



Bioaccessibility of PBDEs in indoor dusts

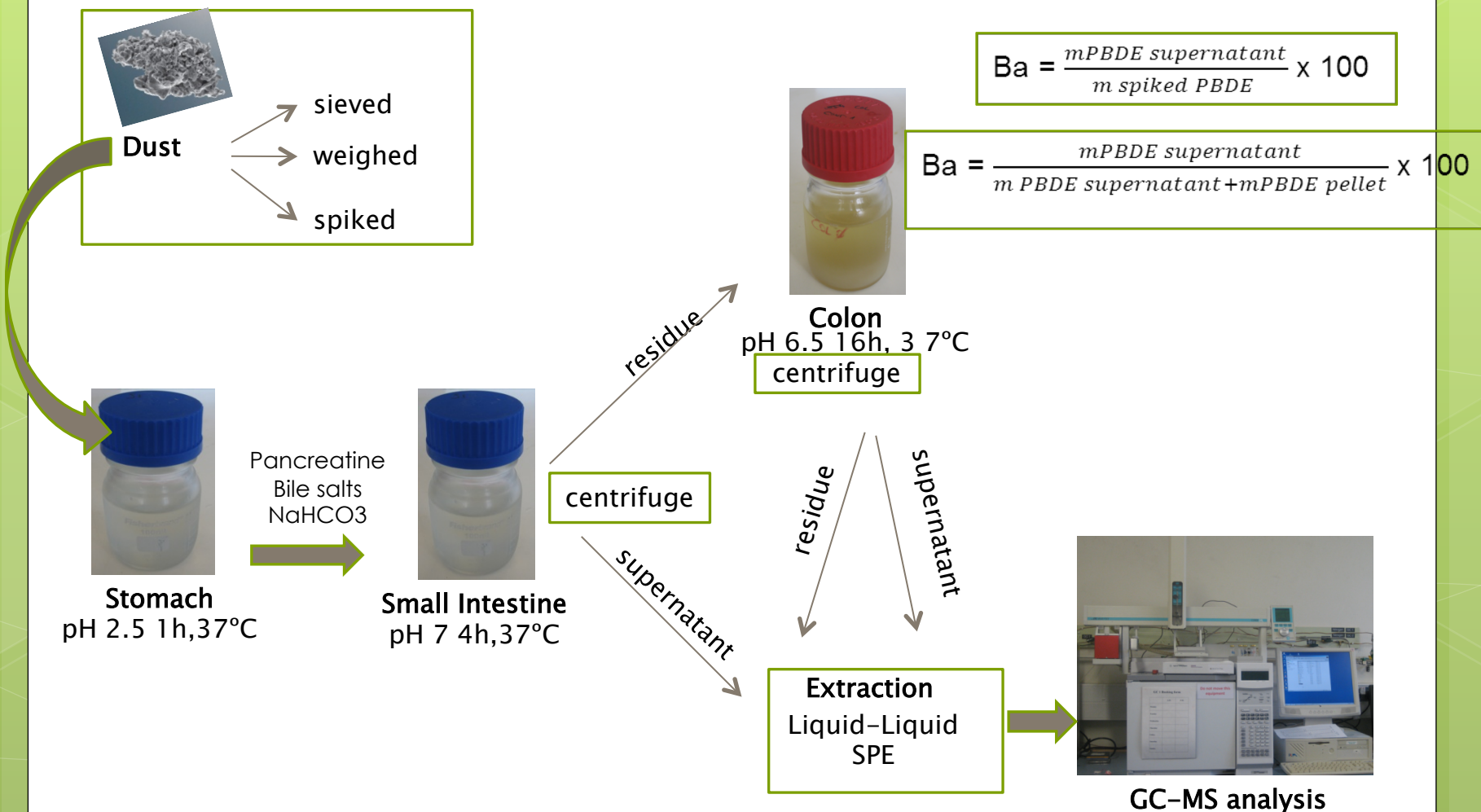
Sonia García Alcega



- The main exposure of PBDEs is via indoor dusts
- Daily intake:
 - children:** 100-400 mg dust / day
327-6540 ng PBDEs / day
 - adults:** 50 mg dust / day
13-213 ng PBDEs / day
- Objective:** determination of the bioaccessibility and bioavailability of PBDEs in indoor dusts.

Material and methods

1. CE-PBET to determine the bioaccessibility



Material and methods

2. Clean up:

- ✓ samples treated with concentrated sulphuric acid .
- ✓ SPE column filled with acidified silica gel.
 - Elution: 5 ml hex + 5 ml DCM

3. Evaporation: never until dryness

4. Resuspension: toluene, iso-octane.

Material and methods

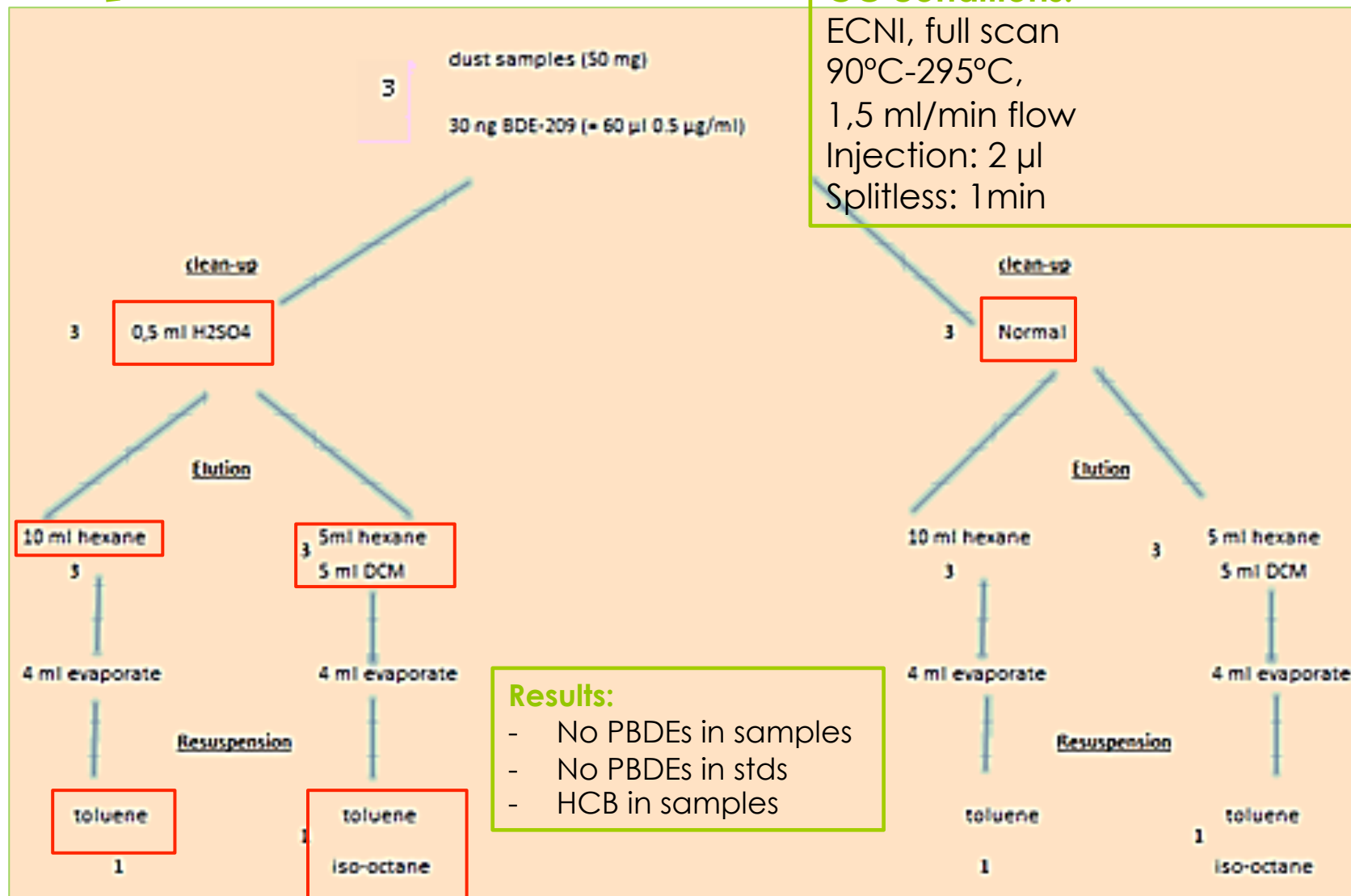
5. Analytical determination

- ✓ **GC-MS:** CI using methane as a reagent gas.
- ✓ **Injector:** Splitless during 1.5 min. T^a 280°C. Vol 2.5 µl.
- ✓ **Oven:** Starting temperature 90°C during 1 min. Increase to 300°C with 15°C / min ramp during 15 min.
- ✓ **Column:** DB-5 15m x 0.25 mm x 0.10 µm, carrier gas flow 1.5 ml/min.

Experiment

GC conditions:

ECNI, full scan
90°C-295°C,
1,5 ml/min flow
Injection: 2 µl
Splitless: 1 min



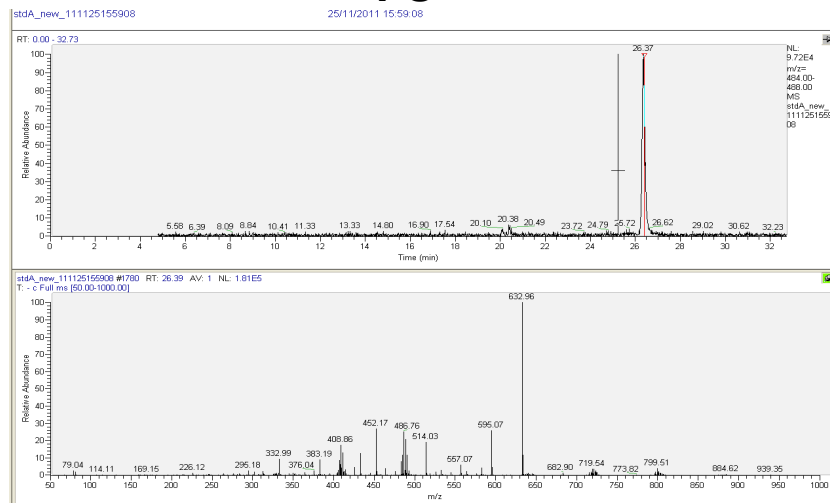
Results

Tuning...

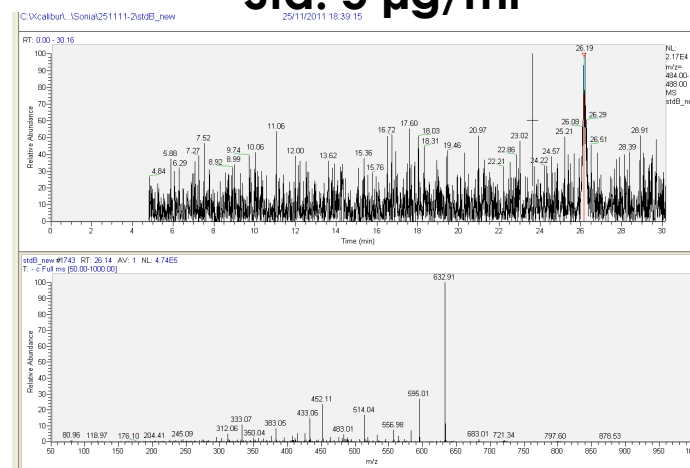
- **Reagent gas flow:** the best is to be less than 1.5 ml/min. It was at 0.3 and we have increased to 0.6 ml/min.
 - **Lenses:** 1 at 25, 2 at 100 and 3 at 25
 - **General:** AGC target value: 60, maximum ion value:25
 - **Trap offset:** 10
 - **Filament:** 70 electron energy, 10 electron sensitivity (V), 250 emission current (μ A).
- ➡ $\uparrow$ signals ions 400 and 500, $\downarrow$ signal ions 633, TIC: from e^4 to e^6
- ➡ Only stds $2\mu\text{g/ml}$, BDE-209 not detectable in dust samples spiked with $5\mu\text{g/ml}$.

Results

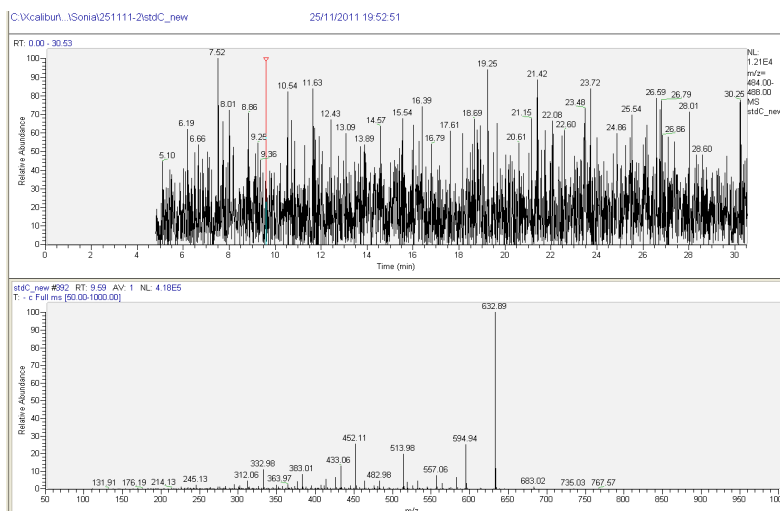
Std 10 µg/ml



Std. 5 µg/ml

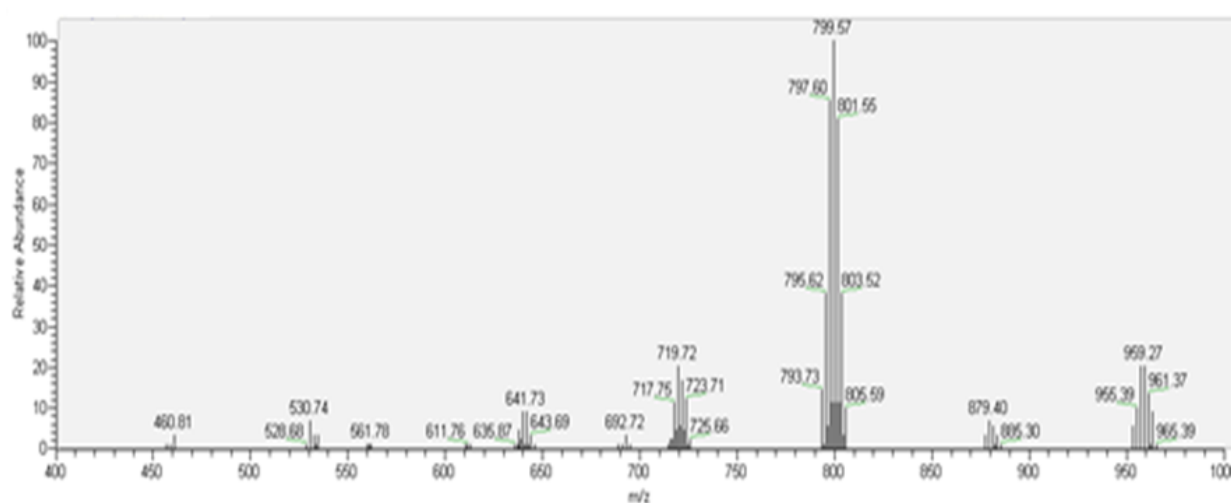


Rest of stds:
0,5 µg/ml
0,05 µg/ml



Results

After another tuning:



Visible concentrations in stds. until 0.5 $\mu\text{g/ml}$
 Samples???? Nothing but all < 0.5 $\mu\text{g/ml}$

Experiment

Dust extraction without gut system and without clean up steps

- 3 dust samples with 1ml BDE-209 0.05 $\mu\text{g}/\text{ml}$
- 3 dust samples without spiking solution
- Sonicate samples 10 min
- LLE with A:H + HCB as IS
- Filtration to remove the dust
- GC: 5 μl sample injection

Experiment

PBDEs extraction from stomach solution without dust

- 6 stomach samples spiked with 0.5 ml of 10 $\mu\text{g}/\text{ml}$ BDE-209
- 1h incubation
- LLE (A:H) + 10 min shaking
- 10 min ultrasounds
- Evaporation + 1ml mHex + 0.5 ml H_2SO_4
- 3 vials: concentrate + 1 ml isooctane
- 3 vials: columns with 1 gr ac. Silica
- Elution: 5ml nHex+5ml DCM
- Concentrate + 1ml iso-octane

Results

PBDE mix: BDE 17, 47, 66, 100, 153, 183 and 209

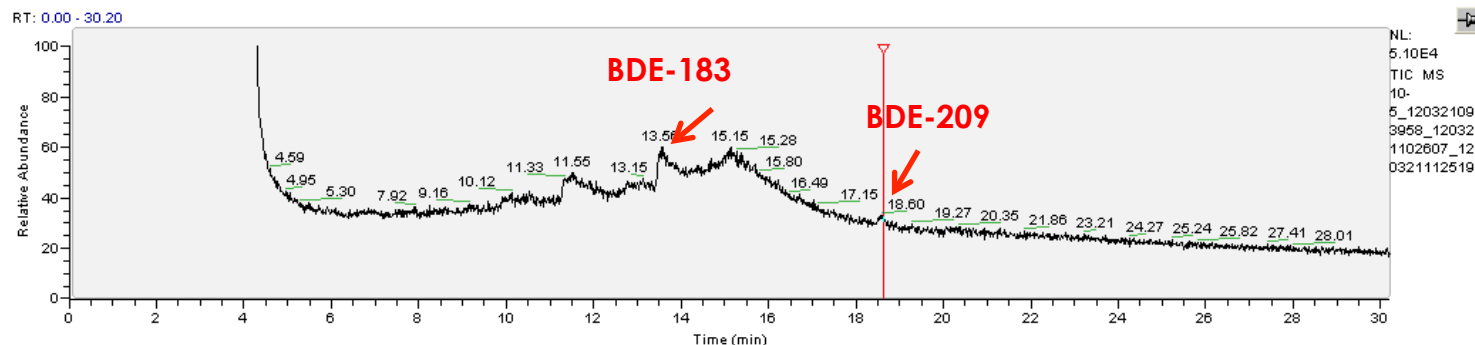
A) 10 μ g/ml BDE 209; 5 μ g/ml rest

B) 1 μ g/ml BDE 209; 0,5 μ g/ml rest

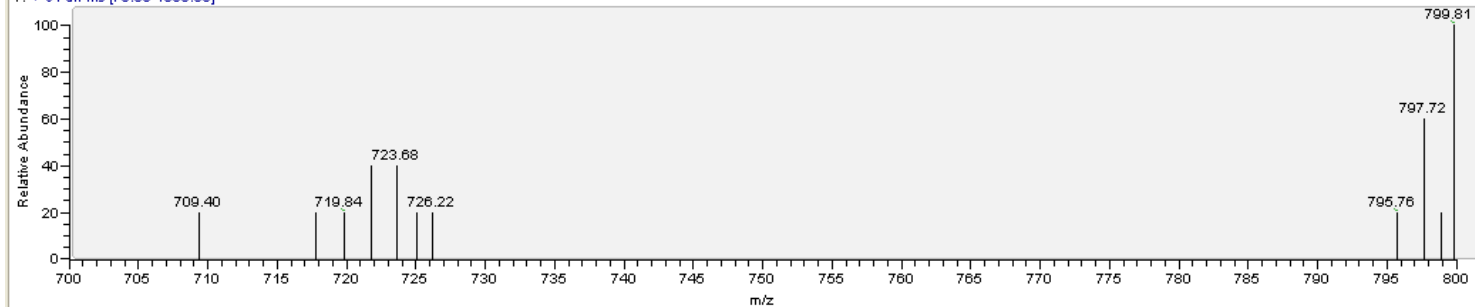
C) 0,1 μ g/ml BDE 209; 0,05 μ g/ml rest

10-5_120321093958_120321102607_120321...

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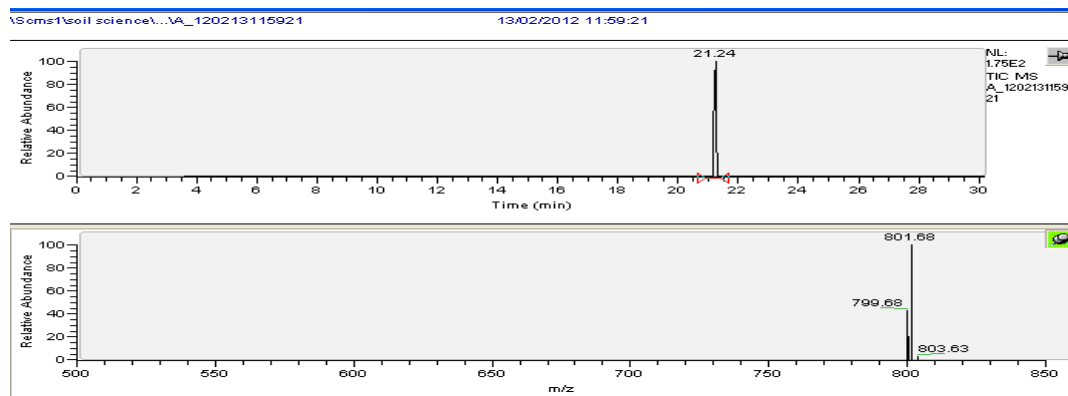


10-5_120321093958_120321102607_120321112519 #967 RT: 18.61 AV: 1 NL: 1.25E2
T: + c Full ms [70.00-1000.00]



future work

- Sort out the problems with the GC-MS
- Design a MRM method for all the PBDEs



BDE-209, 10 µg/ml

- Add 'sink' to CEPBET system – not silicone, possibly TECAM

The image is a classic Looney Tunes ending screen. It features a series of concentric red circles that create a tunnel-like effect, leading towards a dark blue center. The text "That's all Folks!" is written in a white, cursive script across the middle of the circles.

That's all Folks!