Chemical Contaminants & Residues in Food Community
Subgroup – ‘Environmental & Emerging Contaminants’

Tuesday, September 20th, 2016
4:30 p.m. to 6:00 p.m.

130th AOAC Annual Meeting & Exposition
Dallas, California
September 18 – 21, 2016
Chemical Contaminants & Residues in Food Community
Subgroup – ‘Environmental & Emerging Contaminants’

Agenda

■ 4:30 p.m. to 4:40 p.m.
   Introduction

■ 4:40 p.m. to 4:50 p.m.
   Polycyclic Aromatic Hydrocarbons (PAH) in seafood (AOAC 2014.08) – Progress for a Final Action Method

■ 4:55 p.m. to 5:10 p.m.
   Projects at CEN/TC 275/WG 13 – Update

■ 5:10 p.m. to 5:20 p.m.
   Update on Mepiquat as a new process-contaminant

■ 5:20 p.m. to 5:50 p.m.
   Multiresidue method for veterinary drugs in food – Discussion

■ 5:50 p.m. to 6:00 p.m.
   Wrap-up and closure
Polycyclic Aromatic Hydrocarbons (PAH) in Seafood (AOAC 2014.08) – Progress for a Final Action Method
Projects at CEN/TC 275/WG 13 – Update
prEN 16995:2016 – Vegetable oils and foodstuff on basis of vegetable oils – Determination on mineral saturated hydrocarbons (MOSH) and mineral oil aromatic hydrocarbons (MOAH) with on-line HPLC-GC-FID analysis

prEN 16987:2016 – Determination of acrylamide in coffee and coffee products by HPLC-MS/MS and GC-MS

Draft CEN/TS – Determination of acrylamide in potato-based products, cereal-based products and coffee by GC-MS
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Possible items for future standardization and/or mandating

- 3-MCPD
- 3-MCPD- and glycidyl-esters
- Bisphenol A (BPA) and analogues
- Illegal dyes
- Heterocyclic amines
- Methyl imidazoles
- Perchlorate
- Perfluorinated alkyl substances (PFAS)
- Brominated flame retardants (BFR)
- Metalloids (Cr, Ni, preferably to be dealt with in WG 10)
Update on Mepiquat as a New Process-contaminant
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Reducing sugar + 250°C → Piperidine

230 – 240°C

Arabica: 7.7 – 14.8 mg/g
Robusta: 5.2 – 15.5 mg/g

Trigonelline

Mepiquat

Nikolov & Yaylayan, 2010
Stadler et al., 2002
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<table>
<thead>
<tr>
<th>Methylation agent</th>
<th>‘Lysine’</th>
<th>Methylated piperidine derivatives</th>
</tr>
</thead>
<tbody>
<tr>
<td>Trigonelline</td>
<td>Piperidine</td>
<td>N-Methylpiperidine</td>
</tr>
<tr>
<td><img src="image" alt="Trigonelline" /></td>
<td>138.054956 Da</td>
<td><img src="image" alt="N-Methylpiperidine" /></td>
</tr>
<tr>
<td></td>
<td></td>
<td><img src="image" alt="Mepiquat" /></td>
</tr>
<tr>
<td></td>
<td></td>
<td><img src="image" alt="Mepiquat" /></td>
</tr>
</tbody>
</table>
Heat treatments (240°C, 5 min) in the presence of trigonelline + ...

... fructose ... fructose & lysine ... piperidine

**Piperidine**
[M+H]^+  
m/z 86.09643

**N-Methylpiperidine**
[M+H]^+  
m/z 100.11208

**Mepiquat**
M^+  
m/z 114.12773

**Trigonelline**
M^+  
m/z 138.05496
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Mepiquat content (μg/kg)

- Arabica, 1.1 (Columbia)
- Robusta, 7.1 (Vietnam)
- Robusta, 7.2 (Vietnam)

LOQ at 5 μg/kg

Mepiquat content (μg/kg)

- Arabica, 1.1 (Columbia)
- Robusta, 7.1 (Vietnam)
- Robusta, 7.2 (Vietnam)
### Chemical Contaminants & Residues in Food Community
**Subgroup – ‘Environmental & Emerging Contaminants’**

<table>
<thead>
<tr>
<th>Cereal</th>
<th>n</th>
<th>Betaine (mg/g d.m.)</th>
<th>Choline (mg/g d.m.)</th>
<th>Trigonelline (mg/kg d.m.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Winter wheat</td>
<td>130</td>
<td>1.59 ± 0.35</td>
<td>0.22 ± 0.02</td>
<td>3.18 ± 1.50</td>
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<tr>
<td>Spring wheat</td>
<td>20</td>
<td>1.62 ± 0.32</td>
<td>0.23 ± 0.02</td>
<td>2.56 ± 1.54</td>
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<tr>
<td>Durum wheat</td>
<td>10</td>
<td>2.32 ± 0.41</td>
<td>0.27 ± 0.02</td>
<td>5.12 ± 2.46</td>
</tr>
<tr>
<td>Spelt</td>
<td>5</td>
<td>2.31 ± 0.33</td>
<td>0.21 ± 0.01</td>
<td>2.32 ± 0.94</td>
</tr>
<tr>
<td>Einkorn</td>
<td>5</td>
<td>2.57 ± 0.25</td>
<td>0.26 ± 0.04</td>
<td>1.08 ± 0.63</td>
</tr>
<tr>
<td>Emmer</td>
<td>5</td>
<td>2.05 ± 0.34</td>
<td>0.22 ± 0.02</td>
<td>2.21 ± 3.31</td>
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<tr>
<td>Rye</td>
<td>10</td>
<td>2.27 ± 0.48</td>
<td>0.26 ± 0.02</td>
<td>31.13 ± 12.5</td>
</tr>
<tr>
<td>Barley</td>
<td>10</td>
<td>1.02 ± 0.23</td>
<td>0.32 ± 0.03</td>
<td>0.25 ± 0.24</td>
</tr>
<tr>
<td>Oat</td>
<td>5</td>
<td>0.43 ± 0.09</td>
<td>0.17 ± 0.01</td>
<td>111.7 ± 10.1</td>
</tr>
</tbody>
</table>

_Corol et al., 2012, J. Agric. Food Chem., 60, 5471-5481_

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**Betaine (glycine betaine)**
Monoisotopic mass: 118.086258 Da

**Choline**
Monoisotopic mass: 104.106987 Da
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Mepiquat content (µg/kg)

Roasting Time (min)

- Barley
- Rye

Roasted Barley (32%)
Roasted Rye (19%)
Roasted Chicory (18%)
Malt (31%)
Finished Product

97 µg/kg
150 µg/kg
< LOQ_{15}
180 µg/kg
205 µg/kg


Multiresidue Method for Veterinary Drugs in Food

– Discussion –
New Topics to be Adressed?
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- Methods for possible officialization
  - Bisphenol A
  - Flame retardants
  - Packaging-related contaminants (to be defined)
  - Phthalates
  - Quaternary ammoniums
  - Nitrogenous compounds

- Validation procedure (guidelines?) for fingerprinting-based methods

- Guidelines for untargeted analysis aimed at identifying unknowns

- Platform for suitable information in the case of a response to crisis
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Thierry DELATOUR

Group Chemical Contaminants
Competence Pillar Analytical Sciences
Nestlé Research Centre
Vers-chez-les-Blanc
1000 Lausanne 26, Switzerland

Phone: +41.21.785.9220
E-mail: thierry.delatour@rdls.nestle.com