Network of reference laboratories and related organisations for monitoring and bio-monitoring of emerging environmental pollutants

2nd Interlaboratory Study

Validation and Harmonisation of Analytical Methodology for Routine Laboratories: Brominated Flame Retardants (DecaBDE)

Sixth Framework Programme

2nd Interlaboratory Study

Aim

- Validate the harmonized procedure and,
- To assess the applicability of the harmonized procedure for the determination of decaBDE in environmental samples by routine laboratories

Participating laboratories

- Institute of Chemical Technology, Prague, Czech Republic
- CEFAS, Essex, UK
- University of Siena, Italy
- Applus+ LABAQUA, Alicante, Spain
- Unilever, UK
- University of Antwerp, Wilrijk, Belgium
- Waterdienst, Lelystad, Netherlands
- IVM, Amsterdam, The Netherlands
- LANUV, Düsseldorf, Germany
- EMPA, Dübendorf, Switzerland
- Department of Innovation, Industry, Science and Research, Pymble, Australia
- Ministry of the Environment, Ontario, Canada

Procedure

Test samples:

- House Dust
- Sediment sample
- GC-test solution

- Internal standard
  - $^{13}C_{12}$-labelled decaBDE
- Detailed harmonised analytical protocol, reporting sheets and a questionnaire regarding experimental conditions
- Modification of the analytical protocol according to the results

Test Materials (I)

- **House dust**
  - Organic Contaminants in House Dust
  - High concentration decaBDE (mg/kg range)
  - Store at room temperature between 15 °C and 30 °C
  - Avoid direct sunlight
  - Sample intake 0.1 to 0.5 g

- **Sediment**
  - Sieved <63µm, freeze dried, homogenation
  - Low concentration decaBDE (µg/kg range)
  - Store at room temperature between 15 °C and 30 °C
  - Avoid direct sunlight
  - Sample intake 5 -7 g

Test Materials (II)

- **GC-test solution** (toluene)
  - Dilution of a certified decaBDE standard solution in toluene purchased by Wellington Laboratories Inc. (Guelph, Ontario, Canada)
  - Undisclosed concentration (µg/kg range)
  - Store at refrigerator (4 °C)
  - Analysed directly by GC-MS, after addition of internal standard

Methodology

- Any appropriate analytical methodology is allowed to use
  - Use of $^{13}C_{12}$-labelled decaBDE as IS obligatory
  - Use of short GC column (<15 m) obligatory
    - DB-5
    - CP-Sil 8
  - Check GC-MS system that no deterioration of decaBDE in the injection system and the column occurs

- Replicates
  - 4 replicate analyses of the three samples
  - 4 independent blank replicates

Sample treatment

- Use your own method for the determination of DecaBDE
- Extraction
  - Use of organic solvent (e.g. toluene) or mixture of apolar and polar solvent recommended (e.g. hexane:acetone)
  - Soxhlet, pressurised liquid extraction, sonication, shaking
- Clean-up
  - GPC, silica gel, alumina, acid
  - Removal of sulphur from sediment sample
- Evaporation
  - Evaporation to dryness must be avoided unconditionally

Analysis

- High resolution gas chromatography (GC) combined with mass spectrometry (MS), either in electron impact ionisation (EI) or electron capture negative ionisation (ECNI) mode
- Use short (<15 m) and narrow (<0.25 mm) GC column
- Short injector residence time or cold injection
- Moderate injector temperature (e.g. 275°C)

Quantification

- Selected ion monitoring (SIM), peak area
- Concentration of decaBDE using isotope dilution technique
- Dust and sediment, results expressed on a dry weight basis (µg/kg)
- GC-test solution expressed in ng/ml

Reporting of results

- Dust, sediment and GC-test solution reported in Excel templates
  - "Results of dust, sediment, GC-test solution"
- Experimental conditions Word-document
  - "Experimental conditions"
- Provide typical chromatograms of dust, sediment and GC-test solution

Timeline

- Test materials, including a questionnaire on experimental conditions, an instruction protocol and a standard form for the reporting of results:
  21st of January

- Reporting: **15 March**