Extraction of Dioxins and PCBs from Food and Feed

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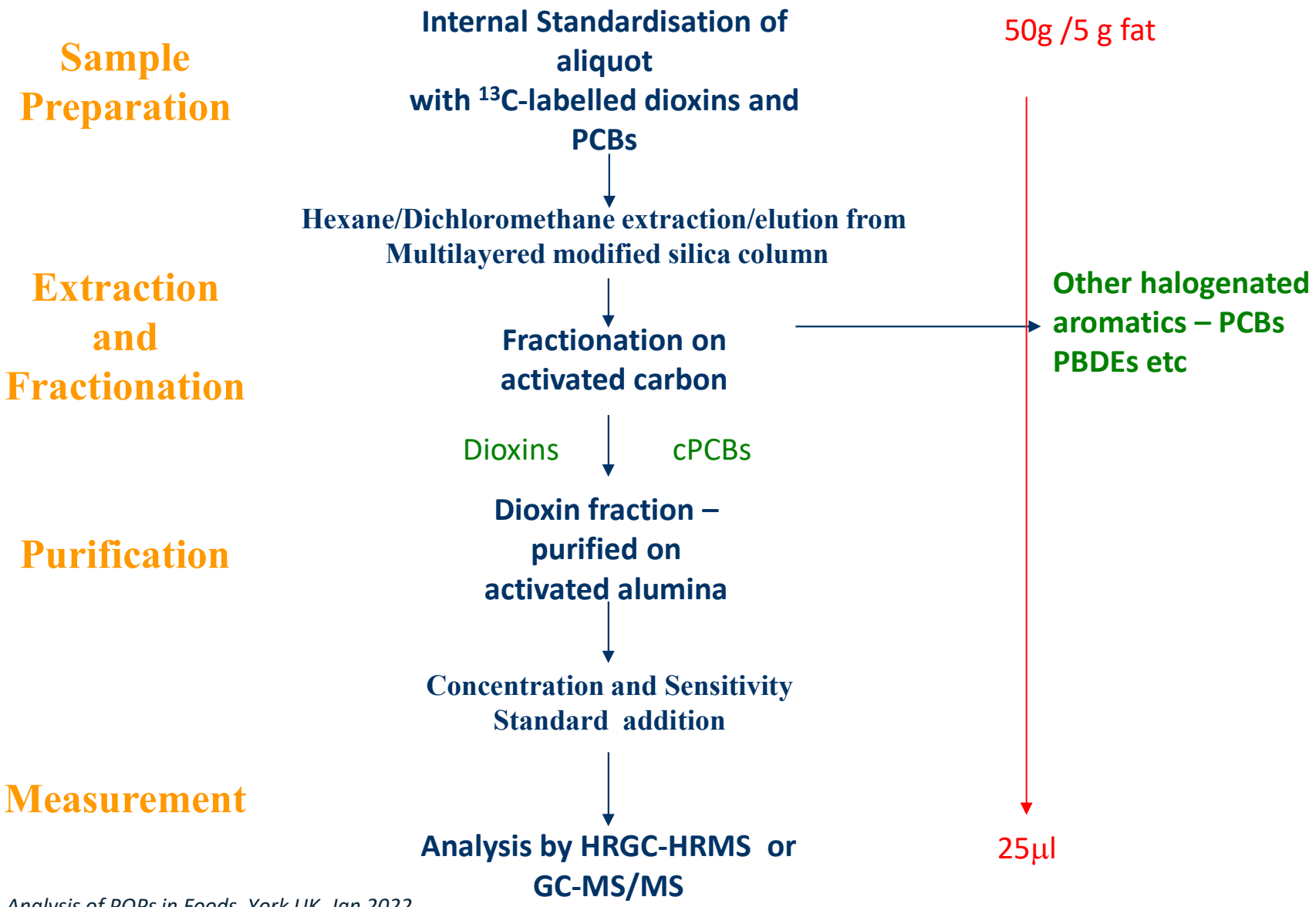
Principle

- The subsample is fortified with appropriate $^{13}C_{12}$- labelled internal standards and subjected to exhaustive solvent extraction.
- The crude extract is mixed with solvent and sulphuric acid impregnated silica gel to remove co-extracted materials, including lipid and labile chemical contaminants.
- The slurry produced by this is quantitatively transferred to the large column upon which further extraction occurs.
- The eluent from the large column is passed through a column containing activated carbon. This retains the planar compounds of interest (PCDD/Fs, PBDD/Fs and non-ortho-PCBs/PBBs) whilst allowing the ortho-PCBs/PBBs and PBDEs to pass through.
- The planar compounds are eluted as a separate fraction

Method Schematic



Large Multilayered Column

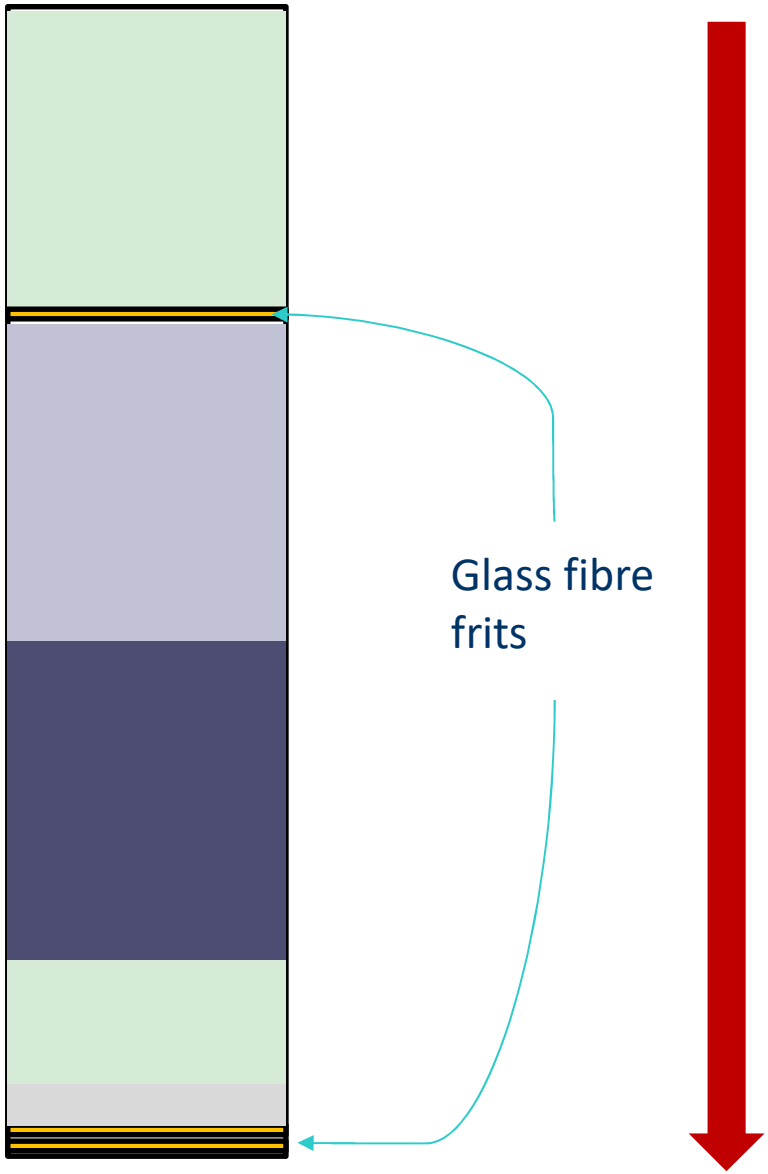
$NaSO_4$, 30 g to 50 g

Acid modified silica, 50 g

Base modified silica, 50 g

$NaSO_4$, 10 g

Silanised Glass wool



Reagent Preparation

$NaSO_4$

- Washed with dichloromethane (2 x 3 L, then 4 L)
- Elute under N_2
- 120°C to 140°C for at least 12 hours
- Place in crystalising dishes in muffle oven for 5 to 9 days to remove water

Reagent Preparation

Silica Gel, activated

- Washed with dichloromethane (12.5 L then 10 L)
- Elute under N_2
- 130°C to 140°C for at least 12 hours
- Place in crystalising dishes in muffle oven for 5 to 9 days to remove water

Reagent Preparation

Silica Gel, Base modified

- KOH dissolved in methanol (280 g/L)
- Stir for 20 minutes
- Add silica gel
- Exothermic!!!
- Add methanol, mix slowly for 2 hours
- Elute methanol under N_2
- Elute with dichloromethane under N_2 (2 x 300 mL)

Reagent Preparation

Silica Gel, Acid modified

- Add 1 Kg H_2SO_4 to 1.5 kg activated silica
- Careful!!!
- Roller mix for at least 4 hours
- Free flowing powder

Reagent Preparation

Glass wool, Silanised

- Trimethylchlorosilane (5% v/v) in hexane
- Leave for 30 to 60 minutes
- Rinse with methanol then dichloromethane
- dry at 45°C to 50°C for at least 1 hour

Sample extraction

- Typically, 5g of fat or equivalent sample weight
- Reagent blank: 10 mL hexane
- Internal standard added
- Samples mixed with acid silica and hexane to create a slurry

Sample extraction

- Sample slurry added to top of multilayered column
- PCBs (and PBDEs) eluted with a 600 mL hexane: dichloromethane (60:40)
- Dioxins and non-ortho PCBs stick to carbon column

Sample Clean-Up

PCBs (and PBDEs)

- Only 1/5 of 600 mL extract used – concentrate to 1 mL
- Mini-column with NaSO4 and acid silica
- Elute with 10 mL hexane
- Concentrate to 100 µL, add syringe standard reduce to 50 µL for analysis

Sample Clean-Up

Dioxins

- Reverse carbon column
- Elute with toluene (170 mL)
- Concentrate and exchange to hexane
- Further clean-up on activated alumina

Sample Clean-Up

Dioxins – activated alumina

- Elute with 40 mL hexane:dichloromethane (70:30)
- Concentrate to 100 µL, add syringe standard
- reduce to 50 µL for analysis

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