CEM

Application Note for Speciation Analysis

Species : Methyl Mercury
Sample Type : Tuna Fish (BCR-464)
Sample Category : Biological Tissue
Analysis : Species-Specific-Isotope Dilution Analysis + SPME-GC-MS

Sample Preparation Procedure

Sample Weight : 0.1g
Isotopic Spike : ^{201}Hg enriched MeHg[1]
Reagent(s) : 4mL TMAH (25%)

1 **Microwave Heating Program**
Temperature Target : 70°C
Ramp Time : 1 minute
Heating Time : 4 minutes

2 **Derivatization Procedure**
1mL Extract + 3mL buffer HAc/AcO 0,1M pH 3,9
Adjust pH to 4.0[2]
Add 1mL $NaBEt_4$ (2%w/v)
Close SPME vial — **Magnetic stirring for 5 min**

3 **Clean up Procedure**
Insert SPME fiber — **DVD/CAR/PDMS 50µm/30µm**
Sampling — **Room temperature during 15 min**

4 **Chromatographic Separation**
T Injector - 250 °C / Splitless 1 min
40 °C(1min), 15 °C/min to 90 °C
30 °C/min to 280°C
Carrier gas: He (1 mL min^{-1})

DB-5MS Column
5% Phenyl Methyl Siloxane
30m x 0.25mm i.d. x 0.25 µm coating

Recommended Equipment

Explorer 24

Explorer 'S' Class

File: tuna1.doc

Detection and Analysis

Equipment Used

GC-MS: JEOL JMS-Q1000GC

MeHg concentrations obtained in BCR-464 (tuna fish): 4967 ± 87 ng Hg/g[3]

Certified Value : 5117 ± 160 ng Hg/g

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[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] With HCl conc. or NH_4OH conc.

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