Tracking chlorate residue levels from milk to powder

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Chlorate Scenario

- Ireland supplies 15% of the global infant milk formula

- **CONCERN:** high levels of chlorate
  - Health and safety
  - Quality requirements from the international market

- What we know:
  - Factors that affect chlorate levels
  - How to control those factors

- Questions that remain:
  - Combined effect of processing conditions and operations
  - Influence of individual milk suppliers on levels in bulk milk
From farms to milk powder

Dairy suppliers
Spring: 67
Winter: 150

11 collection tankers

Whole Milk Silo
300,000 L

Pasteurisation and Cream separation

Evaporation and Spray-drying

Skim Milk Silo

Skim Milk Powder

9 x 25 kg bags (start, middle and end of the run)
**Sampling Details**

- Samples were collected by processing plant personnel
- Sample collection from farms on the same day of milk collection
- Frozen samples were transported in cooling boxes and delivered within 6 h after collection

<table>
<thead>
<tr>
<th></th>
<th>Spring</th>
<th>Winter</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mean Volume in Bulk Tanks</td>
<td>298 to 21,572 L</td>
<td>114 to 10,525 L</td>
</tr>
<tr>
<td>Mean Storage time on-farm</td>
<td>44 h</td>
<td>70 h</td>
</tr>
<tr>
<td>Mean N farms in tankers</td>
<td>6</td>
<td>14</td>
</tr>
</tbody>
</table>

- Milk powder sampling (9 bags):

- Chlorates were quantified by High Performance Liquid Chromatography and Tandem Mass Spectroscopy
From Farms to Whole Milk Silo

- Chlorate concentration (ppb)

<table>
<thead>
<tr>
<th>Period</th>
<th>Bulk tanks (n=67/ n=150)</th>
<th>Tankers (n=11)</th>
<th>Whole Milk Silo (n=2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spring</td>
<td>2.1 (14 farms)</td>
<td>2.0 (6 tankers)</td>
<td>1.0</td>
</tr>
<tr>
<td>Winter</td>
<td>41.0 (6 tankers)</td>
<td></td>
<td>2.5</td>
</tr>
</tbody>
</table>

Detection limit chlorate: 1 ppb; between parenthesis n of samples chlorate was detected.

1. Farm samples (bulk tanks)
   - Spring: none over limit (< 10 ppb)
   - Chlorate not measured in 150 farms samples
   - Winter (same 67 farms): 5 over the limit; mean 37 ppb; detected in 32 farms)

2. Levels are higher during winter months
   - Changes in cleaning practices
   - Lower volume of milk supplied

2. Tankers
   - Spring: none over limit
   - Winter: 5 over limit
From Farms to Whole Milk Silo

- Chlorate concentration (ppb)

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Detection limit chlorate: 1 ppb; Between parenthesis n of samples chlorate was detected.

1. Contribution from tankers cleaning
   - Chlorate not detected in the majority of bulk tank samples that supplied milk to the 6 tankers during winter

2. Dilution effect
   - High volume of milk with low chlorate levels was supplied to the factory in both seasons
   - Dilution in the Whole Milk Silo in both seasons (< 10 ppb – default limit)
From Whole Milk Silo to Powder

- Chlorate concentration (ppb)

<table>
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<tr>
<th>Period</th>
<th>Whole Milk Silo (n=2)</th>
<th>Skim Milk Silo (n=2)</th>
<th>Skim Milk Powder (n=9)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Spring</td>
<td>1.0</td>
<td>1.0</td>
<td>57.0</td>
</tr>
<tr>
<td>Winter</td>
<td>2.5</td>
<td>2.5</td>
<td>126.3</td>
</tr>
</tbody>
</table>

1. No difference from Whole to Skim Milk Silo
   - Chlorate levels were not affected by cream separation or pasteurisation
   - No effect from CIP

2. High levels in winter powder
   - High levels in winter raw milk (limit for powder 50 or 100 ppb)

3. 50 times increase from Skim Milk Silo to Powder
   - Evaporation and Spray Drying – 10 fold increase (based on results reported in other studies)
   - Possible contribution from CIP/ Water (>40 µg/ L)
Inside the Spray Drying

- **Chlorate levels throughout operation:**
  - Start: 63 ppb
  - Middle: 61 ppb
  - End: 47 ppb

- **Spray dryer CIP or chlorate levels in water**
  - First skim milk to enter spray dryer got in contact with most of the remaining chlorate on the equipment interior surface
Conclusion

Attention during winter
- Long storage of cleaning products
- Lower volume of milk

Low chlorate levels in raw milk
- Low levels in bulk milk
- However! Need to correct practices on farms with issues

Drying milk
- Large contribution
- However! Need to monitor levels in water

What could be the next step?
- Removal of chlorine detergents from CIP
- Use of chlorine gas to sanitise water
Monitoring residue concentrations in milk from farm and throughout a milk powder manufacturing process

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Abstract

The experiments reported in this research paper aimed to investigate differences in the levels of chlorate (CHLO), perchlorate (PCHLO), trichloromethane (TCM) and iodine residues in bulk tank (BT) milk produced at different milk production periods, and to monitor those levels throughout a skim milk powder (SMP) production chain (BTs, collection tankers [CTs], whole milk silo [WMS] and skim milk silo [SMS]). Chlorate, PCHLO and iodine were measured in SMP, while TCM was measured in the milk cream. The CHLO, TCM and iodine levels in the mid-lactation milk stored in the WMS were lower than legislative and industrial specifications (0.0100 mg/kg, 0.0015 mg/kg and 150 μg/l, respectively). However, in late-lactation, these levels were numerically higher than the mid-lactation levels and specifications. Trichloromethane accumulated in the cream portion after separation. Perchlorate was not detected in any of the samples. Regarding iodine, the levels in mid-lacta-
Acknowledgements

- Farmers
- Tanker drivers
- Milk quality advisors
- Processing plant personnel
- Martin Danaher – Chlorates quantification
Thank you!