Determination of calcium by EDX

a fast method for the determination of mechanically recovered meat (MRM)
What is mechanically recovered meat?

EC Regulation No 853/2004 (Annex I):
“...the product obtained by removing meat from flesh-bearing bones after boning or from poultry carcasses, using mechanical means resulting in the loss or modification of the muscle fiber structure”
Production of mechanically recovered meat

Various methods for separating bones from MRM

Drum-belt separator

Endless screw separator

Images from EFSA Journal, 2013, 11.
Bone fragments

- Mechanically recovered meat contains small bone fragments

Analytical implications

• Calcium can be used as marker element for MRM:
  • Ca content of bone ash is constant (36.3 – 36.9 % Ca)[1]
  • Low Ca concentration in meat

• Samples will be heterogeneous
  • Homogenization of a larger sample mass prior subsampling

Regulatory requirements

- EC Regulation No 2047/2005
  - Ca in MRM “shall not exceed 0,1 % of fresh product”

- Austrian food codex, Chapter B14
  - MRM mustn’t be used for the production of sausages
  - Ca content < 200 mg/kg if bone particles are present
“Traditional” way of analyzing Ca

Sample homogenization

Digestion in acid (HNO₃)

Ca quantification
Alternative approach: EDX

Sample homogenization

Ca quantification
Determination of Ca

- **Samples**
  - 3 different types of sausages
  - Liver meat paste
  - Dog food

- **Homogenization**
  - Büchi Mixer (500 g batches)
  - Retsch Cryo Mill for homogeneity studies
EDX: Ca determination

- Gravimetrically prepared standards in the range of 400 – 1600 mg kg\(^{-1}\)
- Standard conditions for Ca in the Shimadzu EDX 8000
- Collimator: 10 mm
- Integration time: 100 s
EDX: Ca calibration

![Graph showing Ca calibration](image-url)
EDX: Ca determination

- EDX limit of quantification (LOQ): 170 mg kg\(^{-1}\)

- Reference digestions and Ca-quantification of several samples (sausages and pet food) by ICP-OES
## Comparison EDX / classical approach

<table>
<thead>
<tr>
<th>Sample</th>
<th>Reference value (ICP-OES)</th>
<th>EDX</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sausage (Krakauer)</td>
<td>42 ± 1 mg/kg</td>
<td>&lt; LOQ (160 mg/kg)</td>
</tr>
<tr>
<td>Sausage (Huber)</td>
<td>52 ± 2 mg/kg</td>
<td>&lt; LOQ (160 mg/kg)</td>
</tr>
<tr>
<td>Sausage (Tann)</td>
<td>48 ± 1 mg/kg</td>
<td>&lt; LOQ (160 mg/kg)</td>
</tr>
<tr>
<td>Liver meat paste (Inzersdorfer)</td>
<td>83 ± 2 mg/kg</td>
<td>&lt; LOQ (160 mg/kg)</td>
</tr>
<tr>
<td>Dog food (Pedigree)</td>
<td>2610 ± 80 mg/kg</td>
<td>2690 ± 70 mg/kg</td>
</tr>
</tbody>
</table>
EDX: Spike recovery

Spike concentration: 1000 mg kg\(^{-1}\) Ca
Conclusion: Spike recovery

- Spike recoveries in general acceptable – potential problems with the homogenization of the Ca-standard into the sample
  - relatively high RSD’s for EDX
  - some samples were particularly rich in fat

- Sample heterogeneity was troublesome in ICP-OES
  - although some sausages “look” homogeneous, they aren’t!
  - Production of the sausages can have an effect on the distribution of the raw materials
Time required for an analysis

“Traditional” approach:
Acid digestion and ICP-OES

- digestion of 16 samples → 60 min
- ICP-OES: 60 min (including calibration)

→ about 7-8 min per sample

EDX

→ less than 2 min per sample
Conclusion

• EDX provides a fast and reliable method for screening meat products

• LOQ sufficient for Austrian food codex (and similar regulations throughout the EU) → identification of products potentially containing MSM

• Requirements of EC Regulation No 2047/2005 are easily fulfilled in terms of LOQ
Acknowledgements

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Thank you for your attention!