Sampling, Clean-Up and Separation

Josep Rivera
Institut d'Investigacions Quimiques i Ambientals
CSIC- Barcelona SPAIN

This session is comprised of 27 presentations addressing the different steps in preparing a sample. Different types of sample (environmental, biological, food and feed) are investigated, as well as different groups of compounds ranging from dibenzodioxins and dibenzofurans (PCDD/Fs), organochlorine pesticides, polychlorinated biphenyls (PCBs), polybrominateddiphenylethers (PBDEs) to polycyclic hydrocarbons (PAHs).
Formally, the session will be divided in to separated groups comprising 8 oral and 18 poster presentations.
In the oral Session, **Chung** et al. from China, presents a method for the analysis of PCDD/Fs and DL-PCBs in food stuff, and gives several examples applied to samples of chickenm fish, milk and shelfish. **Jeong** and **Kwon** from Korea,compares Soxhlet, force convection and microwave oven extraction applied to the analysis of food samples. **Bernsmann and Fürst** (Germany), compares the use of accelerated solvent extraction (ASE) with integrated sulphuric acid clean up and Soxhlet extraction for feeding stuffs analysis. **Bert van Bavel** and collaborators from Sweden develop a method for the determination of PBDD/DFs in environmental samples. Some results on fat samples are given. **Robinson** et al.(Australia), presents a "rapid" method based on ASE extraction and clean up on open silica, florisil and alumina columns. Several examples on some biologiacal and environmental CRMs are presented for PCDD/Fs and DL-PCBs. **Mueller** and coworkers from Australia, presents an evaluation of the reproducibility of sample and analysis on a study in soils. PCDD/Fs and DL-PCBs are determined on different sites in Australia. Sampling reproducibility is emphasized.
Koinichi et al. from Japan, describes an interesting "citizen participating campaign", using Pine Needles to monitor PCDD/Fs in air. Results from 56 monitored area monitored in 2003 are presented. **Lamparsky** et al. from USA, describes a simplified sample train for PCDD/Fs determination from incinerators gas emissions. A scheme and quantitative comparison of the new sampling train and Method 23 are given.
As described, in the oral session, several aspects covering the different aspects of the topic (sampling, extraction and clean-up methods) are covered by scientists coming from laboratories in seven different countries.
To overview the *poster session*, the contributions can be examined grouping them putting the emphasis on the part of the methodology (sampling, clean-up or separation) that the authors develop in the presentation:

a) ***Five presentations are involved on the development of sampling methodology***.

Strandberg and col. from Sweden describe the application of Semipermeable membrane devices to the determination of polycyclic aromatic hydrocarbons (PAHs)on indoor air. They discuss the influence of the levels of wood burning as domestic heater.
Bartkow et al. (Australia) study the influence of different wind speeds on Performance Reference Compounds (PRCs) used on PUF passive air samplers. They describe extraction and quantification of PAH on this system.
Lucaciu et al. from Canadá determine levels of DL-PCBs in air samples and vegetation in an area surrounding a metal recovery incinerator. Their results brings to the conclusion that air samplers and foliage can be used to identify sources of DL-PCBs at sub pg/g TEQ levels.
Hashimoto and col. (Japan). The authors compares results of samples from different incinerators, sampled with a standard sampling train and with two described "simple sampling methodologies"
Wielgosinsky et al. study the catalytic destruction of PCDD/Fs, by monitoring, in an experimental installation the levels of o-dichlorobenzene. The vanadium-tungsten catalyst on a monolithic carrier revealed a very good activity.

b) ***Ten presentations describe methodology for extraction of different matrices***.

André et al. from France presents an analytical approach for the analysis of PBDE in biological samples. Further clean-up stages are described, including the use of Oasis HLB for solid phase extraction.
Hodak-Kobsic and coworkers (Croatia) describes de application of Accelerated Solvent Extraction for the determination of PCBs in soil samples and determine efficiencies of extraction and influence of rinsing cycles on ASE 200 operation.
Saito and collaborators from Japan develop a systematic analysis method of dioxins and HCB in human milk. They calculate the increase on the TEQ as a result of the addition of the HCB Toxicity Factor (0.0001).
Gómara and coworkers from Spain, determine the presence of PCBs and DDTs on biotic samples, by introducing the use of a miniaturised sample preparation method followed by GC-Micro-ECD.
Fujimine and col. from Japan, describes a Small Volume method. About 1.0 mL and 1.5 mL are used to analyse PCBs and PBDEs respectively in human blood. The method is compared with their conventional method.
Tachino et al. (Japan), describe the analysis of PCDD/Fs and DL-PCBs in human blood by using 10 mL of sample and improving the sensitivity by the injection trough a large volume injection system (SCLV). They present several chromatograms and the comparison of the coefficient of variation between the recommendation method and the described method.
Sanz and coworkers (Spain), compares the efficiencies of soil and fly ash extractions through the application of a pressurized fluid extraction-PFE (ASE 100) system and a Microwave-Assisted Extraction-MAE. Recoveries comparing Soxhlet and MAE extractions are presented.
Masaaki et al. from Japan, introduces the extraction of dioxins from solutions using a Solid phase MicroExtraction (SPME) system. They present the extraction of a soil and ash

samples through a new system SE-100. After extraction, the sample in solution is followed by SPME-HRGC/HRMS analysis.
Thomsen and col. (Norway), presents a method for the simultaneous extraction of PCDD/Fs, PCBs and PBDEs applied to biological (breast milk) and several different foodstuff.
Marchand et al. from France present a complete work on analysis of PCDD/Fs and DL-PCBs and markers on human blood, comprising information about sample preparation, clean-up and analysis. Complete validation has been performed. The method has been applied to two groups of French volunteers.

c) Four presentations are related with clean-up studies

Hölscher (Germany), the presentation describes a strategy for PCDD/Fs and DL-PCBs applicable to different matrices (feed, milk, fish, meat, oils, fats, vegetables, feed additive), aiming at having results in a minimum time while keeping high quality standards. The method is based on the utilisation of Accelerate Solvent Extraction (ASE or PLE) and Power-Prep clean-up strategy. Data describing recoveries and LOQ are given for different matrices.
Jeong (Korea) describes the optimisation of eluting solvent to find out the best eluting solvent and its amount, by using corn oil as a lipid stimulant. He presents results on the recovery of homologue profiles using Power Prep with toluene as eluting solvent.
Planas et al. from Spain, presents the automatic SPE extraction of 16 pesticides and metabolites from water, with the Power Prep system, using polymeric (ENV+) and C_{18} sorbent phases. Recovery of pesticides are calculated using d_{10}-anthracene as recovery standard.
Matsumura et al. present a full automatic clean-up robot for dioxin and PCB analysis,constituted by one sulphuric acid treatment unit and a column chromatography clean-up unit.