2400	1	5	250	N	0	0
6	250	0	0	250	N	0	0



## RESULTS



Swordfish (CCQM-K43.1)	Swordfish Reference Value Proposed	Tort-2	Tort-2 Reference Value	
As (mg/kg)	6.53 ± 0.22	6.65 ± 0.09	21.4 ± 1.7	21.2 ± 1.8
Dorm-2	Dorm-2 Reference Value	Dolt-2	Dolt-2 Reference Value	
AS (mg/kg)	18.6 ± 1.2	18.0 ± 1.1	14.6 ± 1.6	16.6 ± 1.1
DL: 0.8 mg/kg		CL: 2.5 mg/kg		

## CONCLUSION

- There are not significant difference between 1 or 2 step power in the MW wet digestion, using HNO<sub>3</sub> + H<sub>2</sub>O<sub>2</sub>.
- Concentration of chloride between 10 to 100 mg/L, do not interfere with the As signal of 60 ng As/mL, if THGA with Zeeman effect background correction and stabilized temperature platform furnace (STPF) condition with Pd+Mg matrix modifier are used.
- The results for total As in arsenobetaine with and without microwave assisted wet digestion are similar.
- The solution obtained after wet digestion should dilute at least 5 fold.
- Very good agreement with certified or reference values are obtained by this methodology.

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