Improvement of ESR spectrometer sensitivity and its implication on detection of irradiated food products and dosimetry

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Aerial, Technology Resource Centre

→ Technical Assistance

→ Expert Advice

→ Applied Research

→ Training

Strasbourg - France

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Since 1985, Aérial’s goal is to anticipate tomorrow’s industrial problems to assist all companies, particularly SME’s in all their needs on:

- Radiation processing
- Food Processing
- Freeze-Drying
Outline

• Introduction/Context
• Improvements in ESR benchtop spectrometers
• Implication on dosimetry applications
• Implication on detection of irradiated food
• Conclusion – Further developments
Research & Development and Technical Assistance for the Radiation Processing Industry:

Quality assurance for Radiation Processing:
- Dose measurements (COFRAC accredited laboratory-SSDL)
- IQ/OQ/PQ of irradiation plants
- Expertise/Training

Detection of irradiated food products:
- Accredited laboratory by french ministry of economy, finance and industry
ESR Spectroscopy / Dosimetry

\[ h\nu = \beta \cdot g \cdot B \]

→ Concentration of free radicals
→ Identification of free radical
Introduction/Context

ESR Spectroscopy / Dosimetry

• ISO / ASTM51607 – 13 : Standard Practice for Use of the Alanine-EPR Dosimetry System
  ➔ Amplitude of ESR Signal
• EN 1786:1996 : Detection of irradiated food containing bone - Method by ESR spectroscopy
• EN 1787:2000 : Detection of irradiated food containing cellulose. Method by ESR spectroscopy
• EN 13708:2001 : Detection of irradiated food containing crystalline sugar by ESR spectroscopy
  ➔ Presence/absence of specific ESR Signal
ESR Spectroscopy / Dosimetry

**Need**
- Best reproducibility
- Highest signal/noise ratio

**How**
- Improve spectrometer sensitivity
- Reduce noise signal
- Improve signal treatment
Improvements in ESR spectrometers

Benchtop Spectrometers …

MS100, 200, 300, 400, Magnettech by Freiberg Instruments

1991

Adani, Active Spectrum, …

MS5000, Magnettech by Freiberg Instruments

2014
Improvements in ESR spectrometers

**MS100**
- Original version by Magnettech

**MS200**
- Microwave amplifier 3 time more sensitive

**MS300**
- Adjustment electronics precision and thus better reproducibility
- Measurement resonator higher Q-factor of cavity and flat baseline

**MS400**
- Microwave bridge
- Transistor oscillator less heat and long term stability
- Cavity critically coupled for better reproducibility

**MS5000**
- Phase noise reduction
- Digital field control
- Digital Signal processing
- Very compact housing

1991

2014

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Implication on dosimetry applications

Bare Alanine pellets
(4 mm diam, 2.3 mm thick, 36 mg)

Packaged Alanine pellets

Traceability during irradiation/measurement
Protection of pellet
Precise dose location

→ dosimeter which is readout with its packaging

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Implication on dosimetry applications

Dosimeter

Spectrometer

ESR signal

Calibration curve → Dose

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## Implication on dosimetry applications

<table>
<thead>
<tr>
<th>Applications</th>
<th>Dose range</th>
</tr>
</thead>
<tbody>
<tr>
<td>SIT</td>
<td>few 10 Gy</td>
</tr>
<tr>
<td>Fruits/vegetables</td>
<td>few 100 Gy</td>
</tr>
<tr>
<td>Food decontamination</td>
<td>few kGy</td>
</tr>
<tr>
<td>Food Sterilization</td>
<td>few 10 kGy</td>
</tr>
</tbody>
</table>

→ ESR/Alanine dosimetry system applicable to all applications
Implication on dosimetry applications

DOE approach for the choice of ESR parameters

- Sweep time
- Number of scans (reading time)
- Modulation
- MW power
- Signal analysis (Central Hpp, Integrals, Sum of Hpps, ...)
Implication on dosimetry applications

Alanine Signal Comparison

MS200
MS400
MS5000

At Room Temperature

Comparaison pour 2Gy

Amplitude arbitraire

Champ magnétique en mT

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### Implication on dosimetry applications

<table>
<thead>
<tr>
<th>Dose Gy</th>
<th>Reproducibility* % MS200</th>
<th>Reproducibility * % MS 400</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>-</td>
<td>4,8</td>
</tr>
<tr>
<td>2</td>
<td>8,9</td>
<td>5,3</td>
</tr>
<tr>
<td>5</td>
<td>4,2</td>
<td>2,2</td>
</tr>
<tr>
<td>10</td>
<td>1,9</td>
<td>0,8</td>
</tr>
</tbody>
</table>

* 20 dosimeters irradiated at same dose
Implication on dosimetry applications

HPP = f(dose)

y = 598.10264x + 260.01006
R² = 0.99993

MS400: Overall uncertainty < 3.4 % (at 95%) for doses > 10 Gy and readout time of less than 30 seconds
Implication on detection of irradiated food

Chicken bones

Walnuts

86 Gy to 12 kGy
X Rays (100 kV)
Electron Beam 2,2 MeV
Measurement D0 at room Temperature

≈ 40 mg
MS200, MS400, MS5000

≈ 30 mg
MS200, MS400
Implication on detection of irradiated food

\[ h\nu = \beta \cdot g \cdot B \]

\[ g \text{ (signal)} = 71,448 \cdot \nu / B \]

\[ \nu \text{: MW Frequency (GHz)} \]
\[ B \text{: Magnetic field (mT)} \]

EN 1786

\[ g_{\text{sym}} = 2.005 \pm 0.001 \]
\[ g_1 = 2.002 \pm 0.001 \]
\[ g_2 = 1.998 \pm 0.001 \]
Implication on detection of irradiated food

MS400

258 Gy
86 Gy
172 Gy
Implication on detection of irradiated food

Detection limit depends on spectrometer, bone crystallinity, irradiation and storage conditions, ...
Implication on detection of irradiated food

EN 1787

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Implication on detection of irradiated food

Detection limit depends on spectrometer, moisture content, irradiation and storage conditions, ...

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Implication on detection of irradiated food

86 Gy

High spectrometer sensitivity is not enough! ...
Conclusion

Further developments

• ESR Spectroscopy for dosimetry and detection applications
  → Need for high sensitivity/reproducibility

• Improvements in Spectrometer technology
  → Lower dose measurements with better reproducibility
  → Easier analysis of radiation specific signals

• Further improvement
  → Low temperature measurement (LN, Pelletier, …)
  → Dielectric cavity to enhance fill factor
  → Insert (sample holder) to concentrate Microwave on sample
  → Software to improve signal treatment/analysis (autocorrelation)

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