

Application Note for Speciation Analysis

Species : Methyl Mercury

Sample Type : Lobster Hepatopancreas (TORT-2)

Sample Category : Biological Tissue

Analysis : Species-Specific-Isotope Dilution Analysis + GC-ICP-MS

Sample Preparation Procedure

Recommended Equipment

Sample Weight : 0.1g

Isotopic Spike¹ : ²⁰¹Hg enriched MeHg

Reagent(s) : 4mL TMAH (25%)

1

Microwave Heating Program

Temperature Target: 70°C

Ramp Time: 1 minute

Heating Time: 4 minutes



Explorer 24



Explorer 'S' Class

2

Derivatization Procedure

1mL Extract + 3mL buffer HAcO/AcO 0,1 M at pH 3,9

Adjust pH to 3.9

Add 0,5 mL Hexane

Add 1mL NaBEt₄ (2%w/v)

5 minutes mechanical shaking

3

Clean Up Procedure

Separate Organic Layer

Pass Through Florisil Column

Elute with 4mL of Hexane and collect

Preconcentration with N₂ or Ar

Florisil Column

N-evap or equivalent

4

Chromatographic Separation

T Injector - 250 °C / Splitless 0.5min

40 °C(0.5min) to 280 °C (4min)

Ramp Rate : 30 °C / min

Tr-5 Column

5% Phenyl Methyl Siloxane

30m x 0.25mm i.d. x 0.25 µm coating

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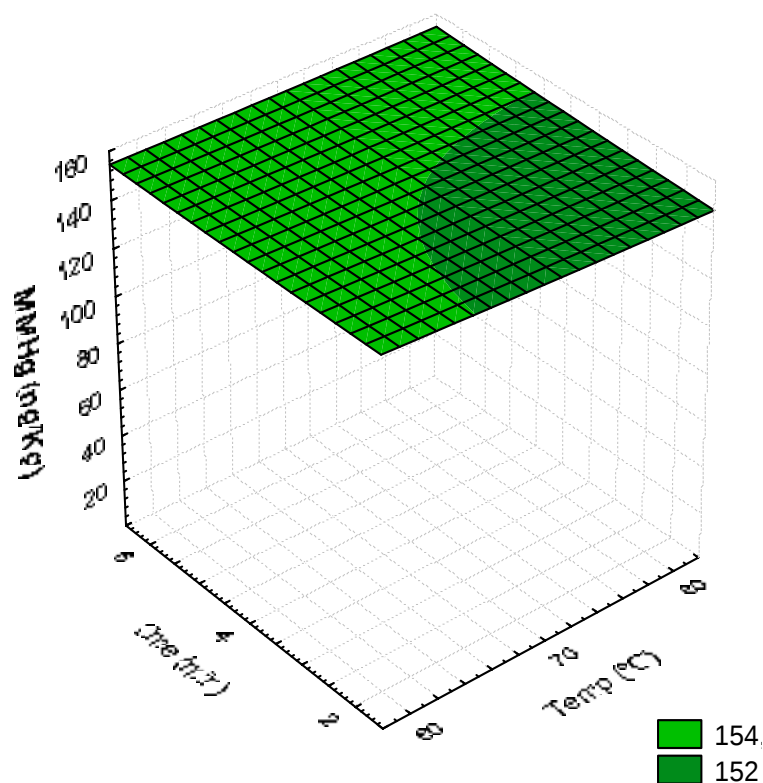
Detection and Analysis

Equipment Used



GC-ICP-MS: Focus GC & Thermo X series

Optimization of Sample Preparation



Results ng Hg / g

60°/ 2 min	152
60°/ 4 min	155
60°/ 6 min	154
70°/ 2 min	156
70°/ 4 min	148
70°/ 4 min	148
70°/ 4 min	158
70°/ 6 min	157
80°/ 2 min	145
80°/ 4 min	158
80°/ 6 min	151

Average 153

Stdev 5

RSD 3%

Certified Value : MeHg 152 +/- 13 ng Hg/g²

¹ In-house synthesized isotopically enriched MeHg: *Appl. Organomet. Chem.*, 2002, 16, 610-615. Addition of the enriched species was done in a range of 1-5 times the expected concentration in the tissue.

² Be aware that the use of species-specific isotope dilution analysis prevents the degradation of methylmercury during the extraction.