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CRT CENTRE DE RESSOURCES TECHNOLOGIQUES

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INSTITUT CARNOT MICA

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

→ Training

→ Expert Advice

→ Applied Research

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Aérial - La bonne dose d'innovation

www.aerial-crt.com

Since 1985, Aérial's goal is to anticipate tomorow'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

Aérial

Research & Development and Technical Assistance for the Radiation Processing Industry :

Quality assurance for Radiation Processing :

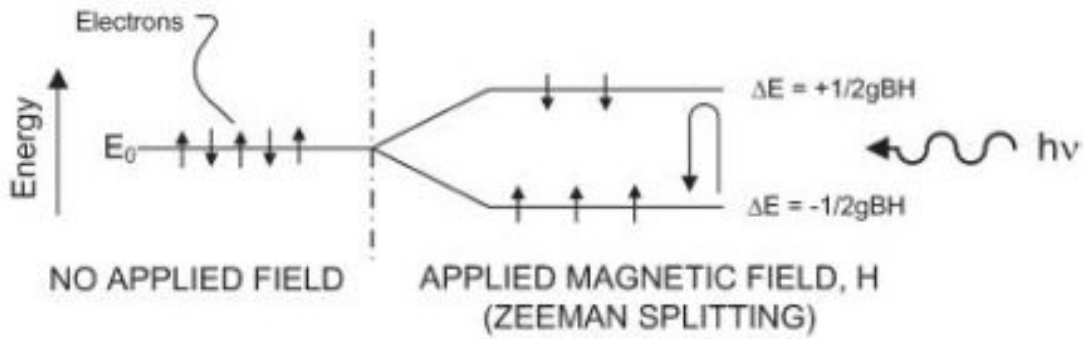
- Dose measurements (COFRAC accredited laboratory-SSDL)
- IQ/OQ/PQ of irradiation plants
- Expertise/Training

Detection of irradiated food products :

-Accredited laboratory by french ministry of economy, finance and industry

Introduction/Context

ESR Spectroscopy / Dosimetry



$$h\nu = \beta.g.B$$

→ 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

Introduction/Context

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

1991

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

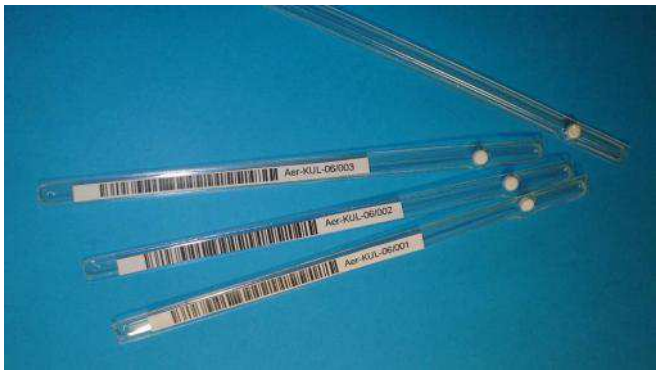
MS400
- Microwave bridge
- Transistor oscillator less heat and long term stability
- Cavity critically coupled for better reproducibility

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

2014

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

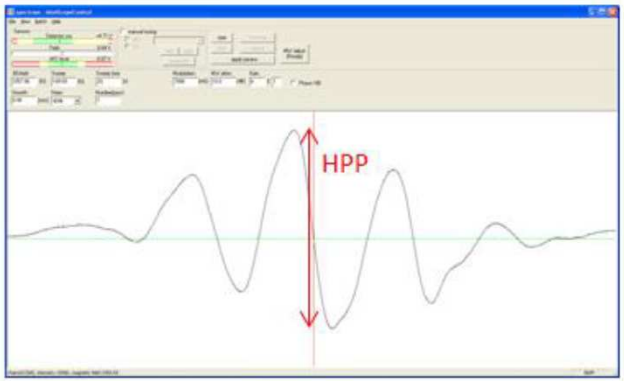
→ dosimeter which is readout with its packaging

Implication on dosimetry applications

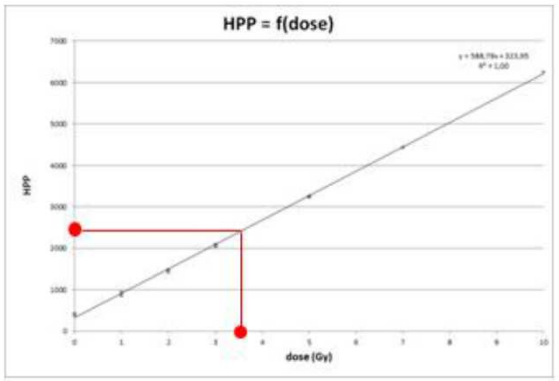
Dosimeter

Spectrometer

ESR signal



Calibration curve → Dose



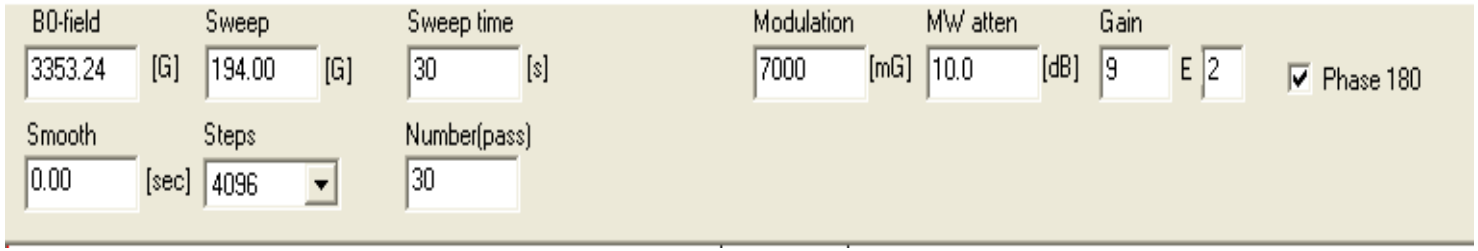
Implication on dosimetry applications

Applications	Dose range
SIT	few 10 Gy
Fruits/vegetables	few 100 Gy
Food decontamination	few kGy
Food Sterilization	few 10 kGy

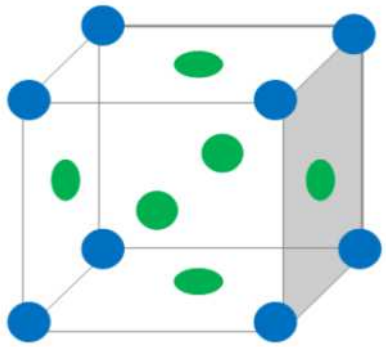
→ ESR/Alanine dosimetry system applicable to all applications

Implication on dosimetry applications

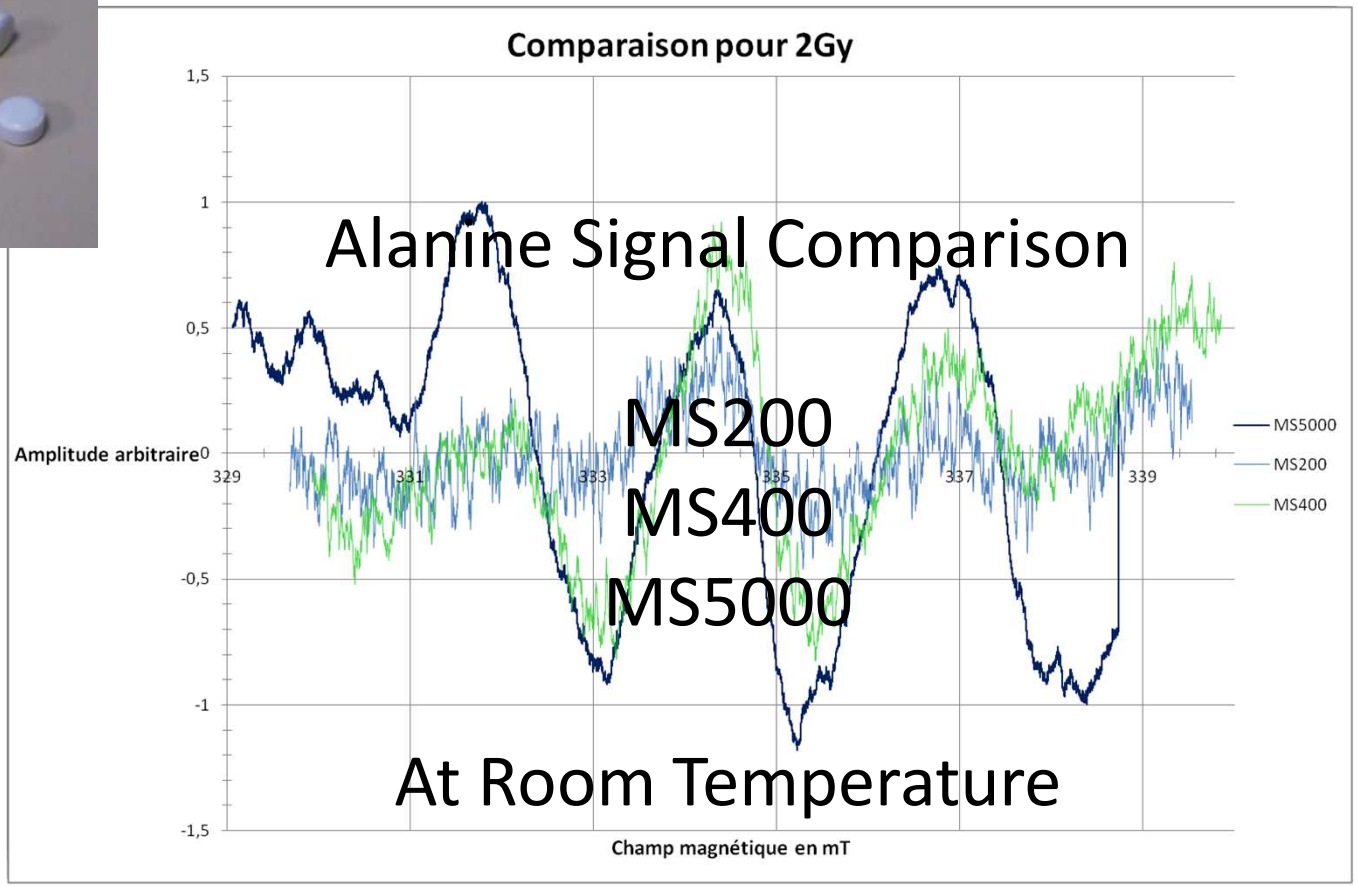
DOE approach for the choice of ESR parameters



- Sweep time
- Number of scans (reading time)
- Modulation
- MW power
- Signal analysis (Central Hpp, Integrals, Sum of Hpps, …)



Implication on dosimetry applications



Implication on dosimetry applications

Dose Gy	Reproducibility* % MS200	Reproducibility * % MS 400
1	-	4,8
2	8,9	5,3
5	4,2	2,2
10	1,9	0,8

* 20 dosimeters irradiated at same dose

Implication on dosimetry applications

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

walnuts

Chicken bones

86 Gy to 12 kGy

X Rays (100 kV)

Electron Beam 2,2 MeV

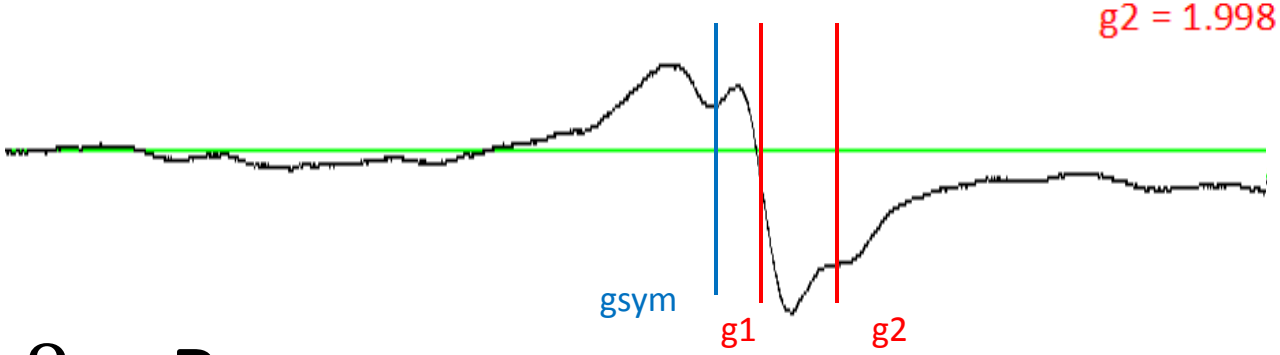
Measurement D0 at room Temperature

≈ 40 mg	≈ 30 mg
MS200, MS400, MS5000	MS200, MS400

Implication on detection of irradiated food

EN 1786

gsym = 2.005 ± 0.001
g1 = 2.002 ± 0.001
g2 = 1.998 ± 0.001



$h\nu = \beta.g.B$

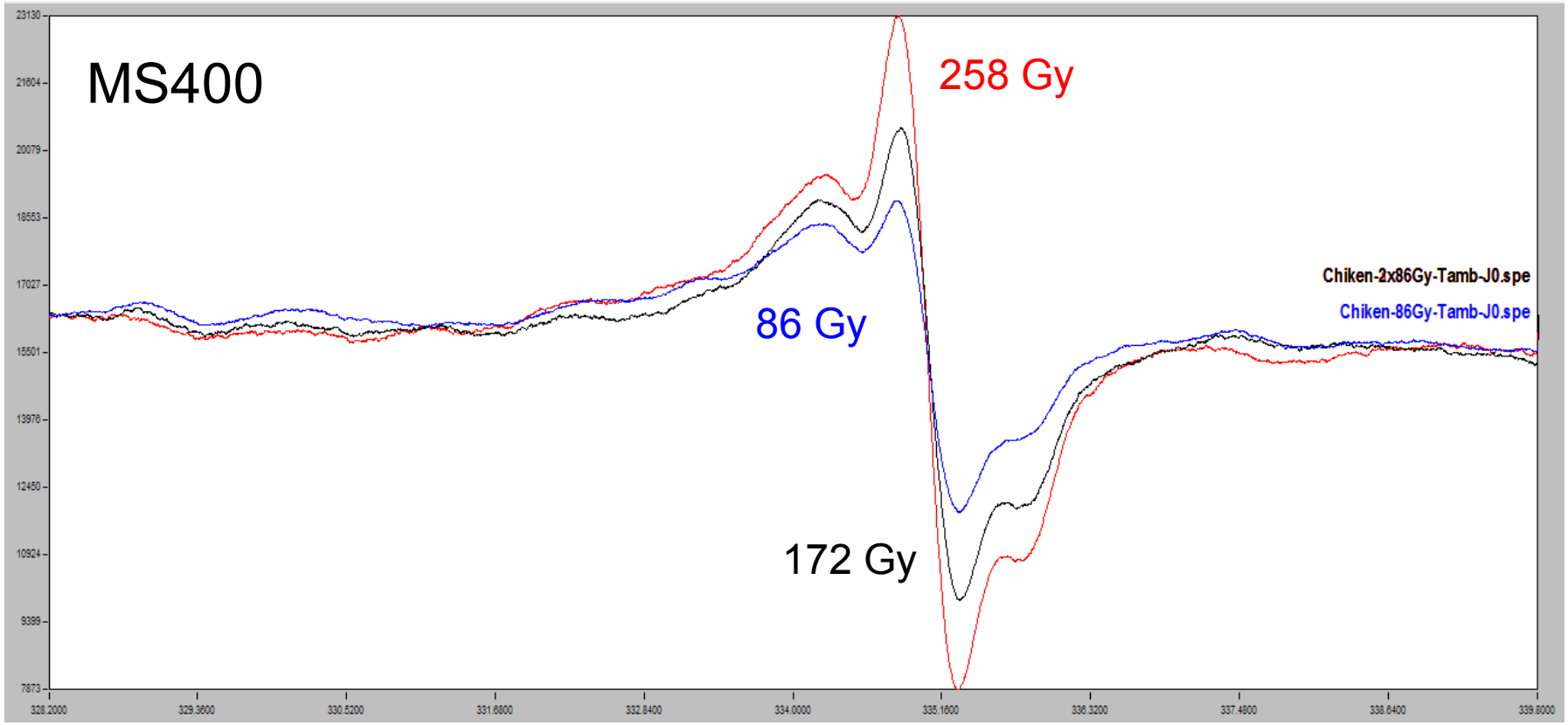
Signal of chicken bone irradiated at 6 Gy

g (signal) $= 71{,}448 \cdot \nu / B$

ν: MW Frequency (GHz)
B: Magnetic field (mT)

Implication on detection of irradiated food

B0-field		Sweep		Sweep time		Modulation		MW atten		Gain			
3340.49	[G]	116.40	[G]	30	[s]	4000	[mG]	16.0	[dB]	9	E	2	Phase 180
Smooth		Steps		Number(pass)									
0.00	[sec]	4096		120									



Implication on detection of irradiated food

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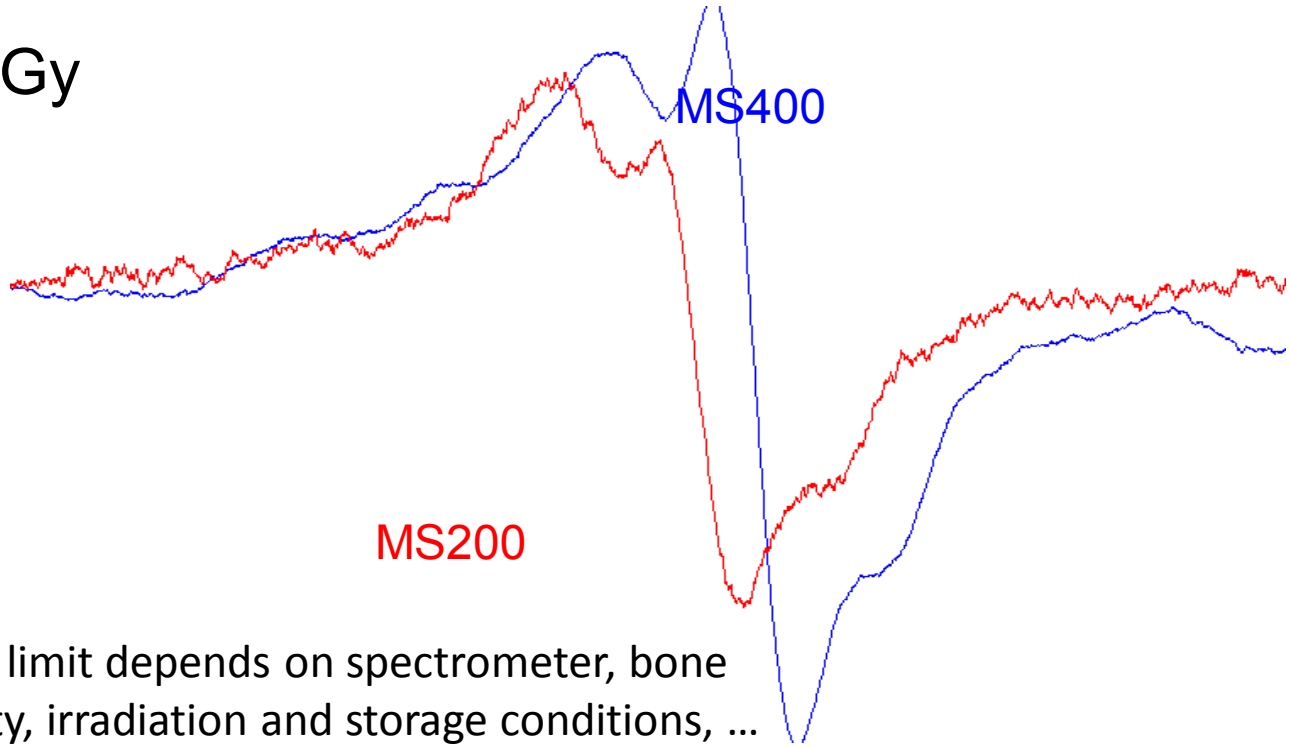
0.00	[sec]	4096		120									

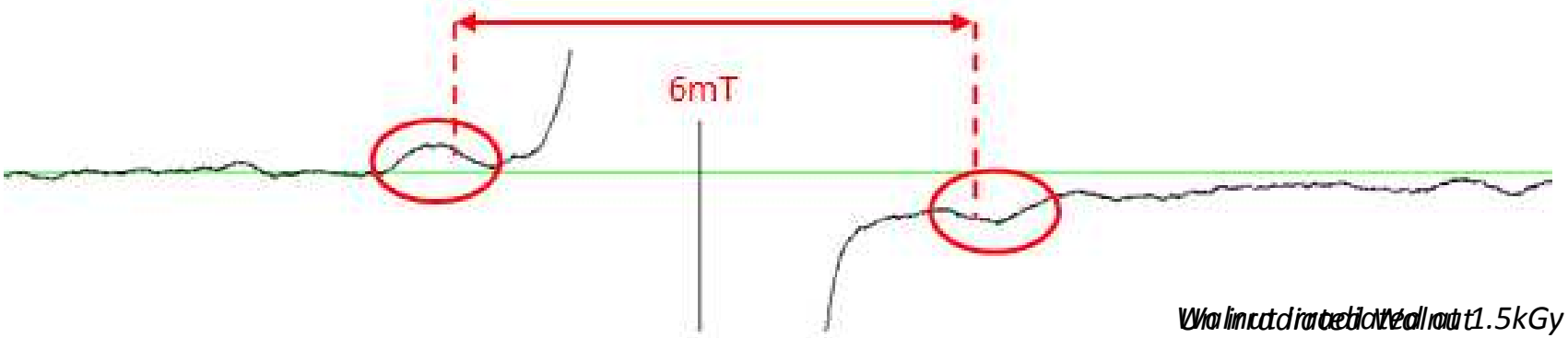
86 Gy



Detection limit depends on spectrometer, bone crystallinity, irradiation and storage conditions, …

Implication on detection of irradiated food

EN 1787

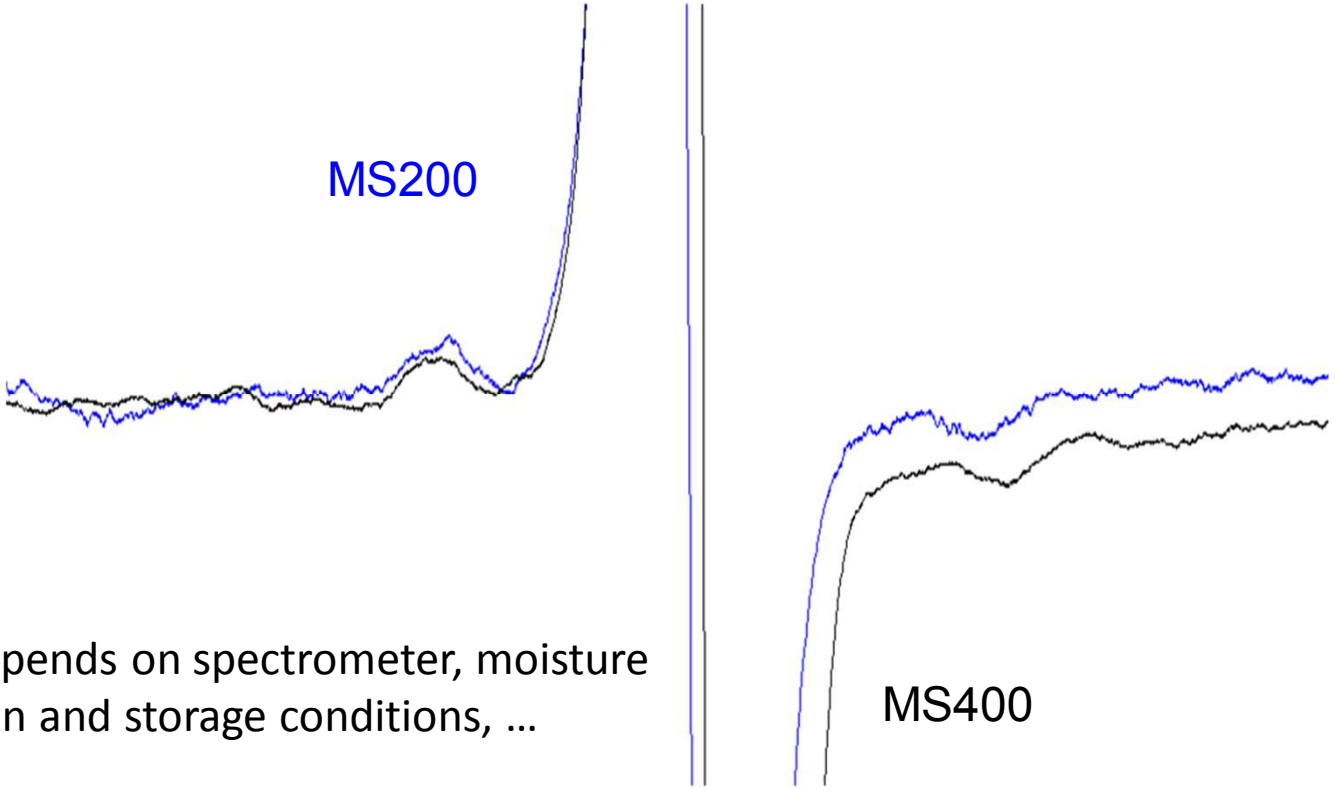


Walnut irradiated at 1.5kGy

Implication on detection of irradiated food

B0-field		Sweep		Sweep time		Modulation		MW atten		Gain			
3340.49	[G]	116.40	[G]	30	[s]	4000	[mG]	16.0	[dB]	9	E	2	Phase 180
Smooth		Steps		Number(pass)									
0.00	[sec]	4096		120									

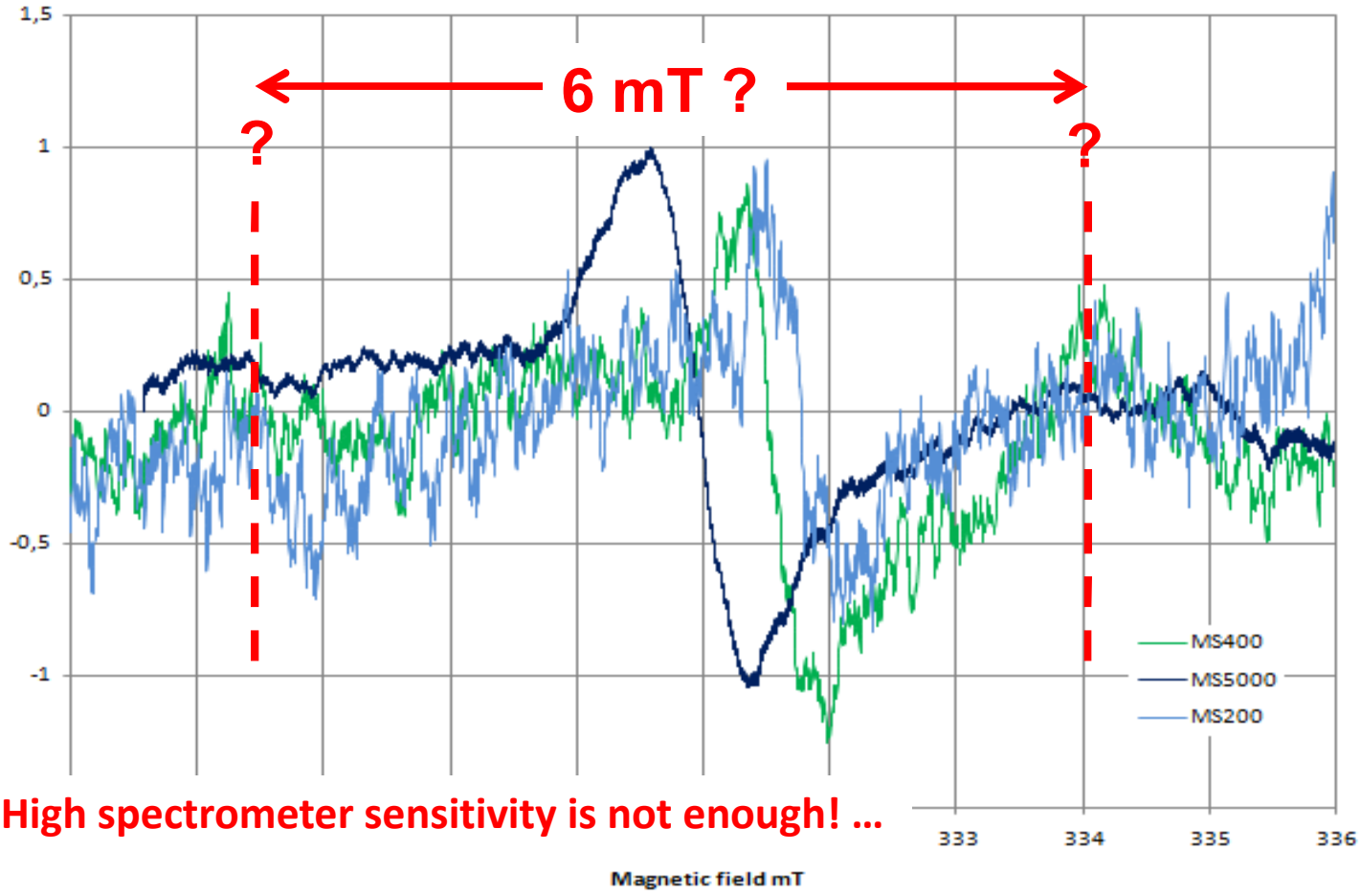
1 kGy



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

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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Thank you for your attention !

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