Title:

A 'COOK BOOK' METHOD FOR DIOXINs & PCBs ANALYSIS

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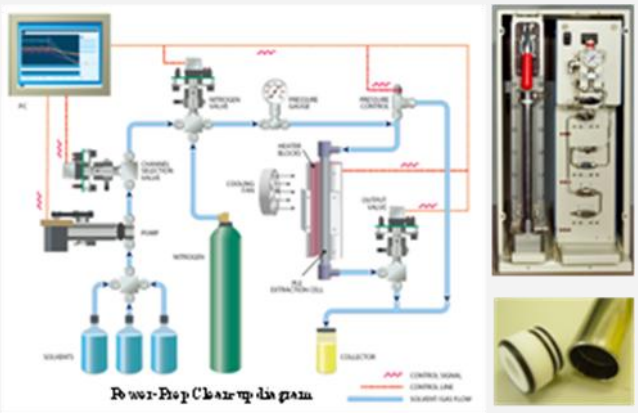
Introduction

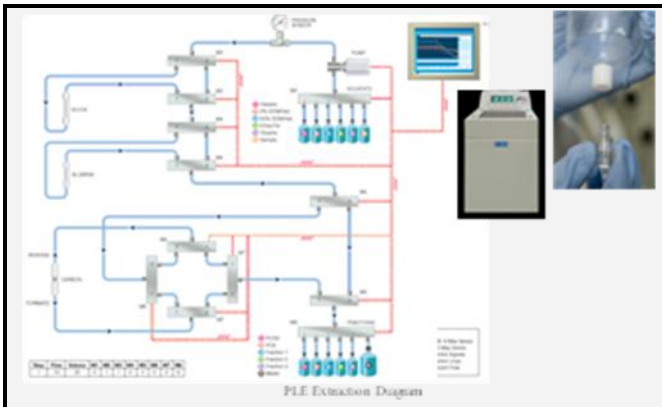
Preparing food samples for dioxin and PCB measurement is laborious. Accurate femtogram measurement requires high standard analytical strategies, time, extensive know-how, and money. Additionally, matrix-related interferences are present in concentrations at orders of magnitude higher than the analytes of interest. A complex multi-step approach is required to extract (PLE), isolate (LC), separate (GC) and quantify (MS) analytes of interest under strict quality assurance/quality control (QA/QC) criteria. Accredited laboratories often require a week or more for accomplishing the job because of the complex sample preparation procedure. Efforts to develop alternative procedures are carried out to cut the cost, improve the sample throughput, and ease the implementation of the procedure.

Methodology

Extraction: A PLE system was developed (FMS Inc.) to accommodate 1 to 6 samples in parallel in order to match the sample capacity of the automated clean-up system. Extraction procedures can be customized on demand. Classical extraction parameters are 1500 psi, 100-120°C, pure or mixed organic solvent can be used for 1 or several cycle with static or dynamic sequences. Cells sizes range between 5ml and 100ml.



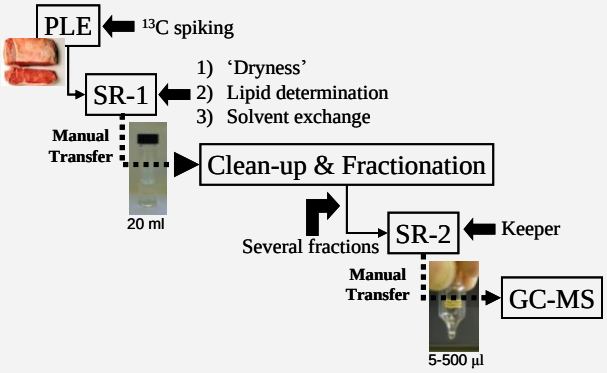
Clean-up: The automated Power-Prep™ system (FMS Inc.) was used for sample clean-up [1,2]. Both PLE and clean-up systems were connected to on-line evaporation devices that ensured proper solvent reductions.



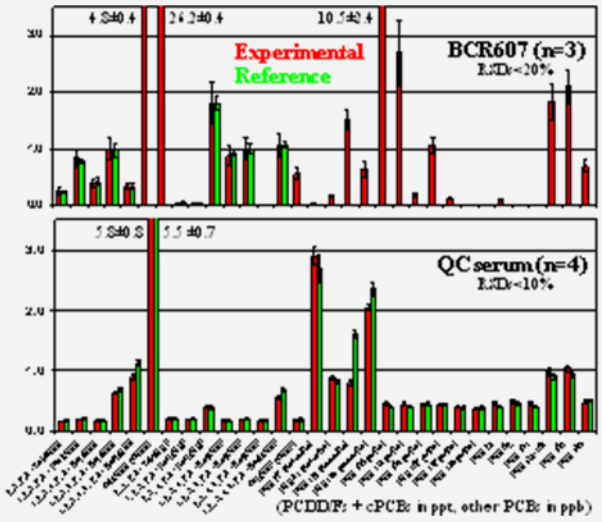
GC-IDHRMS: All data produced using Belac accredited ISO17025 methods.

Results

Both PLE and clean-up systems operate independently. Intermediate solvent reductions and isolation allow gravimetric lipid determination on the go. Additionally, automated solvent reduction take place while sample collection still goes on. From sample reception to GC-MS reporting takes approximately 7h.



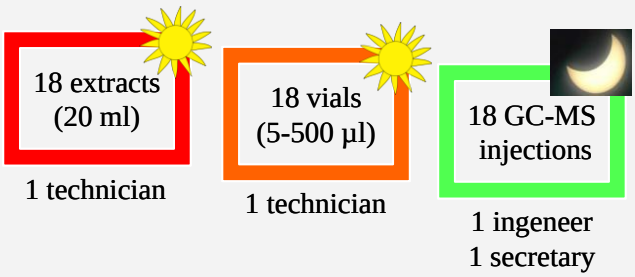
The large internal diameter of the PLE tubing reduces the risk clogging with delicate matrices such as low granulometry animal feed additives. The strategy was successfully applied to various matrices (RSDs<20%, recovery rates oked EU legislation).



Conclusions

- ✓ Same-day physico-chemical testing now feasible
- ✓ GC-IDHRMS high throughput for crisis
- ✓ Potential use in classical food control laboratories

- ✓ Reduced ‘down-time’ in production or exportation
- ✓ Continuous turnover of 18 sample per day with 6 line systems



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[1] Rocand *et al.*, J. Chromatogr. A 925 (2001) 207.
[2] Rocand *et al.*, Talanta 63 (2004) 1101.

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