IDEAL DOSIMETRY SYSTEM

DOSIMETRY SYSTEMS IN CURRENT USE

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In radiation processing validation and process control (sterilization, food irradiation, etc.) depend on the measurement of absorbed dose.

- Measurements of absorbed dose shall be performed using a dosimetric system or systems having a known level of accuracy and precision (European standard EN552:1994).

- The calibration of each dosimetric system shall be traceable to an appropriate national standard.
Dosimetry – principles and requirements

- Determination of absorbed dose in product specific dosimetry systems – similarity in radiation absorption characteristics.

- Dosimetry – as part of the total quality system - provides quality assurance and documentation that the irradiation procedure has been carried out according to specifications.

- Accurate, traceable dose measurements provide independent, inexpensive means for quality control in radiation processing.
Dosimetry – application and systems

- Fields of application:
  - installation qualification;
  - operational qualification;
  - performance qualification;
  - process control;

- Dosimetry provides documentation in the processes, if
  - the measurement is traceable to a national standard,
  - the uncertainty of the system is known.

- Applied systems:
  - primary-, secondary-, transfer standards;
  - routine systems;
Dosimetry systems in radiation processing

**Primary standard systems:**

- Dosimeter of the highest metrological quality, established and maintained as an absorbed dose standard by a national or international standards organization for calibration of radiation environments (fields);

- Application is based on measurement of basic physical quantities;

- Most common systems: ionization chambers, calorimeters;
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-cerosus solution;
- Calorimeters;
Alanine dosimetry (ISO/ASTM 51607)

- ESR analysis; Measures free radical concentration;
- Dose range: 10 Gy – 100 kGy;
- Reproducibility < 0.5 %;
Dichromate dosimetry (ISO/ASTM 51401)

- Colour change by spectrophotometry;
- Dose range: 10 – 50 kGy;
- Reproducibility < 0.5 %;
Dosimetry systems in radiation processing

**Transfer standard systems:**

- Intermediary system with **high metrological qualities**, suitable for **transferring dose information from an accredited/standard laboratory to an irradiation facility** to establish traceability;

- These systems **require calibration** and **post irradiation stability**;

- Dosimetry systems:
  - alanine;
  - ECB, ceric-cerous, potassium dichromate solutions;
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:
  - Perspex (red-, amber-, Gammachrome);
  - Radiochromic films (FWT-60, B3, Gafchromic);
  - ECB, ceric-cerous solutions;
  - Process calorimeters (water, graphite, polystyrene);
Perspex dosimetry (ISO/ASTM 51276)

Colour change - spectrophotometry;
Dose range: 0.5 – 50 kGy;
Reproducibility < 3 %;
Post irradiation change of signal;
Radiochromic dye films (ISO/ASTM 51275)

Colour change - spectrophotometry;
FWT-60: 3 – 150 kGy;
B3: 2 – 100 kGy;
GafChromic: 1 Gy – 40 kGy;
## Dosimetry systems in present practice

<table>
<thead>
<tr>
<th>Dosimeter system</th>
<th>Method of analysis</th>
<th>Useful dose range, Gy</th>
<th>Nominal precision limits</th>
<th>References</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fricke solution</td>
<td>UV – spectrophotometry</td>
<td>$2 \times 10^{-2} - 4 \times 10^2$</td>
<td>1 %</td>
<td>ASTM E 1026 - 04</td>
</tr>
<tr>
<td>Ceric – cerous sulphate</td>
<td>UV – spectrophotom./Potentiometry</td>
<td>$10^3 - 10^6$</td>
<td>3 %</td>
<td>ISO/ASTM 51205</td>
</tr>
<tr>
<td>Potassium dichromate</td>
<td>UV-VIS spectrophotom.</td>
<td>$5 \times 10^3 - 4 \times 10^4$</td>
<td>1 %</td>
<td>ISO/ASTM 51401</td>
</tr>
<tr>
<td>Ethanol-mono chlorobenzene</td>
<td>Titration, or HF oscillometry</td>
<td>$4 \times 10^2 - 3 \times 10^5$</td>
<td>3 %</td>
<td>ISO/ASTM 51538</td>
</tr>
<tr>
<td>L - alanine</td>
<td>EPR</td>
<td>$1 - 10^5$</td>
<td>0.5 %</td>
<td>ISO/ASTM 51607</td>
</tr>
<tr>
<td>Perspex systems</td>
<td>VIS - spectrophotometry</td>
<td>$10^3 - 5 \times 10^4$</td>
<td>4 %</td>
<td>ISO/ASTM 51276</td>
</tr>
<tr>
<td>FWT – 60 film</td>
<td>VIS - spectrophotometry</td>
<td>$10^3 - 10^5$</td>
<td>3 %</td>
<td>ISO/ASTM 51275</td>
</tr>
<tr>
<td>B 3 film</td>
<td>VIS - spectrophotometry</td>
<td>$10^3 - 10^5$</td>
<td>3 %</td>
<td>ISO/ASTM 51275</td>
</tr>
<tr>
<td>Cellulose triacetate</td>
<td>UV – spectrophotometry</td>
<td>$10^4 - 10^6$</td>
<td>3 %</td>
<td>ISO/ASTM 51650</td>
</tr>
<tr>
<td>Calorimetry</td>
<td>Resistance/temperature</td>
<td>$1.5 \times 10^3 - 5 \times 10^4$</td>
<td>2 %</td>
<td>ISO/ASTM 51631</td>
</tr>
</tbody>
</table>
## Environmental effects on dosimetry systems

<table>
<thead>
<tr>
<th>Dosimeter</th>
<th>Measurement time after irr.</th>
<th>Humidity</th>
<th>Dose rate (Gy s(^{-1}))</th>
<th>Irradiation temp. coeff., (°C(^{-1}))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Alanine</td>
<td>immediately</td>
<td>yes</td>
<td>&lt; 10(^8)</td>
<td>+ 0.25 %</td>
</tr>
<tr>
<td>Dichromate</td>
<td>24 hours</td>
<td>no</td>
<td>0.7 – 5x10(^2)</td>
<td>- 0.2 %</td>
</tr>
<tr>
<td>Ceric-cerous</td>
<td>immediately</td>
<td>no</td>
<td>&lt; 10(^6)</td>
<td>conc. dep.</td>
</tr>
<tr>
<td>ECB</td>
<td>immediately</td>
<td>no</td>
<td>&lt; 10(^8)</td>
<td>+ 0.05 %</td>
</tr>
<tr>
<td>Calorimeters</td>
<td>immediately</td>
<td>no</td>
<td>&lt; 10(^8)</td>
<td>-</td>
</tr>
<tr>
<td>Perspex</td>
<td>24 hours</td>
<td>yes</td>
<td>&lt; 10(^5)</td>
<td>+ 1 %</td>
</tr>
<tr>
<td>FWT-60</td>
<td>5 min/60 °C</td>
<td>yes</td>
<td>&lt; 10(^{13})</td>
<td>+ 0.2 %</td>
</tr>
<tr>
<td>B3</td>
<td>5 min/60 °C</td>
<td>yes</td>
<td>&lt; 10(^{13})</td>
<td>+ 0.3 %</td>
</tr>
<tr>
<td>Sunna</td>
<td>20 min/70 °C</td>
<td>no</td>
<td>&lt; 10(^{13})</td>
<td>+ 0.2 %</td>
</tr>
</tbody>
</table>
NEW APPROACHES

- New requirements:
  - standardization of existing dosimetry methods;
  - increased reliability to encourage industry for wider use of ionizing radiation;
  - new technologies (may) require new dosimetry methods;
  - introduction of improved calibration procedures;
New approaches – novel dosimetry systems

- **Requirements:**
  - New technologies (environmental processes, food irradiation at low temperatures, anthrax, pharmaceuticals, X-ray technologies, high dose control);

- **Achieved by:**
  - Improvement of existing dosimetry systems;
  - Introduction of new systems;
Systems based on conductivity evaluation

- Ethanol-monochlorobenzene solution (1 – 300 kGy) (non-destructive method)

- Aqueous – alanine solution (1 – 100 kGy)

- Polyaniline based polymer composites (5 – 150 kGy)
Systems based on fluorimetry

Principles:
- Absorbed energy is emitted as fluorescent light due to optical excitation (OSL – optically stimulated luminescence);
- Fluorescence appears micro- or nanoseconds after excitation;

Advantages:
- Wide dynamic range;
- High sensitivity;
- Passive and real time dosimetry;
- Variable geometries;
- Inexpensive detectors;
- Multipurpose applications (medical diagnostic, radiation processing, radiation protection, space studies, etc);
Systems based on fluorimetry

- Application possibilities:
  - Radiation induced decay of originally fluorescent molecules (anthracene, fluorescein derivatives, etc);
  - Appearance of radiation induced fluorescence due to formation of new fluorescent radiolysis products (Sunna film);
The Sunna dosimeter

**Principles:**

- LiF dispersed uniformly in PE (1 cm x 3 cm x 0.4 cm);
- Colour centers (F-, M-, N-, R centers) form due to ionizing radiation;
- Red, green or IR OSL or UV absorption used for dosimetry;

![Net Fluorescence for Sunna 0399-20 Dosimeter](image)
The Sunna dosimeter

- Application possibilities:
  - Evaluation of green OSL (50 Gy – 250 kGy);
  - Evaluation of UV absorbance (5 – 100 kGy);
  - Evaluation of IR OSL (10 Gy – 10 kGy);

![Graph showing net signal vs absorbed dose](image)
Systems based on optical absorption (tetrazolium salts)

- Tetrazolium salts studied:

<table>
<thead>
<tr>
<th>Compound</th>
<th>Product $\lambda_{\text{max.}}$</th>
<th>Dose range:</th>
</tr>
</thead>
<tbody>
<tr>
<td>tetrazolium violet (TV)</td>
<td>525 nm</td>
<td>0.01 – 30 kGy</td>
</tr>
<tr>
<td>tetrazolium red (TTC)</td>
<td>490 nm</td>
<td>0.01 – 100 kGy</td>
</tr>
<tr>
<td>tetrazolium blue (TB)</td>
<td>520 nm</td>
<td>0.01 – 10 kGy</td>
</tr>
<tr>
<td>nitro blue tetrazolium (NBT)</td>
<td>522 and 612 nm</td>
<td>0.01 – 25 kGy</td>
</tr>
</tbody>
</table>

- heterocyclic organic compounds, which upon irradiation yield highly coloured water insoluble formazans due to radiolytic reduction.
Measurement of absorbed dose by measuring optical absorption of or reflected light from NBT - PVA film
Instrument to measure reflected light

Reflectometer:
Calorimetric systems

- 4 – 10 MeV:
  - graphite, water, PS calorimeters;
  - calibration, nominal dose measurements;

- 1.5 – 4 MeV:
  - PS calorimeter;
  - calibration, nominal dose determination;

- 80 – 120 keV:
  - graphite calorimeter;
  - to develop a primary standard system;
  - calibration;
Novel approaches - methodology

- Dosimetry automation – to reduce human errors
  - Automated evaluation systems (Aerial, Bruker, etc.);
  - Automated scanning of images (dose distribution) – RisøScan;

- Real time dosimeter application
  - To determine combined irradiation temperature – dose rate effects;

- Mathematical methods (RPSMUG – for modeling and simulation)
  - To assist product design;
  - To determine dwell time setting;
  - Rapid estimation of dose distribution;
  - Parallel irradiation of complex products;
Quality control has to be based on the assurance that the process was carried out within prescribed dose limits.

This requires proper use and selection of dosimetry systems.

Selection criteria:
- according to the process to be controlled (sterilization, polymer modification, food processing, etc.); dose range: 50 Gy – 300 kGy;
- according to dosimeter characteristics;
Selection criteria

- Reliable calibration and standardization;
- Broad absorbed dose range;
- Limited or no dependence of response with dose, dose rate, energy;
- Radiation absorption characteristics similar to product;
- Stability, reproducibility, simplicity, low cost, availability;
- Small size – good resolution capabilities;
- Known environmental (influence) factors;
- Simple handling and read-out procedure;
- Limited variation within and among batches;
- Long shelf life;
- Ruggedness;
- Portability;
"Ideal dosimetry system"

W.L. McLaughlin: „If you have one dosimeter you think you know the dose. If you have two dosimeters you start to wonder…”

**Optimum conditions:**
- wide dose range – gamma/EB/X-ray
- no environmental effects
- immediate evaluation after irradiation
- low price
- multiple use
- automated, non-destructive evaluation

**Presently available systems:**
- process calorimeters
- ESR alanine
- oscillometric ECB
- (Sunna film)
FACTORS AFFECTING DOSIMETER ACCURACY

1. Irradiation conditions are different from calibration conditions:
   - temperature, dose rate, relative humidity, energy spectrum, irradiation geometry, etc.

2. Storage conditions:
   - before and after irradiation

3. Instrumental errors:
   - absorbance and wavelength scale, scattered light, transfer of calibration curve from one instrument to another one, etc.
Accuracy and Precision

- Poor accuracy, poor precision
- Improved measurement technic
- Poor accuracy, good precision
- Calibration
- Good accuracy, good precision
**AIM OF CALIBRATION**

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

- **Influence factors:**
  - dose rate
  - temperature
  - time
  - humidity, etc.

- Minimize effects of influence factors by optimum calibration conditions.
Dosimetry calibration – new trends

- **In-plant calibration:**
  - Calibration phantoms (Gamma, electron);

- **Internet calibration (NIST):**
  - protected software;
  - alanine dosimetry;
CALIBRATION OF DOSIMETRY SYSTEM

- Calibration of dosimeter
- Calibration of equipment
All measurement equipment must be calibrated and be traceable to national standards.

Certain measurement equipment cannot be calibrated (e.g. signal amplitude from an EPR spectrometer)

therefore

the stability of the equipment has to be demonstrated by the use of measurement standards (e.g. stable EPR spin standards).
CALIBRATION OF EQUIPMENT (2)

- **Spectrophotometer:**
  absorbance and wavelength scale with calibrated optical filters;

- **Thickness gauge:**
  calibrated gauge blocks;

- **Thermometers:**
  calibrated thermometers;

- **Resistance measurement (Ohm-meter for calorimeters):**
  calibrated reference resistor;

- **Humidity meters:**
  saturated salt solutions;
CALIBRATION OF DOSIMETERS (1)

- Irradiation of dosimeters
- Measurement (analysis) of dosimeters (with calibrated instrument)
- Generation of calibration curve or response function
- Initial calibration verification, and periodically confirmation of validity
- Traceability chain
- **Dose range:**
  Larger dose range than intended use;

- **Number of dose points:** (4 dosimeters at each point)
  Dose range less than one decade: 5 points (at least)
  - arithmetically (10 - 20 - 30 - 40 - 50 kGy);

  Dose range greater than one decade: 5 points (at least) per decade
  - geometrically (1 - 1.5 - 2.3 - 3.4 - 5.1 - 7.6 - 11.4 - 17 - 26 - 38 - 58 - 87 kGy);

- **Batch calibration:**
  Each new batch must be calibrated (annual checks);
  Don’t use manufacturers` s calibration curve - unless verified;

- **Post irradiation stability:** to control!
IRRADIATION OF DOSIMETERS

1. Irradiation at a **calibration facility**

Irradiation of dosimeters in the reference radiation field of a calibration laboratory (or of an in-house calibration facility) followed by “calibration verification” in the irradiation plant.

a./ **advantages**
- easy to obtain full dose range;
- irradiation to accurately known doses under controlled and documented conditions;

b./ **disadvantages:**
- different conditions from real use (uncertainties);
- transport of dosimeters (pre- and post-irradiation storage effects - uncertainties);
Calibration facility

Calibration holder ↓
IRRADIATION OF DOSIMETERS

2. Irradiation in plant

Routine dosimeters are irradiated together with reference or transfer standard dosimeters in “calibration phantoms” in the irradiation plant.

a./ advantages:
   - calibration and production conditions are similar (environmental conditions);

b./ disadvantages:
   - difficult to obtain full dose range in certain plants;

c./ care must be taken:
   - to ensure that all dosimeters irradiated together receive the same absorbed dose
GAMMA IRRADIATION PHANTOM

- To be placed in a region of low dose gradient;

- The effect of irradiation temperature on the reference dosimeters must be considered:

\[ T_{\text{eff}} = T_{\text{min}} + \frac{2}{3} (T_{\text{max}} - T_{\text{min}}); \]

- Use of temperature labels;
- It is irradiated separately, not in e.g. dummy product;

- Specific location on the depth dose curve should be chosen;

- The effective irradiation temperature can be considered:
  \[ T_{\text{eff}} = \frac{T_{\text{min}} + T_{\text{max}}}{2}; \]
IMPORTANCE OF TRACEABILITY

The ability to show that a measurement is consistent with the appropriate national or international standards through an *unbroken* chain of comparison

(\text{primary standard lab.} \rightarrow \text{secondary standard lab.} \rightarrow \text{routine lab.})

(verification is needed)
CALIBRATION VERIFICATION

- Calibration curves prepared for routine dosimeters in a calibration facility (or in-house calibration facility) must be verified for the actual conditions of use in the production irradiation facility;

- Routine dosimeters have to be irradiated together with reference or transfer standard dosimeters to at least three different absorbed doses.

- Absorbed dose results originating from the two types of dosimeters must be analyzed with respect to any systematic trends for potential corrections if needed.
ANALYSIS

1. Analysis of dosimeters
   - use of calibrated instrumentation;
   - time of analysis after irradiation (potential changes of dosimeter response after irradiation);

2. Analysis of calibration data
   - mean response and sample standard deviation;
   - calculation of coefficient of variation;

3. Preparation of calibration curve
   - signal = f(dose)
   - evaluation of mathematical expression (e.g. calculation of "percentage residuals") to select best fit;
EXAMINATION OF RESIDUALS

- Select the mathematical expression for the \( \text{dose} = f(\text{signal}) \) relationship (e.g. lowest order polynomial);

- Determine the coefficients of the polynomial (use individual dosimeter points);

- Calculate the dose for each calibrated dosimeter;

- Calculate "percentage residuals":

\[
\frac{(D_{\text{calculated}} - D_{\text{delivered}})}{D_{\text{delivered}}} \times 100
\]

- Plot "percentage residuals" against dose and examine data for any systematic trends;
EXAMINATION OF RESIDUALS
(example)

Calibration curve and function → Percentage residual

Harwell AH 4th

\[ y = -1.6466 \times 10^{-7}x^4 + 5.3813 \times 10^{-5}x^3 - 8.2926 \times 10^{-3}x^2 + 8.5069 \times 10^{-1}x - 7.1433 \times 10^{-3} \]

\[ R^2 = 9.9999 \times 10^{-1} \]
THANK YOU FOR YOUR ATTENTION!

Accuracy

Precision