



CENTRE FOR ENERGY RESEARCH HUNGARIAN ACADEMY OF SCIENCES

Process Control Methods in Radiation Technologies

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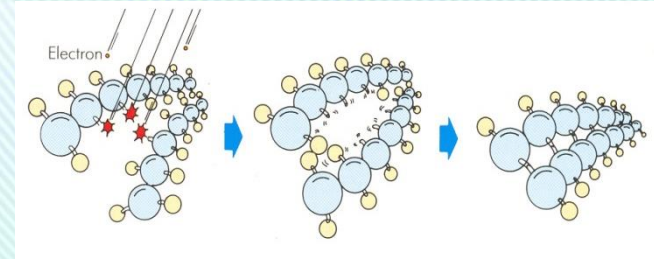
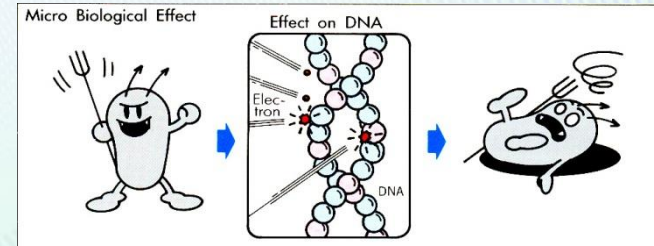
ICARST 2017



International Atomic Energy Agency

Radiation Processing Applications

- *Radiation sterilization: 25 - 50 kGy*
- *Radiation crosslinking : 10 – 200 kGy*
- *Food irradiation: 50 Gy – 10 kGy*
- *Environmental protection: 1 – 10 kGy*



Dosimetry Principles

- In radiation processing, **validation and process control** depend on the measurement of absorbed dose.
- Determination of absorbed dose in **product specific dosimeter** systems;
- Measurements of absorbed dose shall be performed using a **dosimetric system** or systems having a known level of **accuracy and precision**.
- The calibration of each **dosimetric system** shall be **traceable** to an appropriate **national standard**.

Validation and Process Control

To assure that

- the necessary (biological, chemical, physical) effect is achieved and
- the radiation technology is performed safely



the **RELATIONSHIP** between

machine parameters of irradiation facilities

(like dwell time, position of source rack, electron energy and current, conveyor speed, scanning width and homogeneity, etc.)

and

absorbed dose and dose distribution in the product

have to be measured and controlled with suitable

dosimetry systems!

Dosimetry systems in radiation processing

Reference standard systems:

- Dosimeter of high metrological quality used as a standard to provide measurements traceable to measurements made by primary standard systems;
- These systems **require calibration** and are used to calibrate radiation environments and routine dosimeters;

- Solid phase dosimetry systems :
alanine (pellet, rod, film);



- Liquid phase dosimetry systems :
Fricke solution;
potassium dichromate solution;
ethanol-monochlorobenzene solution;
ceric-cerous solution;

- Process calorimeters;



Dosimetry systems in radiation processing

Routine systems:

- Dosimetry systems used in radiation processing facilities for absorbed dose mapping and process monitoring;
- Systems, capable of giving reproducible signals;
- These systems require calibration;
- Dosimeter systems:
 - *Fricke solution*;
 - *Perspex (Red- and amber)*;
 - *Radiochromic films (FWT-60, B3 - Gex, Gafchromic)*;
 - *Radio-photoluminescent film (Sunna)*;
 - *ECB, ceric-cerous solutions*;
 - *Process calorimeters (water, graphite, polystyrene)*;



Dosimetry systems in present practice

Dosimeter system	Method of analysis	Useful dose range, Gy	Nominal precision limits	References
Fricke solution	UV – spectro-photometry	$3 \times 10^{-1} - 4 \times 10^2$	1 %	ASTM E 1026 - 04
Ceric – cerous sulphate	UV – spectro-photometry	$10^3 - 10^6$	3 %	ISO/ASTM 51205
Potassium dichromate	UV-VIS spectrophoto.	$5 \times 10^3 - 4 \times 10^4$	1 %	ISO/ASTM 51401
Ethanol-mono-chlorobenzene	Titration, or HF oscillometry	$4 \times 10^2 - 3 \times 10^5$	3 %	ISO/ASTM 51538
L - alanine	EPR	$1 - 10^5$	0.5 %	ISO/ASTM 51607
Perspex systems	VIS - spectro-photometry	$10^3 - 5 \times 10^4$	4 %	ISO/ASTM 51276
FWT – 60 film	VIS - spectro-photometry	$10^3 - 10^5$	3 %	ISO/ASTM 51275
B 3 film	VIS - spectro-photometry	$10^3 - 10^5$	3 %	ISO/ASTM 51275
Calorimetry	Resistance/temperature	$1.5 \times 10^3 - 5 \times 10^4$	2 %	ISO/ASTM 51631

CALIBRATION OF DOSIMETRY SYSTEM

- **Aim of calibration:**

Determine *relationship* between *response* of a dosimeter and *absorbed dose*.

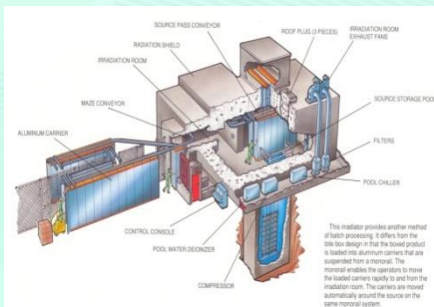
- **Subject of calibration:**

Calibration of *dosimeter* and measurement *equipment*.

- **Calibration methods:**

1. Irradiation at *calibration facility*

2. Irradiation *in-plant* with reference dosimeters



Validation procedures – EN ISO 11137 Standard

1. Process definition

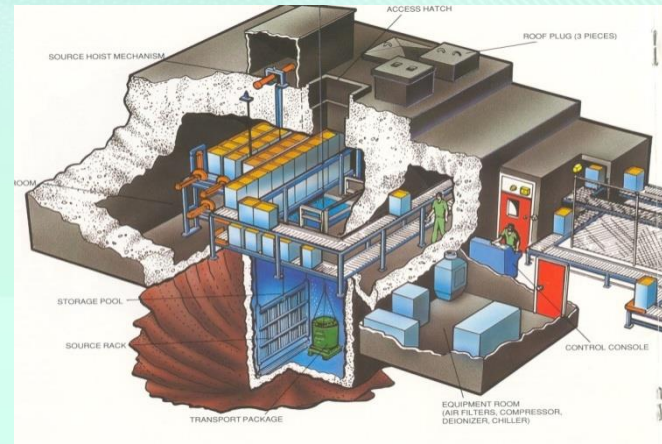
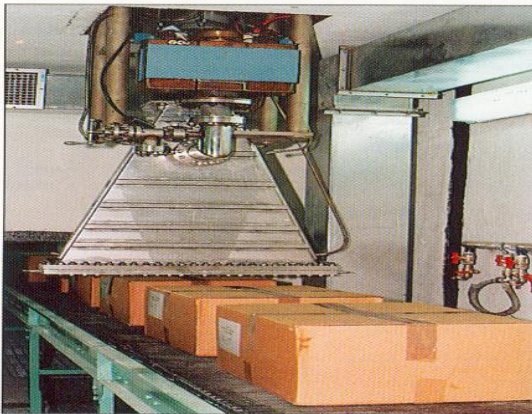
- Establishing **maximum acceptable dose** (e.g. 50 – 75 kGy);
- Establishing **process** (e.g. sterilization) **dose** (25 kGy);

2. Installation qualification

- To demonstrate that the irradiation facility has been supplied and installed according to its specifications:

To determine beam characteristics by dosimetry;

No specific dosimetric requirements to verify operation within specifications;



Validation procedures – EN ISO 11137 Standard

3. Operational qualification

Aim:

To characterize the irradiation facility relating plant parameters to absorbed dose;

Gamma facility:

nominal dose vs. dwell time, dose distribution, process interruption, transit dose;

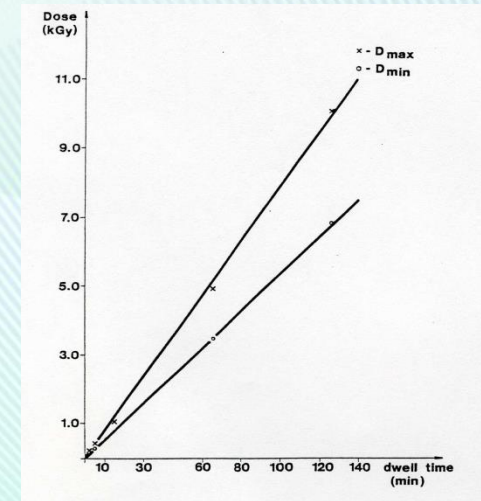
Dichromate, ECB, ceric-cerous, Gex (B3), alanine
FWT- 60, Perspex,;

Electron beam facility:

nominal dose vs. conveyor speed, beam characteristics; dose map in reference product, process interruption;

Calorimeters, ECB, alanine, Gex (B3), dichromate;
FWT-60;

dose-irradiation time:



On-line energy measurement:



Validation procedures – EN ISO 11137 Standard

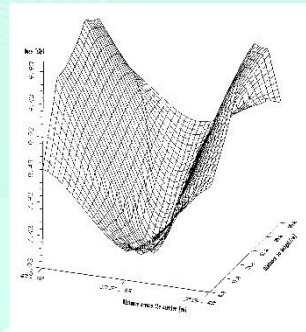
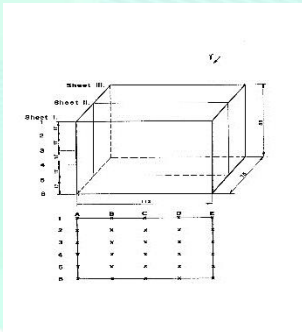
4. Performance qualification

Aim:

- To measure dose map in real product in order to locate D_{min} and D_{max} :
- to establish irradiation conditions according to required specifications:

$$D(\text{product}) > D(\text{required}) \text{ and } D(\text{product}) < D(\text{acceptable})$$

- To determine relationship between D_{min} and D_{max} and the dose at the routine monitoring position



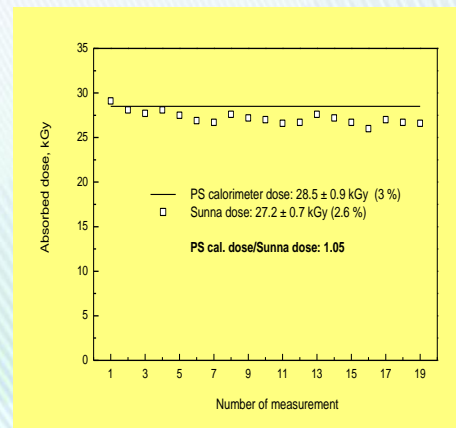
Process control

1. Measurement of process parameters:

To measure dose at the monitoring position to verify that the irradiation process is within established/required limits



knowing the relationship between D_{min} , D_{max} and $D_{monitoring}$



2. Control and monitoring of operating parameters:

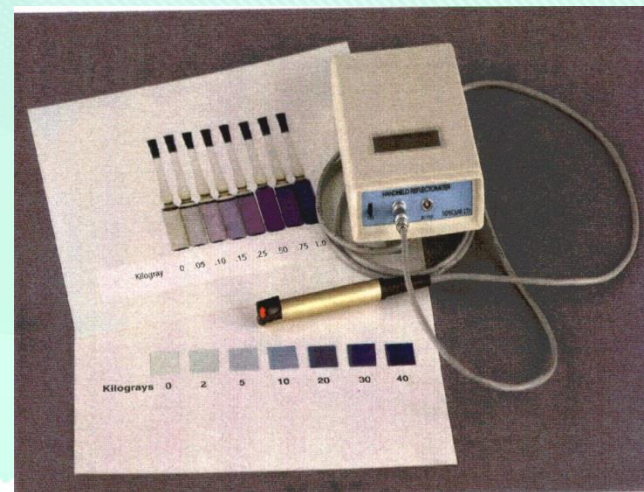
Controlled parameters:

Electron beam facility:

- Electron energy
- Beam current
- Scanned beam width
- Conveyor speed
- Routine dose

Gamma facility:

- Timer setting
- Other products present
- Routine dose



Process control in „flow systems”

1. Waste water treatment:

Aqueous alanine solution



2. Grain irradiation

Al₂O₃ TL dosimeters

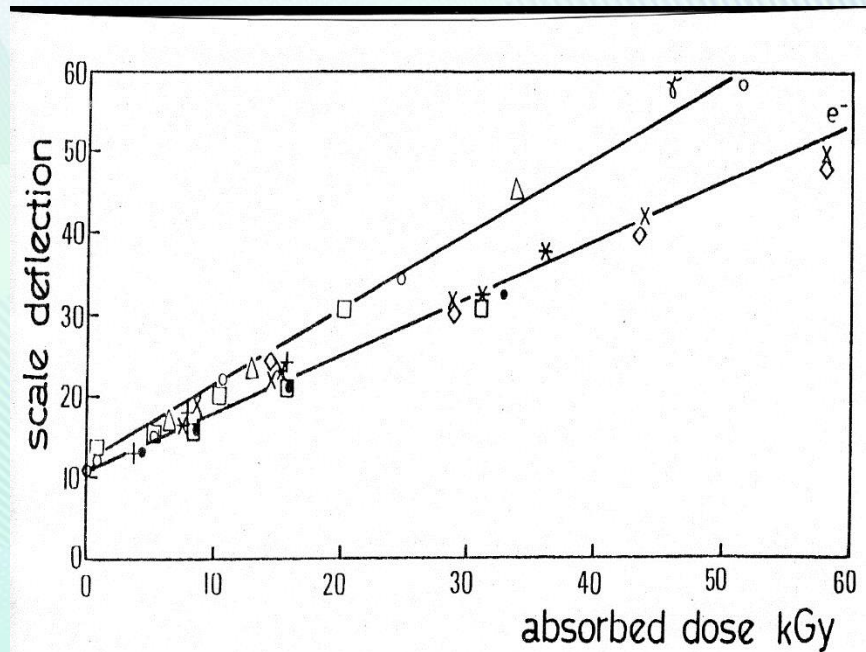
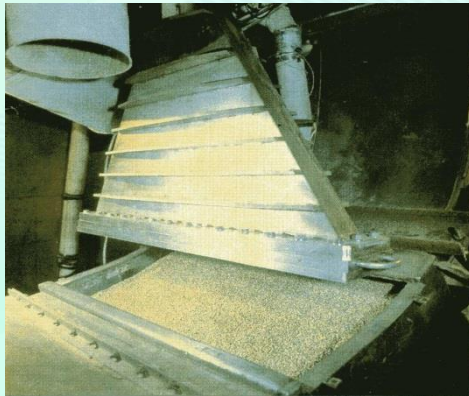


Fig. 2.

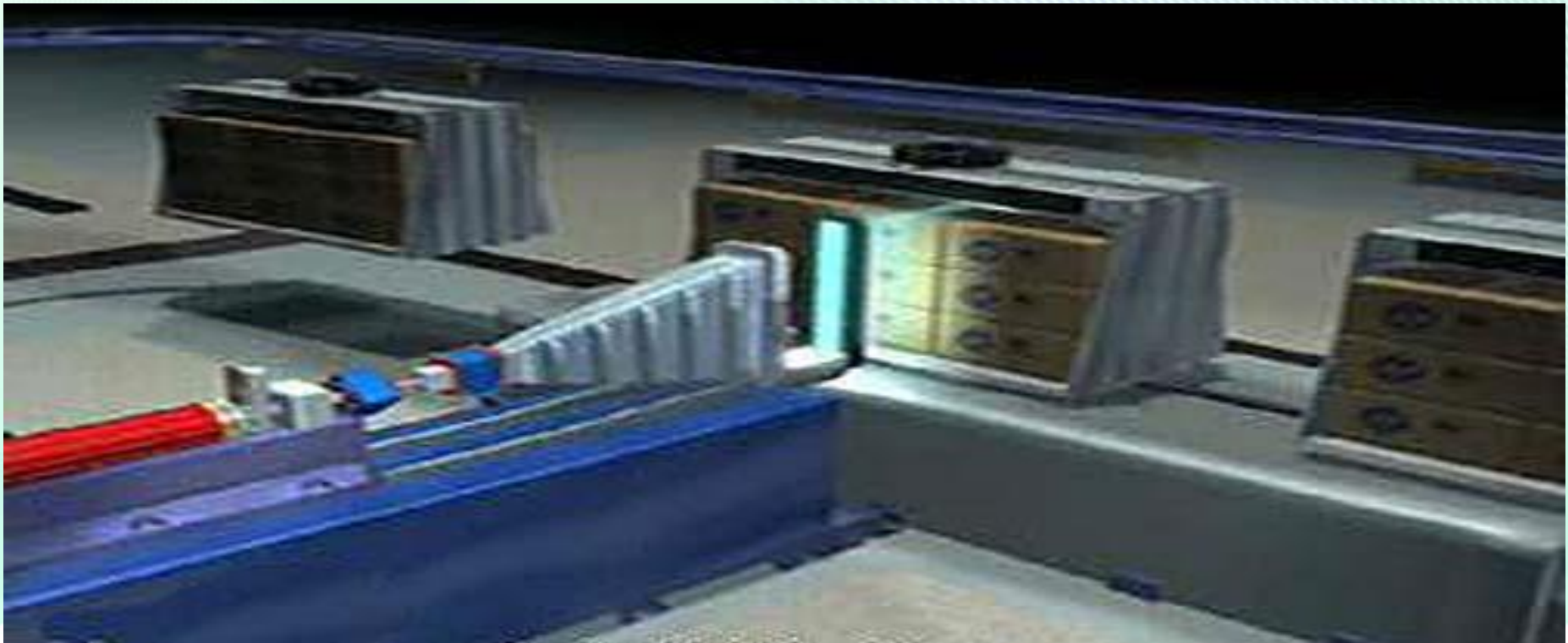
Oscillometric response of alanine solutions as a function of dose at various dose rates (⁶⁰Co irradiation), electron energies and currents (electron irradiation).

⁶⁰Co irradiation: 0.13 kGy/h: □ ; 3.5 kGy/h: Δ ; 30 kGy/h: ○ ;
electron irradiation: 4 MeV, 2.6 μs, 13 μA: ◇ ; 4 MeV, 2.6 μs, 26 μA: × ; 10 MeV,
0.5 μs, 0.4 A: + ; 10 MeV, 1 μs, 1 A: * ; 10 MeV, 4 μs, 1 A: • ;

X-Ray Machines

Dosimeters applied in gamma processing have been proven to be suitable for X-ray dosimetry:

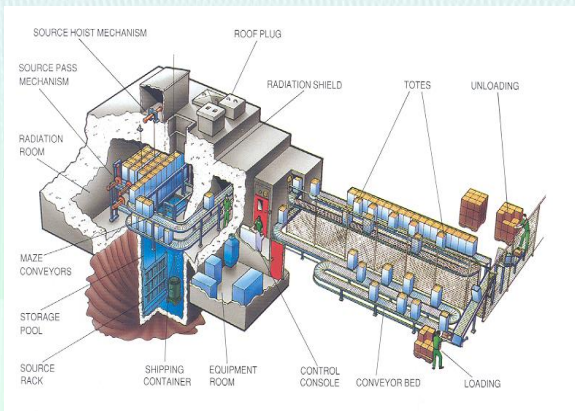
Dosimeters tested: Alanine, ECB, dichromate, ceric-cerous ;



Summary

1. Safe, economic technologies with **reliable QA/QC** methods
2. EN ISO, ISO ASTM **standards** with continuous upgrading
 - e.g. ISO 11137 – 3: Guidance on dosimetric aspects of development, validation and routine control
3. Role of IAEA Regional TC projects in radiation processing:
 - e.g. RER 1017: „Using advanced radiation technologies for materials processing”
 - **training courses** in using QA/QC methods
 - **e-learning** and IAEA **guides** in QA/QC
 - **dosimetry intercomparison** exercises (Warsaw, Poland)





Thank you for your attention