A unified extraction and clean–up procedure for PCDD/PCDF determination in various matrices by GC–MS

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A universal extraction and clean–up procedure for routine PCDD and PCDF determination in various matrices (soils, sediments, surface and waste water, fly ash, plant, animal and fish tissues, blood, milk etc.) is developed based on reported data and own experimental results.

The procedure includes various extraction steps for different matrices:

- **for water samples**
  liquid–liquid extraction of large volumes of water (up to 20 l) directly in the bottle by mixing with hexane/methylene chloride (55:45 v/v)

- **for solid samples (soils, sediments, plant, animal and fish tissues etc.)**
  mixing with Na_2SO_4 and eluting the mixture in a glass column by hexane/methylene chloride (50:50 v/v)

- **for fly ash samples**
  extraction by boiling toluene during 24 hours after treatment by HCl

- **for air samples collected on filter and XAD–2**
  extraction by hexane–acetone

- **for milk samples**
  extraction by mixture hexane/ether with adding of ethanol and sodium oxalate and fat determination by weighing, followed by dissolving in hexane/methylene chloride (50:50 v/v)

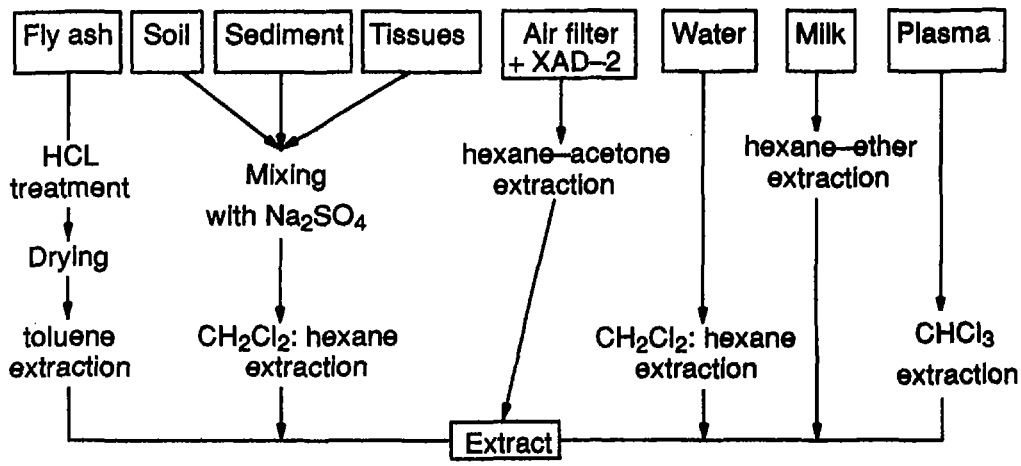
- **for blood samples**
  extraction by chloroform with methanol. Extracts are evaporated to dryness and dissolved in hexane.

Clean–up procedure is alike for extracts of all matrices. It includes some stages:

- "Multilayer" column with layers of silica, three layers of silica impregnated with H_2SO_4, alternating Na_2SO_4 and silica layers, silica impregnated with CsOH, layers of Na_2SO_4, and silica; these alternating layers are used for more effective clean–up and as indicators of the clean–up efficiency; "dirty" samples can be pre–cleaned by vigorous shaking with silica impregnated with H_2SO_4.
- Effluent from the "multilayer" column is transferred to the column with carbon on celite, washed with hexane/methylene chloride (50:50), methylene chloride–methanol–benzene (75:20:5) followed by PCDD/PCDF elution with reverse flow of toluene.
- The final step is PCDD/PCDF solution clean–up using a column with alumina, eluted consecutively by hexane, hexane–methylene chloride (95:5) and hexane–methylene chloride (50:50).

Therefore this procedure includes special extraction modes for each matrix and a unified clean–up technique for extracts. It can be automatized and easily modified for special problem solutions and any new matrix analysis. This unified procedure was used in the laboratory during two years for PCDD/PCDF determination in water, soils, sediments, milk etc.

Extraction and clean–up scheme



Drying by Na_2SO_4

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Concentrating and solvent changing to hexane

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H_2SO_4/SiO_2 treatment

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"Multilayer" column

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Carbon column

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Concentrating and solvent changing to hexane

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Aluminia column

↓

Concentrating

↓

GC–MS

ANA