AUTOMATION FOR THE MEASUREMENT OF SELECTED PBDEs IN HUMAN SERUM

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State of the Art

![Graph showing the number of publications per year for different search terms related to PBDEs.](image)
Methods for Serum

✓ Liquid-Liquid Extraction (LLE)
✓ Solid-Phase Extraction (SPE)

⇒ Disposable cartridge (tube) format

$C_{18}$, Oasis HLB, Diatomaceous earth, Silica, Silica ($H_2SO_4$), …
THE Challenge

✓ Blank levels…

⇒ Impact of the work environment

⇒ (Un)reproducibility of blanks

⇒ Dedicated work place (clean room)

⇒ Minimization of ‘contact’

⇒ Closed-vessel approach
Automated SPE

• 5 samples sequentially
• 5-10 g sample sizes
• 5-10 mL extracts
• Nitrogen drying step
Automated SPE

30 min.
Automated Clean-up

2 g acid, 1 g basic, 0.75 g neutral
Automated Clean-up

- In-line evaporation
- 30 mL Hx/DCM to 500 µL
- Nitrogen flow
- 30°C, heating metal block
400 µL

LVI (80 µL)
Recovery Rates
BCs Automated Procedure
Manual vs Automated (QCs)
Blank Contributions

- PBDE-Free columns: 0%
- Evaporation PwVap: 8%
- SPE barrel-frit (C18): 2%
- Na$_2$SO$_4$: 25%
- SPE module: 18%
- Clean-up module: 47%
PBDE-Free Columns

The graph shows the comparison of PentaBDE residues in different column types:
- Classical columns
- PBDE-free columns
- Blanc Pwp

The x-axis represents the different BDE compounds (BDE-47, BDE-100, BDE-99), and the y-axis represents the concentration (pg) of PentaBDE residues.
Impact of Evap. Device

- Whole BC
- TbVap 2000
- TbVap 2005 (PCB free)
- PowerVap
- Rotavap
Module Contamination?
Module Contamination?
Dust Issue in the Lab...
Take Home Message

✓ Automation is not an issue
✓ Work environment a BIG ISSUE!
✓ PBDE-free consumable are ok
✓ Difficult to precisely trace sources…
✓ PBDE measurement in EU blood requires high level of dedic(education)ation…