

# Determination of lead and cadmium in fish

## EN 14084:2003

The method analyze samples that are digested in closed vessels, using nitric acid and a microwave oven. The resulting solution is diluted with water, and the lead and cadmium contents are determined by graphite furnace atomic absorption spectrometry (GFAAS) with matrix modifier.

**Reagents** (*only use recognized analytical grades, unless otherwise specified.*

*Water should comply with at least grade 3 in accordance with ISO 3696)*

*Thus use only reagents/water, with an element level low enough not to affect results.*

Water, LiChrosolv® (1.15333)

Nitric acid 65% Suprapur® (1.00441)

Magnesium nitrate hexahydrate 99.99 Suprapur® (1.05855)

Lead (1000 mg/l Pb in HNO<sub>3</sub> 0.5 mol/L) traceable to SRM from NIST. CertiPUR® (1.19776)

Cadmium (1000 mg/l Cd in HNO<sub>3</sub> 0.5 mol/L) traceable to SRM from NIST CertiPUR® (1.19777)

### **Apparatus:**

Laboratory mill (e.g. knife mill)

Laboratory microwave oven

Atomic absorption spectrometer

Graphite tubes

Element specific lamps

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### Procedure:

1. Homogenize the sample with a laboratory mill.

*Suggestion: in some cases, the drying of the sample is needed in a way that does not affect the element contents, e.g. by freeze drying.*

### 2. Sample preparation

- Weigh 0.5–1.5 g of sample in a vessel.
- Add 5.0 mL of nitric acid. After 30 minutes add 5.0 mL of distilled water and mix it gently.
- Allow samples to predigest by standing open for a minimum of 15 minutes before sealing vessels and proceeding to heating program.

Typically an oven programme includes a stage at low power with increasing temperature for a few minutes followed by one or more stages at higher power settings. A gradual increase between the selected stages is recommended in order to prevent sudden pressure peaks to occur inside the pressure vessels.

*Suggestion: samples with high carbon content (e.g. sugar, fat) may cause sudden pressure peaks during the process. Allow these samples to predigest by standing overnight.*

### 3. Microwave heating program

Step	Time (min)	Temperature (°C)
1	20	Up to 200 °C
2	20–30	200 °C
3	From 30 and onwards	Cooling down

*Suggestion: when digesting unknown samples, take care since a too large sample amount may rupture the safety membrane of the digestion vessel. In particular, samples with high carbon content (e.g. sugar and fat) may cause sudden pressure peaks during the process. In all cases, the sample intake should be in strict compliance with the manufacturers recommendation.*

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### 4. Measurement with graphite furnace technique

Graphite furnace technique is required for determination of lead and cadmium. Use pyrolytically coated tubes with platforms. Program the autosampler to deliver sample volume to the graphite furnace, which gives a background absorbance of not more than about 0.5 absorbance units. Instrumental parameters with an injection volume of 20 µL.

Element	Wavelength (nm)	Background Correction	Parameter	Step 1	Step 2	Step 3	Step 4
Cd	228.8	Zeeman	Temp (°C)	110	450	1000	2500
			Ramp (°C/sec)	10	150	0	0
			Hold (sec)	30	20	3	3
Pb	217	Zeeman	Temp (°C)	120	800	1200	2500
			Ramp (°C/sec)	10	150	0	0
			Hold (sec)	10	20	3	3

### 5. Calculation

Construct a standard curve and read the concentration of the metal from the curve. Calculate the content (c), as mass fraction of the element to be determined in µg/kg of sample:

$$c = \frac{(a-b) \times V}{m}$$

a: is the concentration in the sample solution in µg/L

b: is the mean concentration in the reagent blank solution in µg/L

V: is the volume of sample solution in mL

m: is the sample mass in gram

Result:

(in case of sample 2015/33632

- Patagonotothen spp - fish)

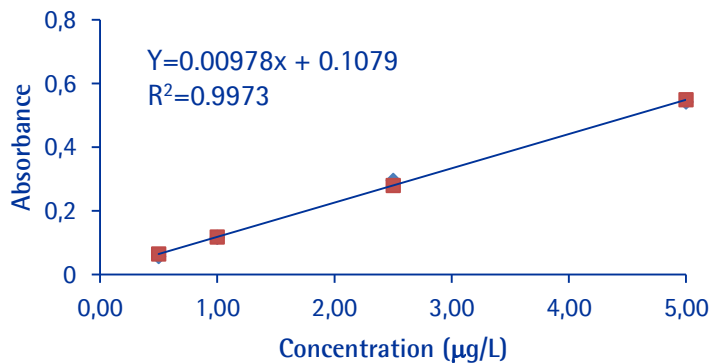
	Lead	Cadmium
m (g)	1.0115	1.0015
a	0.8493	0.3025
b	-	-
V (mL)	100	100
C (µg/kg)	84	29.9

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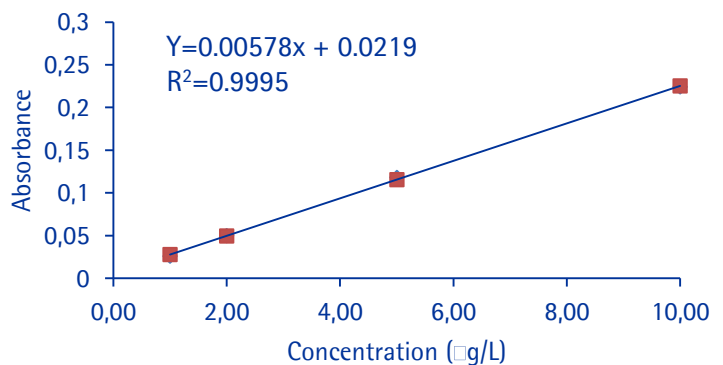
### 6. Calibration data – Cadmium (Cd)

Sample ID	"SignalAbs (Height)"	"Conc (µg/L)"
Cd Blank	0,002	0,0000
Cd standard 1	0,056	0,5000
Cd standard 2	0,116	1,0000
Cd standard 3	0,296	2,5000
Cd standard 4	0,542	5,0000
Cd Blank	0,002	0,0005
Cd Blank	0,002	0,0065
Cd 33632	0,034	0,3025
Cd Ha94	0,191	1,6413
Cd 2.5 ppb std	0,296	2,4974
Cd blank	0,002	0,0005



### 7. Calibration data – Lead (Pb)

Sample ID	"SignalAbs (Height)"	"Conc (µg/L)"
Pb Blank	0,003	0,0000
Pb standard 1	0,026	1,0000
Pb standard 2	0,050	2,0000
Pb standard 3	0,118	5,0000
Pb standard 4	0,224	10,0000
Pb Blank	0,003	0,0022
Pb Blank	0,002	-0,0218
Pb 33632	0,022	0,8493
Pb Ha94	0,036	1,4433
Cd 25.0 ppb std	0,122	5,2115
Pb Blank	0,002	-0,0524



Pb Ha94 and Cd Ha94 are reference materials