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

DecaBDE analysis

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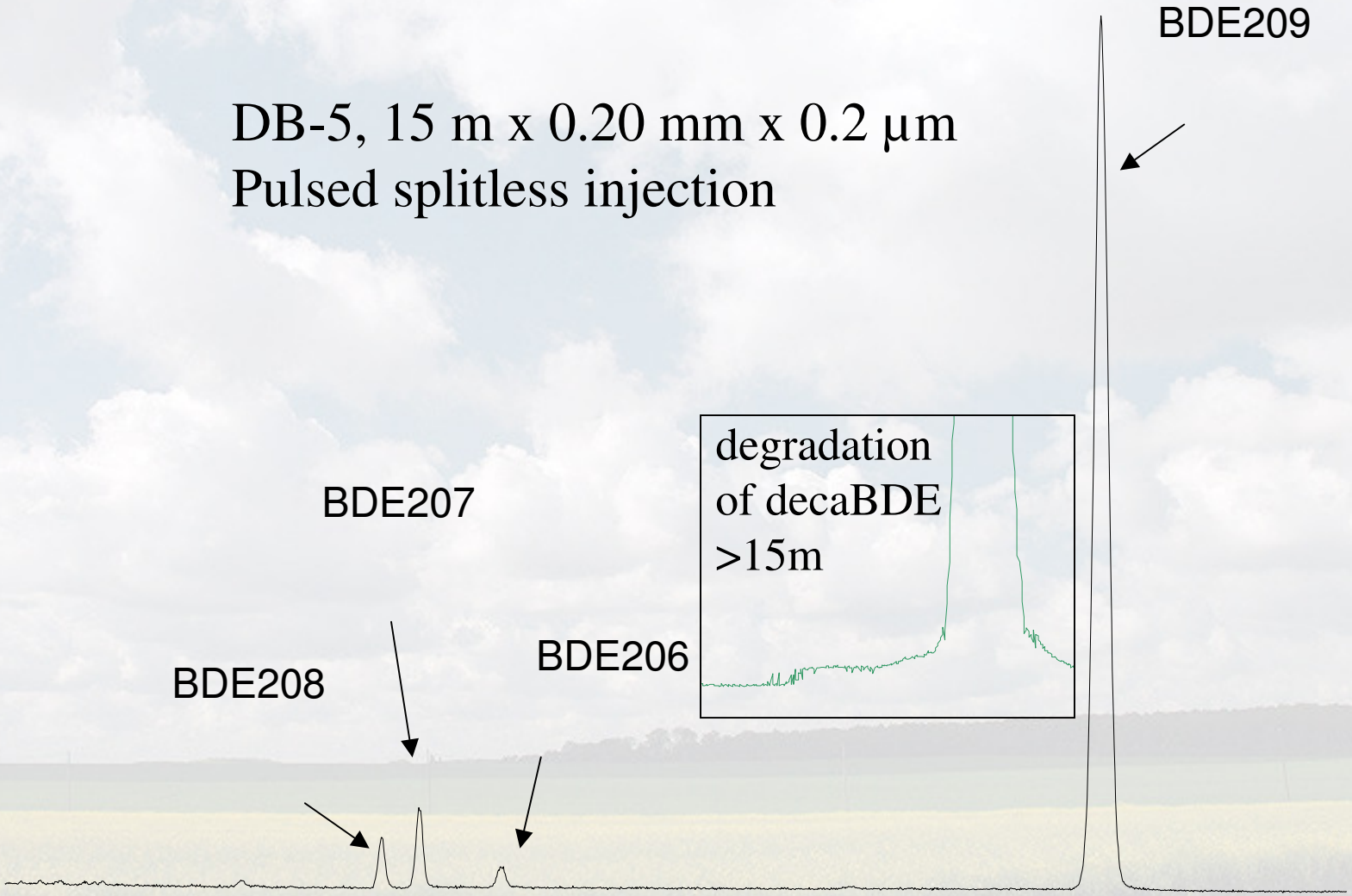
Sixth Framework Programme

Critical factors

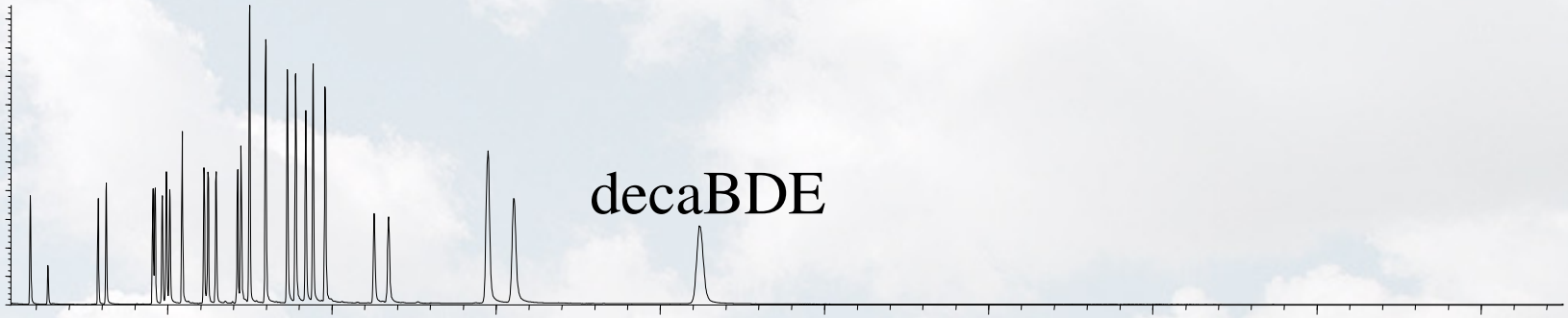
- GC-MS analysis
- Internal standards
- Photo-degradation
- Solubility
- Sources of contamination

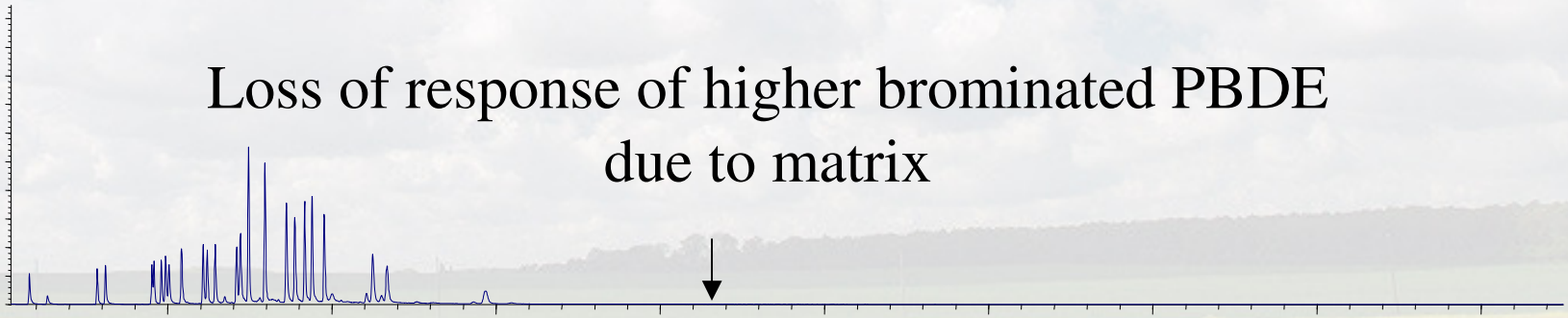
GC-MS (I)

DB-5, 15 m x 0.20 mm x 0.2 µm
Pulsed splitless injection

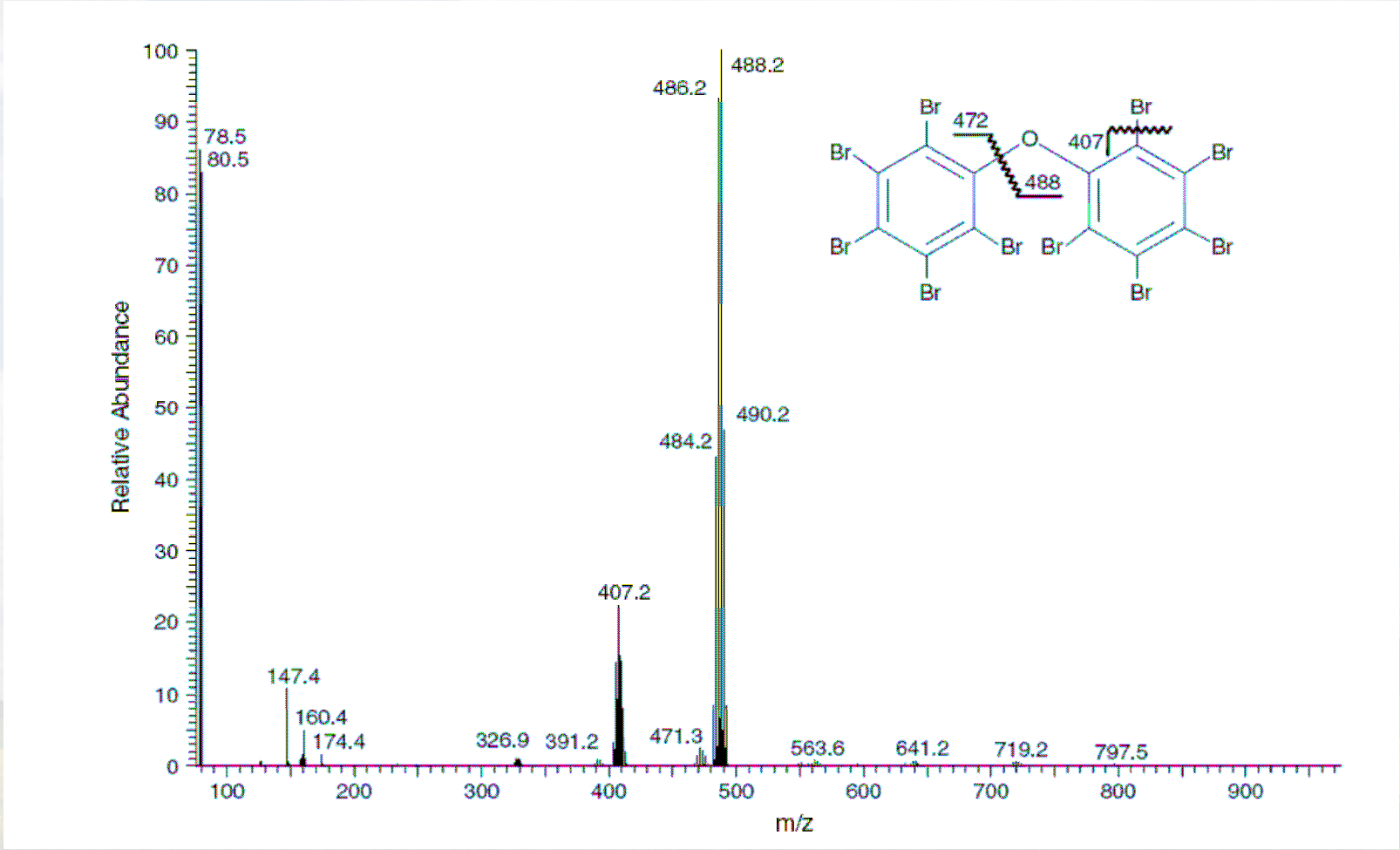


GC-MS (II)

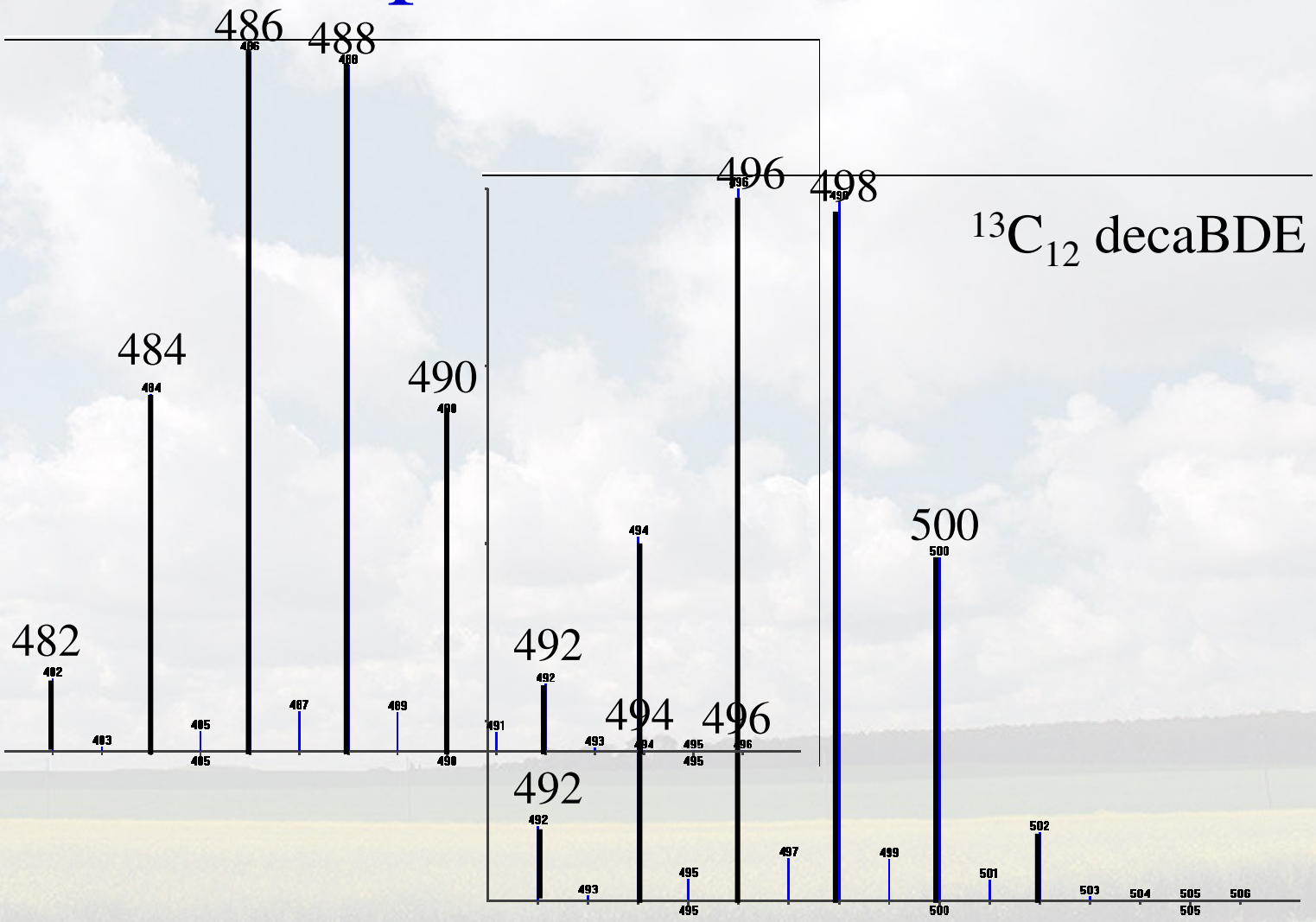

decaBDE


Loss of response of higher brominated PBDE
due to matrix

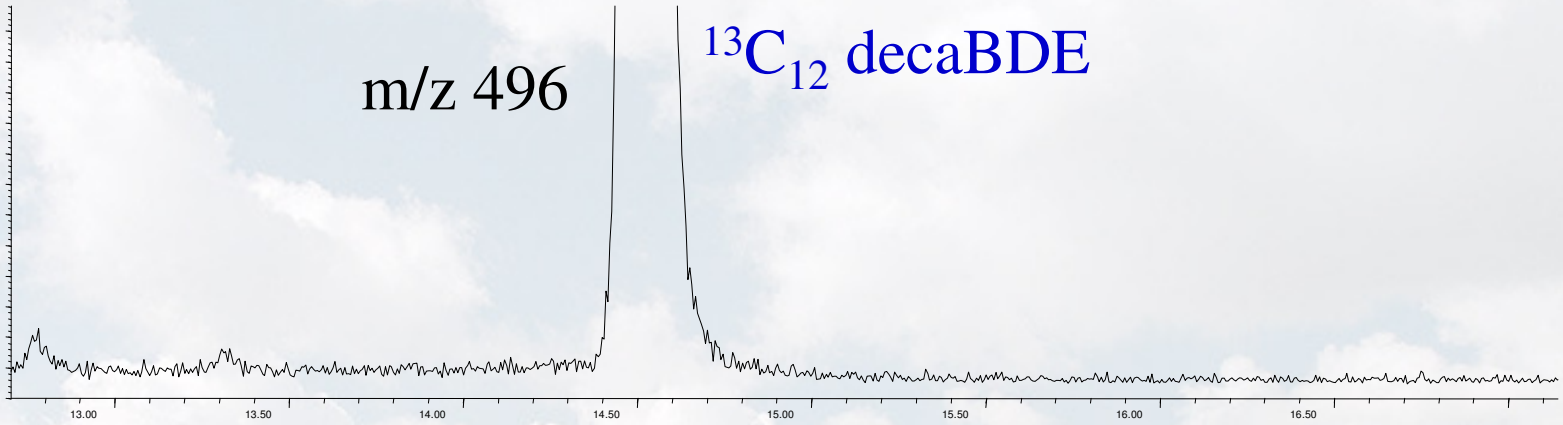
MS spectra (ECNI) decaBDE

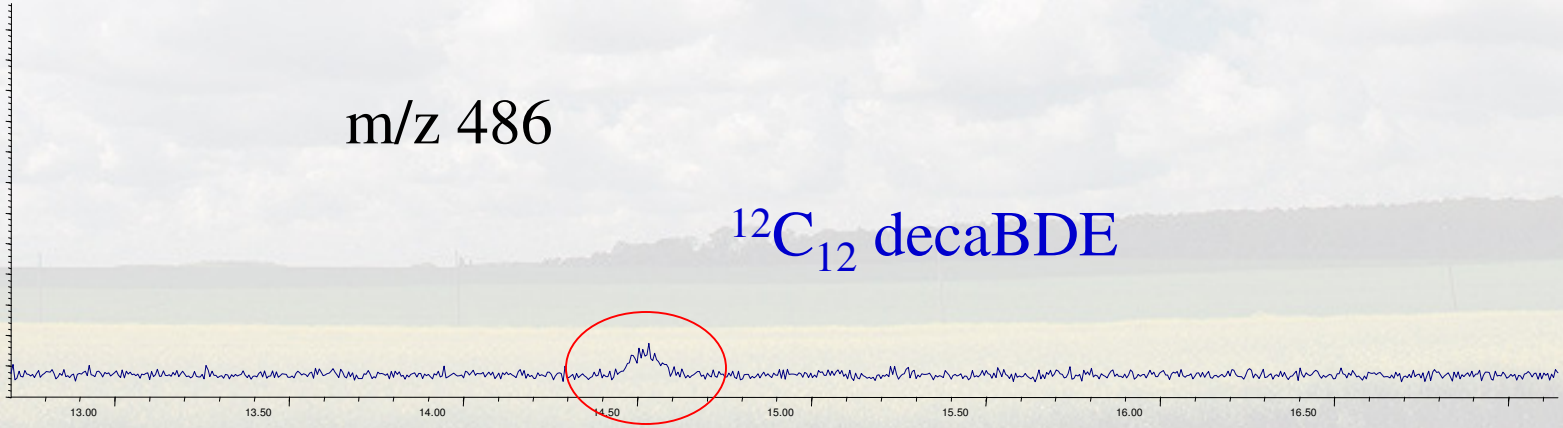


MS spectra decaBDE

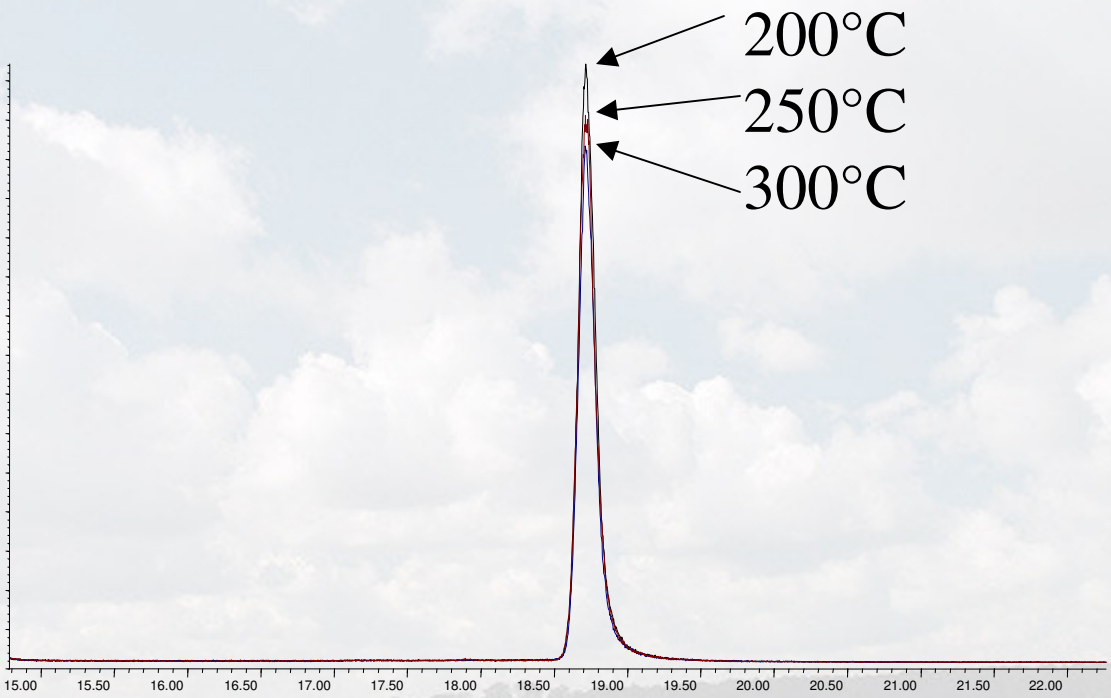


Impurity $^{13}C_{12}$ decaBDE

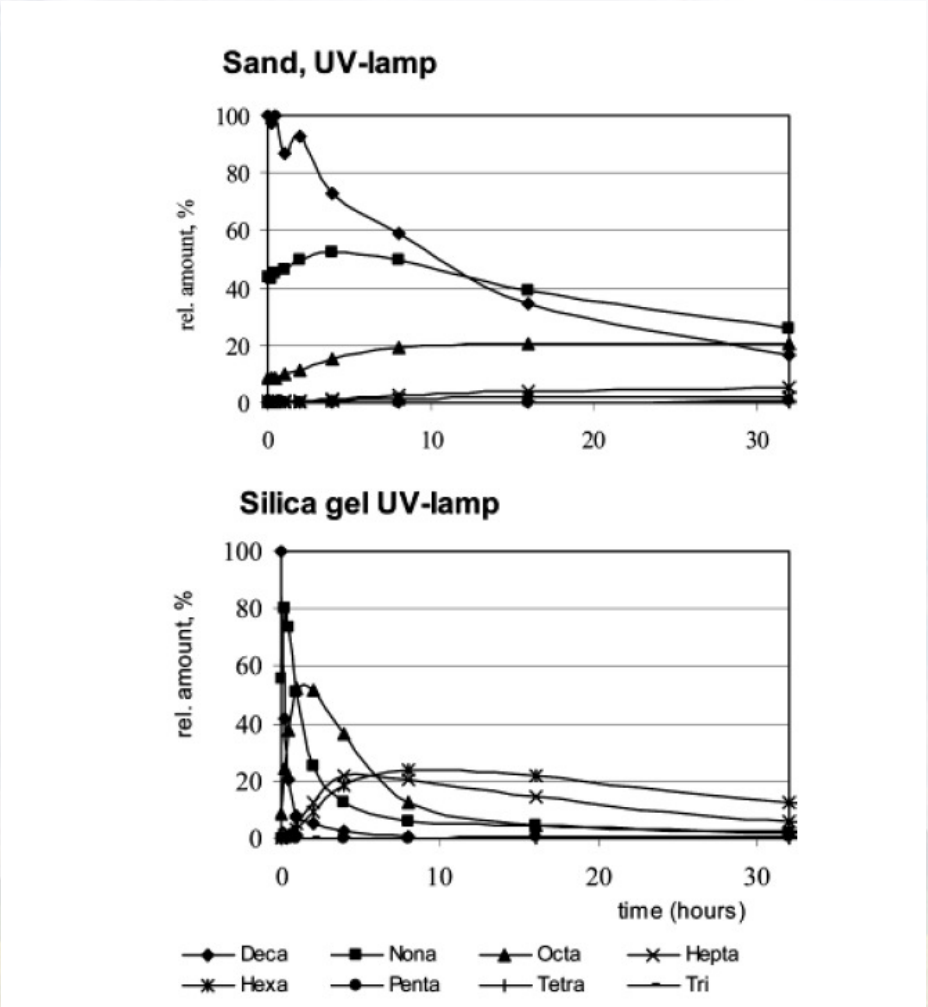




Ion source (ECNI) temperature



Photolytic debromination (I)



Söderström et al., Environ. Sci Technol., 2004, 38 (1), 127-132.

Photolytic debromination (II)

Half-lives (h) BDE209 on different matrices, indoors and outdoors

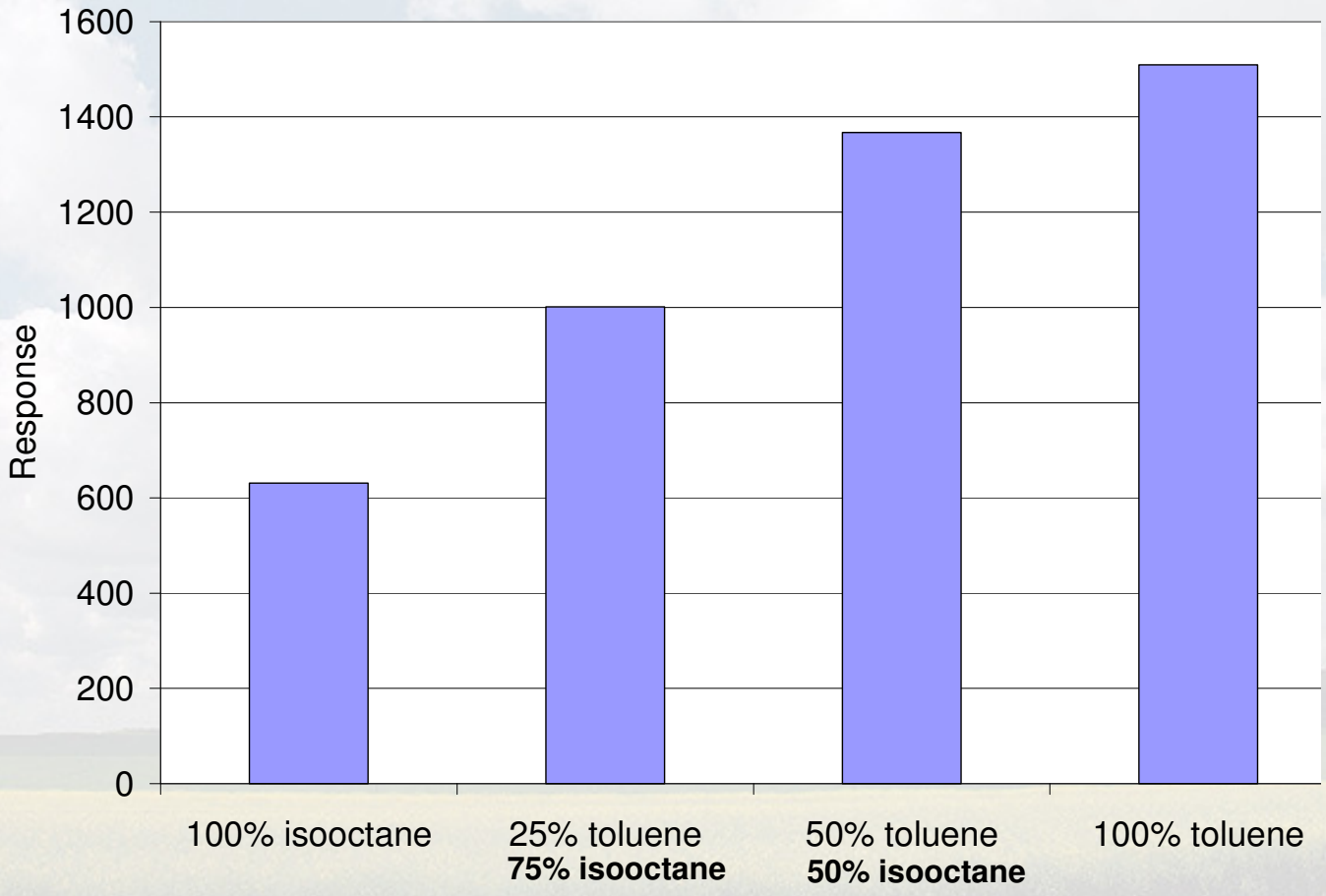
	Artificial UV-light (continuous)	Sunlight (discontinuous)	Sunlight (continuous)
Toluene	<0.25		
Silica gel	<0.25		
Sand	12	37	13
Sediment	40-60	80	30
Soil	150-200		

Söderström et al., Environ. Sci Technol., 2004, 38 (1), 127-132.

Photolytic debromination (III)

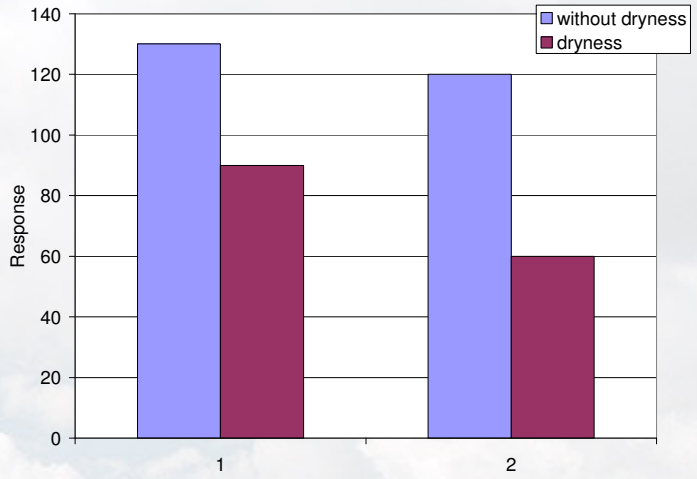
- Use of UV filters at laboratory windows and at fluorescent lightings
- Use of amber glassware or glass covered with aluminium foil

Solubility decaBDE organic solvent



Solubility decaBDE organic solvent

- Evaporation to dryness must be avoided unconditionally
- decaBDE adsorbs strongly to glassware and may not be re-dissolved completely
- Add toluene as keeper before concentrating extracts/solutions



Sources of contamination

- Laboratory infrastructure
  - Plastics, textiles, electronic equipment
- Other samples
- Reagents
- Glassware
- Atmospheric deposition
  - Dust (textile and carpet fibres, human skin, hair etc.)
- Packaging
  - EPS, PS chips, foams etc.
- GC injection system
  - rinse with toluene

Audit of glassware

Traditional v. Modern

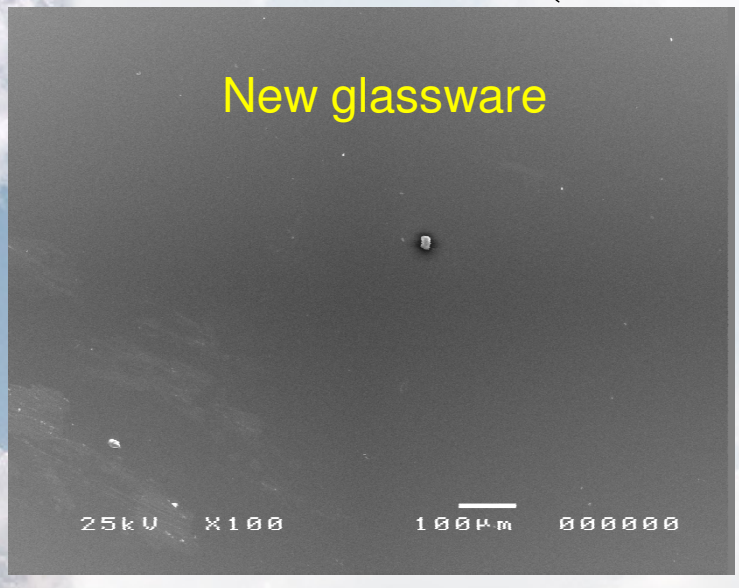
- Sample collection jar
- Soxhlet apparatus (4)
- Volumetric flask
- Evaporation stage (3)
- Column clean up (3)
- Evaporation stage (3)
- Fractionation stage (3)
- Evaporation stage (2)
- GC vial
- **20 pieces of glassware**

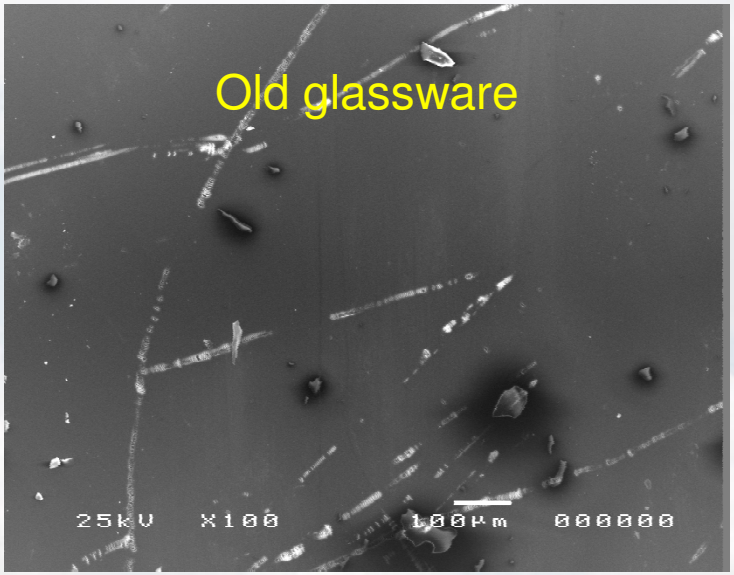
- Sample collection jar
- ASE (1)
- Volumetric flask
- Evaporation stage (3)
- GPC (1)
- Evaporation stage (3)
- Column clean up (1)
- Evaporation stage (3)
- GC vial
- **15 pieces of glassware**

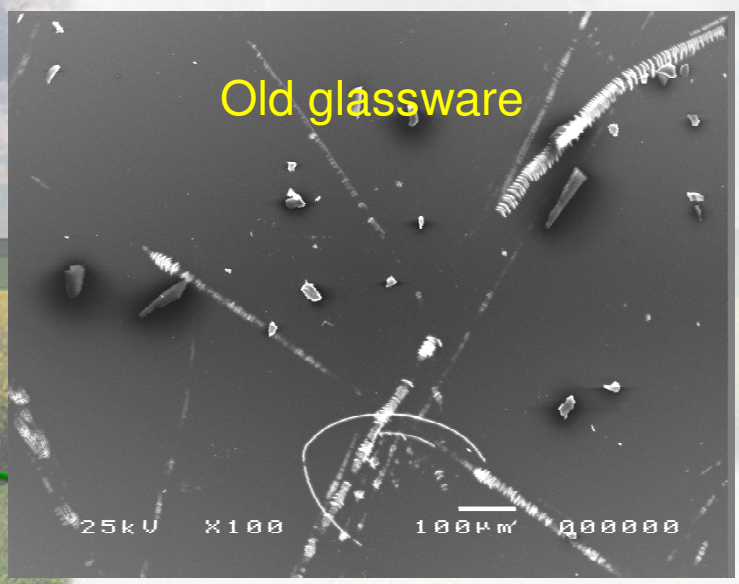
(Colin Allchin, CEFAS)

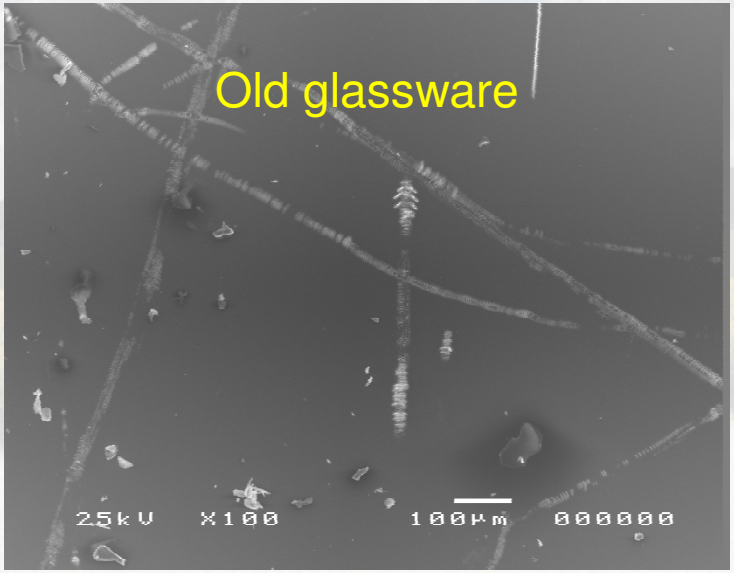
Glassware as a source of contamination

(Colin Allchin, CEFAS)









Glassware issues

- Cleaning glassware is difficult
- Cleaning old, scratched glassware is even more difficult
- When “blanking” glassware think about solvents and exposure time
- Keep clean glassware clean
- Segregate glassware dependant on sample type

Dust issues

- Levels of decaBDE in dust can be very high
  - 0.1 – 10 mg/kg
- Assume 1 mg/kg in dust of laboratory
  - -> 1 pg/µg dust
    - Final volume in GC vial 1 ml
    - Will result in a concentration of 1 pg/ml in GC vial
    - If 20 pieces of glass are used, each with 1 pg decaBDE in dust -> 20 pg/ml
      - LOD 100 pg/ml

* Harrad et al Environ. Sci. Technol. 2004, 38, 2345-2350
Stapleton et al Environ. Sci. Technol. 2005, 39, 925-931
Harrad et al Environ. Sci. Technol. 2006, 40, 4633-4638
Hazrati & Harrod Environ. Sci. Technol. 2006, 40, 7584-7589

Summary guidelines

- Use 13C decaBDE as internal standard
- Use <15 m GC column
- Use short injector residence times (pulsed splitless) or on-column injection
- Reduce sample exposure to glassware and reduce if possible number of pieces of glassware used
- If possible physically segregate sample by type and analysis in separate areas
- Reduce UV-light exposure (UV-filters or amber glassware)
- Reduce and avoid dust

Guidance documents decaBDE analysis

- The International Standard ISO/DIS 22032 “Water quality - Determination of selected polybrominated diphenylethers in sediment and sewage sludge - Method using extraction and gas chromatography/mass spectrometry”
- Bjorklund, Tollback, Ostman. 2003. Mass spectrometric characteristics of decabromodophenyl ether and the application of isotope dilution in the electron capture negative ionization mode for the analysis of polybrominated diphenyl ethers. J. Mass Spectrom. 38, 394-400
- De Boer, J, Allchin, C, Law, R, Zeger, B, Boon JP. 2001. Method for the analysis of polybrominated diphenylethers in sediments and biota. TrAC, Trend in Anal. Chem, 20, 591