Selective analysis of Bromide via LC-MS/MS and comparison with a traditional GC-based method

H₃C—Br

DANGER

THIS UNIT IS UNDER FUMIGATION WITH (fumigant name) APPLIED ON

( date ) (Time and Description where applicable)

DO NOT ENTER

http://www.forstercontainer.ch/

http://www.containerhandbuch.de/chb_e/stra/index.html?/chb_e/stra/stra_03_08_00.html

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11th European Pesticide Residue Workshop
24th-27th May 2016, Limassol, Cyprus
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  - 2008/753/EC: non-approval of Methyl bromide
  - Maximum Residue Levels (MRL‘s) as **Bromide**, Reg. (EC) No. 149/2008
    and Reg. (EC) No. 839/2008: 
    **5 ppm** (berries) – **400 ppm** (spices)

2. Extraction of Bromide: QuPPe method

Weigh sample homogenate in 50 mL centrifuge tube

- Fresh fruits and vegetables (with high content of water): 10 g ± 0.1 g,
- Previously dehydrated dry fruit: 13.5 g ± 0.1 g (containing 5 g sample),
- Cereals and dried pulses (dried commodities): 5 g ± 0.05 g

Adjust water content of sample to 10 mL
e.g. Rye Flour: add 10 g water; Potato: add 2 g of water

Add 10 mL MeOH containing 1 % formic acid

LC-MS/MS analysis
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in-source fragmentation

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- strategies to decrease these interferences:
  - improve chromatographic separation
  - reduce matrix effects
  - more selective measurement (e.g. Differential Mobility Mass Spectrometry)
4. Improving the selectivity of the MS/MS measurement by modifying the Collision Energy (CE)

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<thead>
<tr>
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<td>Containing 2.2 ppm Bromide, approx. 400 ppm Phosphoric acid &amp; 5.4 ppm Phosphonic acid</td>
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Weigh sample homogenate in 100 mL Erlenmeyer flask

- Fresh fruits and vegetables (with high content of water): 5 g ± 0.05 g
- Cereals and dried pulses (dried commoditites): 5 g ± 0.05 g

Add 5 mL Propylene oxide solution (4 % in water, w/v) and 1 mL Sulphuric acid (3 mol/L) solution

Add 50 mL Ethyl acetate and 4 g Ammonium sulfate

GC-ECD analysis
5. Comparison with a traditional GC-based method

- **High fat content**
  - avocado
  - almond

- **High acid content**
  - grapefruit
  - lime

- **High sugar content**
  - rice
  - rye

- **Low water content**
  - apricot (dried)
  - raisin

- **High water content**
  - tomato
  - melon

*GC-ECD, n = 6
LC-MS/MS, n = 5*
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Thank you for your attention!

Questions to [EURL-SRM@CVUAS.BWL.de](mailto:EURL-SRM@CVUAS.BWL.de)