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Joint innovative training and teaching/ learning program in enhancing development and transfer knowledge of application of ionizing radiation in materials processing

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5. Radiation Measurements: Instruments And Methods

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INTRODUCTORY NOTES

- Radiation measurements cover a broad area of instruments and methods focusing on measurements of different parameters of radiation;
- Dose measurements play a fundamental role in radiation processing of materials:

✓ Validation and radiation process control 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***;

✓ The calibration of each dosimetric system shall ***be traceable*** to an appropriate national standard.

INTRODUCTORY NOTES

This presentation is focused on dose measurements from two points of wiev:

1. Instruments and methods for **absorbed dose measurements, validation and verification.**
2. Instruments and methods for **dose monitoring** (occupational dosimetry)

Note

Measurements of irradiation process parameters, dosimetry issues for QC and QA in radiation processing will be covered during practical training and in the future lecture of dr. G. Przybytniak

TABLE OF CONTENTS

1. Classification of dosimetry systems;
2. Selection of dosimetry systems;
3. Characterization of dosimetry systems: Instruments and methods.

Note

Futher reading is suggested: ***Guidelines for the development, validation and routine control of industrial radiation process. IAEA, Vienna, 2013***

CLASSIFICATION OF DOSIMETRY SYSTEMS

Classification of dosimetry systems is based on:

- ***metrological properties*** of the dosemeter

- ***field of application***

CLASSIFICATION OF DOSIMETRY SYSTEMS

New addition: E 2628 – 09 (ASTM Standard);

System classification based on ***metrological properties*** **(type I and II);**

Type I:

dosemeter of high metrological quality which response is affected by individual influence quantities in a well defined way so, that it ***can be expressed in terms of independent correction factors***;

Fricke, dichromate, ceric-cerous sulphate, Ethanol-chlorobenzene solution (ECB), alanine/EPR dosemeters

Type II:

dosemeter, which response is affected by influence quantities in a complex way – ***cannot be expressed in terms of independent correction factors***;

Process calorimeter, Cellulose triacetate (CTA), LiF, PMMA, perspex systems, radiochromic films.

CLASSIFICATION OF DOSIMETRY SYSTEMS

System classification based on ***field of application***

Reference standard systems (type I);

Used to calibrate dosimeters for routine use, therefore high metrological qualities, low uncertainty and traceability to appropriate national or international standards are needed. +/- 3 % (k = 2);

Fricke solution, alanine/ EPR dosimeter system, potassium dichromate solution, ceric-cerous sulphate solution and ethanol-monochlorobenzene (ECB) solution).

Routine systems (type II);

Used for routine absorbed dose measurements (i.e. dose mapping and process monitoring). Traceability to national or international standards is needed. +/- 6 % (k = 2);

Perspex systems, ECB, cellulose triacetate, Sunna film and radiochromic films such as FWT-60 and B3/GEX)

HIERARCHY OF STANDARDS

PSDL: Standards laboratory

Calorimeters, ionization chambers [D_w , Gy (±1%) D_w , kGy (±2%)]

|

SSDL: Reference standard dosimetry systems (±3%)

Fricke, ceric, dichromate, alanine, calorimeters, ECB, ionization chambers

|

Transfer standard dosimetry systems

Alanine; ethanol-chlorobenzene (ECB); potassium dichromate; ceric-cerous

|

Routine dosimetry systems (±6%)

Films, plastics, dyed plastics, TLD, OSLD, semiconductor devices …

Certificates issued by a NMI or a laboratory accredited to ISO 17025 can be taken as proof of traceability and no further action is required by the user.

PRIMARY STANDARDS

- Primary standards are instruments of the highest metrological quality that permit determination of the unit of a quantity from its definition, the accuracy of which has been verified by comparison with standards of other institutions of the same level.
- Primary standards are realized by the primary standards dosimetry laboratories (PSDLs) in about 20 countries worldwide.
- Regular international comparisons between the PSDLs, and with the Bureau International des Poids et Mesures (BIPM), ensure international consistency of the dosimetry standards.

PRIMARY STANDARDS

- Radiation detectors used for calibration of radiation beams (industry, medicine) must have a calibration coefficient traceable (directly or indirectly) to a primary standard.
- Primary standards are not used for routine calibrations, since they represent the unit for the quantity at all times.
- Instead, the PSDLs calibrate secondary standard dosimeters for secondary standards dosimetry laboratories (SSDLs) that in turn are used for calibrating the reference instruments of users, such as therapy level ionization chambers (hospitals) or calorimeters (radiation processing)

REFERENCE AND TRANSFER STANDARD SYSTEMS

Reference standard system

- Dosimeters of high metrological quality are used as a standard to provide measurements traceable to measurements made by primary standard systems.

Transfer standard system

- Intermediary system with high metrological qualities, suitable for transferring dose information from an accredited/standard laboratory to an irradiation facility to establish traceability (comparing absorbed dose measurements). *Dosimetry intercomparison exercises are requested.*

Both systems require calibration

TRACEABILITY

The ISO defines measurement traceability as:

"Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties."

---

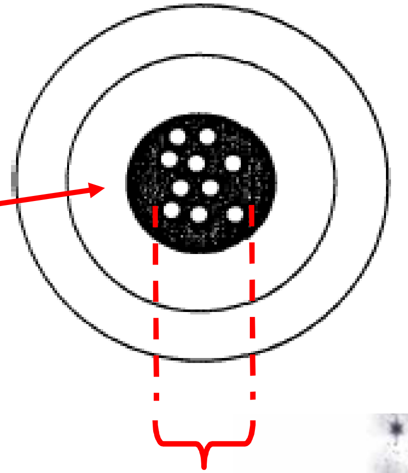
comparison = calibration

ACCURACY AND PRECISION

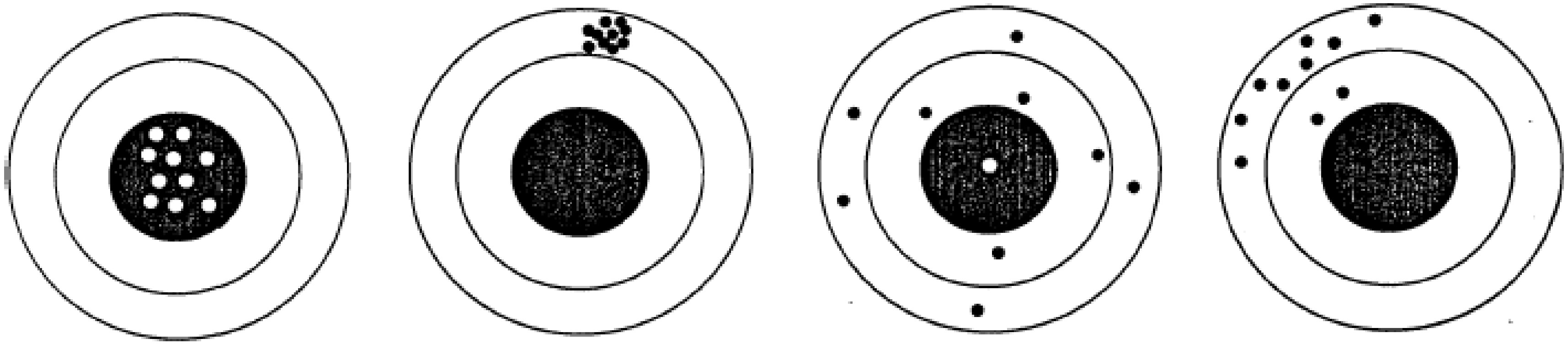
Accuracy specifies the proximity of the mean value of a measurement to the true value.

Precision specifies the degree of reproducibility of a measurement.

Note:
High precision is equivalent to a small standard deviation.



ACCURACY AND PRECISION



High precision
High accuracy

High precision
Low accuracy

Low precision
High accuracy

Low precision
Low accuracy

The accuracy and precision associated with a measurement is often expressed in terms of uncertainty (ISO guidelines)

UNCERTAINTIES

The result of a (dose) measurement is only an approximation or estimate of the (dose) value and it is complete only when accompanied by a quantitative statement of its uncertainty:

Absorbed dose = 27.4 +/- 0.55 kGy

Formal definition of uncertainty:

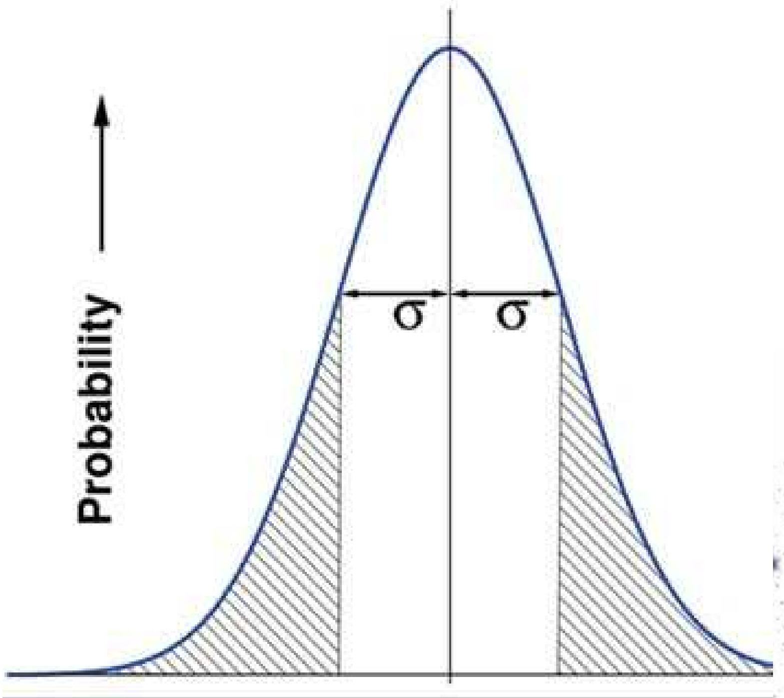
Uncertainty is a parameter associated with the result of a measurement. It characterizes the dispersion of the values (= range of the values) that could reasonably be attributed to the measurand (=absorbed dose).

Note:

Quantities such as the "true value" and the deviation from it, the "error", are basically unknowable quantities. Therefore, these terms are not used in the "Guide to the expression of uncertainty".

UNCERTAINTIES

- In general terms, measurement uncertainty can be regarded as the probability that the measurement lies within a range of values, i.e. it is a statistical concept.
- To a good approximation, the distribution of possible values often follows a Gaussian or normal distribution.



UNCERTAINTIES

There two types of uncertainties that should be investigated performing measurements:

Type A uncertainties (random) are evaluated by statistical analysis of series of measurements (e.g. standard deviation of the mean) and are related mainly to **precision** (i.e. reproducibility) of the dosimeter response.

Type B uncertainties (non-random, systematic) are evaluated by means **other than statistical analysis** (based on scientific judgement, e.g. previous experimental data) – **B type (non-random, systematic)** and are related mainly to calibration (**accuracy**).

Type A standard uncertainties, u_A:

If a measurement of a dosimetric quantity x is repeated N times, then the best estimate for x is the arithmetic mean of all measurements x_i

$$\bar{x} = \frac{1}{N}\sum_{i=1}^{N} x_i$$

The standard deviation σ_x is used to express the uncertainty for an individual result x_i:

$$\sigma_x = \sqrt{\frac{1}{N-1}\sum_{i=1}^{N}\left(x_i - \bar{x}\right)^2}$$

UNCERTAINTIES

The standard deviation of **the mean value** is used to express the uncertainty for the **best estimate**:

$$\sigma_{\bar{x}} = \frac{1}{\sqrt{N}}\sigma_x = \sqrt{\frac{1}{N(N-1)}\sum_{i=1}^{N}\left(x_i - \bar{x}\right)^2}$$

The standard uncertainty of type A, denoted u_A, is defined as the standard deviation of the mean value

$$u_A = \sigma_{\bar{x}}$$

UNCERTAINTIES

Type B standard uncertainties, u_B:

- If the uncertainty of an input component **cannot** be estimated by repeated measurements, the determination must be based on other methods such as intelligent guesses or scientific judgments.
- Type B uncertainties may be involved in:
    - influence factors on the measuring process
    - the application of correction factors
    - physical data taken from the literature

UNCERTAINTIES

Combined uncertainty

Having evaluated standard uncertainties associated with each component of measurement, the **combined uncertainty,** u_C, associated with a particular measurement is obtained by summing in quadrature standard uncertainties of the individual component , i.e. by taking the square root of the sum of the squares of the individual components:

$$u_c = (u_1{}^2 + u_2{}^2 + u_3{}^2 + \ldots\ldots)^{1/2}$$

Expanded uncertainties, U:

The combined uncertainty is assumed to exhibit a **normal distribution.**

Then the combined standard uncertainty u_C corresponds to a confidence level of 67% .

A higher confidence level is obtained by multiplying u_C with a coverage factor denoted by k:

$$U = k \cdot u_C$$

For $k = 2$, the expanded uncertainty corresponds to the 95% confidence level.

SELECTION OF DOSIMETRY SYSTEM

Application criteria is most important one for the selection of a suitable dosimetry system:

- Industry, medicine, research;
- Reference, transfer, routine standard;
- Absorbed dose, dose rate, dose equivalent;
- Active or passive;
- Solid, liquid, gaseous…..

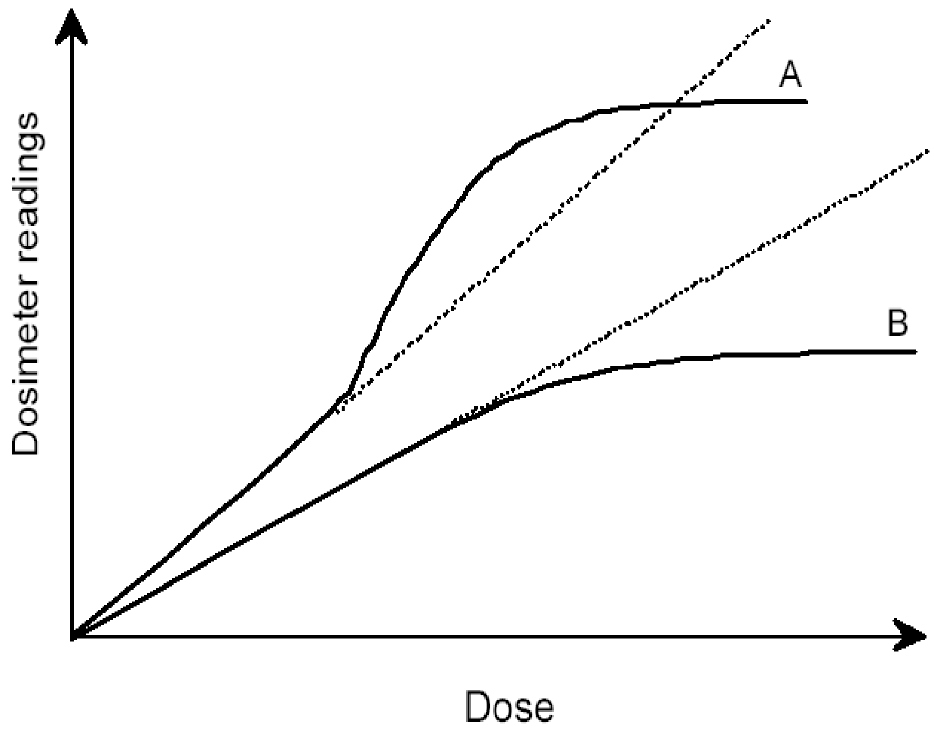
When selected, ***central attention has to be paid to the properties of dose measuring devices (dosemeters)***

GENERAL REQUIREMENTS FOR DOSEMETERS

- High accuracy and precision;
- Linearity of signal with dose over a wide range;
- Small dose and dose rate dependence;
- Flat energy response;
- Small directional dependence;
- High spatial resolution;
- Large dynamic range;
- Stability of dosemeter response;
- High detection efficiency;
- Etc…..

DOSEMETER'S PROPERTIES

Linearity: the dosemeter reading should be linearly proportional to the dosimetric quantity.



Case A:

- Linearity;
- Supralinearity;
- Saturation;

Case B:

- Linearity;
- Saturation.

DOSEMETER'S PROPERTIES

Dose rate dependence

- M/D may be called the response of a dosimeter system
- When an integrated response is measured,

$$M = \int (M/D)(\mathrm{d}D/\mathrm{d}t)\,\mathrm{d}t$$

the dosimetric quantity should be independent of the dose rate dD/dt of the quantity.

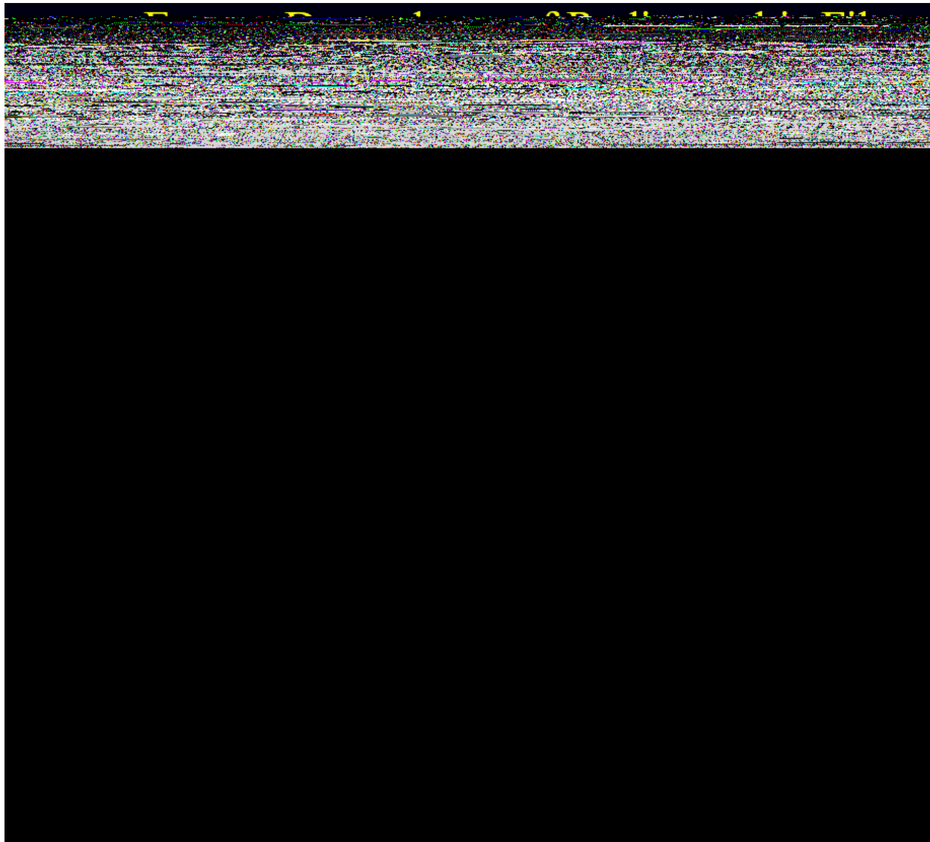
$$M = (M/D)\int (\mathrm{d}D/\mathrm{d}t)\,\mathrm{d}t$$

- Other formulation:
  The response M/D should be constant for different dose rates $(\mathrm{d}D/\mathrm{d}t)_1$ and $(\mathrm{d}D/\mathrm{d}t)_2$.

DOSEMETER‘S PROPERTIES

Energy dependence

- The response of a dosimetric system is generally a function of the radiation energy.
- Since calibration is done at a specified beam quality (=energy), a reading should generally be corrected if the user's beam quality is not identical to the calibration beam quality.



Energy dependence of film dosemeter

Directional dependence

- The variation in response as a function of the angle of the incidence of the radiation is called the **directional dependence** of a dosimeter.
- Due to construction details and physical size, dosimeters usually exhibit a certain directional dependence.

Spatial resolution and physical size

- The quantity absorbed dose is a point quantity
- Ideal measurement requires a point-like detector
- Examples that approximate a 'point' measurement are:
    - TLD;
    - film, gel, where the 'point' is defined by the resolution of the read-out system)
    - pin-point micro-chamber

DOSIMETRY METHODS

Dosimetry methods depend on *radiation induced processes* in *detector materials*:

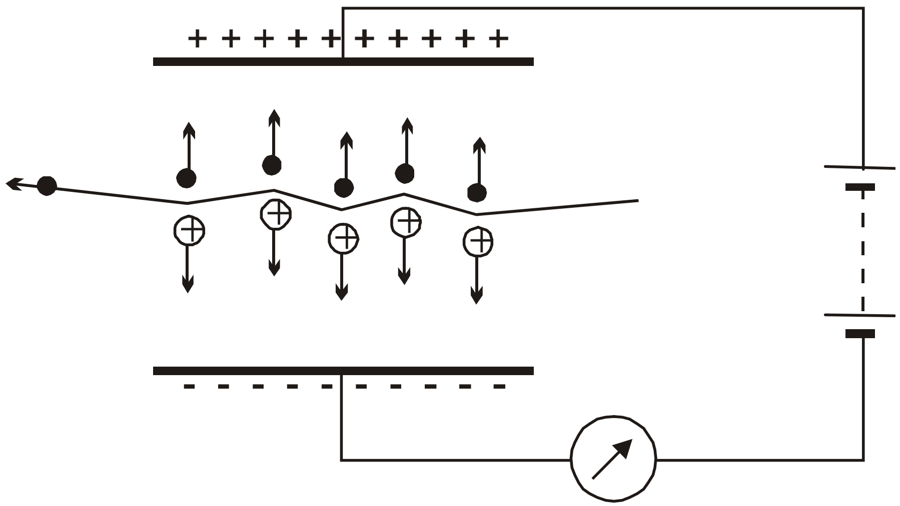
- Ionization (ionization chambers),
- Temperature change (calorimeters);
- Thermoluminescence (LiF);
- Colour change (perspex, radiochromic systems);
- Free radical concentration change (alanine);
- Conductivity change (ECB, alanine solution);
- Radiation chemical oxidation (Fricke);
- Radiation chemical reduction (dichromate, ceric-cerous);
- Optically stimulated luminescence (Al_2O_3, Sunna);
- Radiation defects in semiconductors (diodes, MOSFET (and many others);

Primary standard methods

- ***Calorimetry*** and ***ionization methods*** are considered primary standard methods in dosimetry;
- Both methods enable dose measurements in various radiation fields and are used for calibration of standard and routine dosemeters

Calorimetry is widely used in ***radiation processing of materials***, while ***ionization chambers*** – in ***medical applications*** of ionizing radiation

GAS FILLED DETECTORS



Operation scheme of gas filled detector

Ionization chamber is a detector consisting of a chamber filled with gas, in which the electric field provided for the collection of ions (charges) produced by ionizing radiation in the measuring volume of the detector, is insufficient to initiate gas multiplication

PRIMARY STANDARD: IONIZATION CHAMBERS

Free-air ionization chambers are the primary standard for air kerma in air for X rays (up to 300 kV):

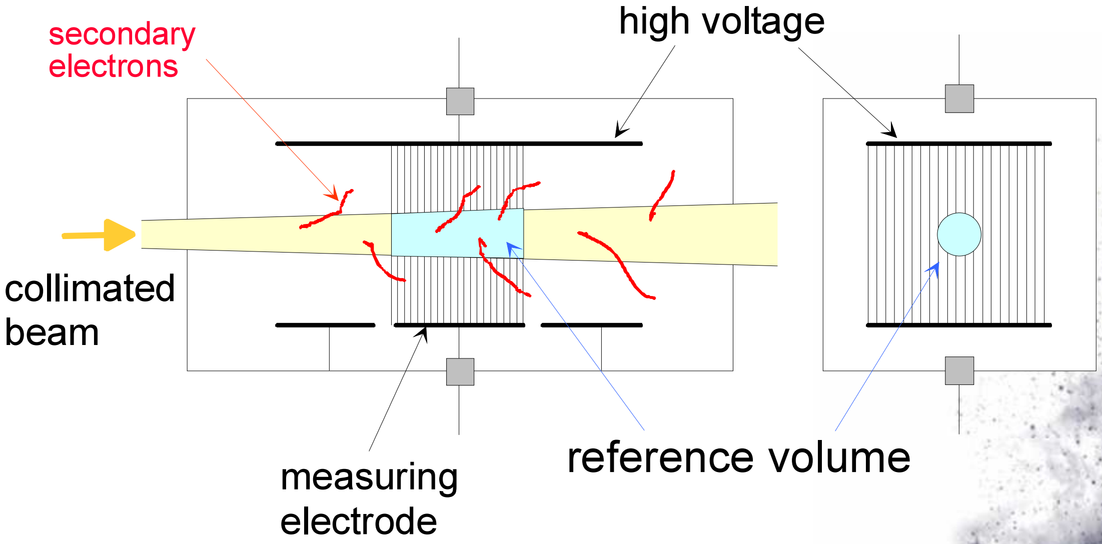
$$K_{air} = X \frac{W}{e} \frac{1}{1-g} = \frac{Q_{air}}{m_{air}} \frac{W}{e} \frac{1}{1-g}$$

W/e=33.97 eV

Principle:

The reference volume (blue) is defined by the collimation of the beam and by the size of the measuring electrode.

Secondary electron equilibrium
in air is fulfilled.



Free-air ionisation chamber is a special instrument used in Primary Standard Dosimetry Laboratories.

It is called a free-air chamber because, in principle, the walls of the chamber do not play any role in its response.

PRIMARY STANDARD – FREE AIR CHAMBER

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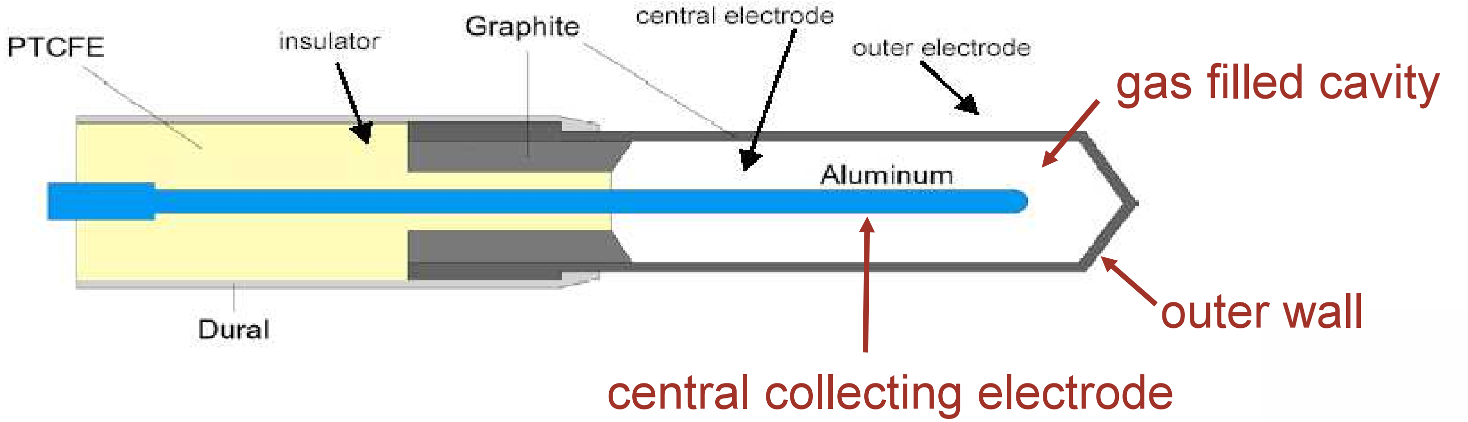
Uncertainty

Medium Energy Free-Air Chamber (MEES)	Standard Uncertainty	
	Type A	Type B
Ionization Current	0.03	0.03
Volume	0.01	0.04
Positioning	0.02	0.01
Correction Factors (excl. k_h)	0.02	0.2
Humidity k_h	0.01	0.03
Physical Constants	-	0.15
Air-Kerma Rate	**0.044**	**0.257**
Combined Uncertainty	**0.26**	

PRIMARY STANDARD: IONIZATION CHAMBERS

- **Free-air ionization chambers** cannot function as a primary standard for ^{60}Co beams or high energy photons and electrons beams generated in medical accelerators , since the air column surrounding the sensitive volume (for establishing the electronic equilibrium condition in air) would become very long.
- Therefore at energies > 300 kV :
  - Graphite cavity ionization chambers with an accurately known chamber volume are used as the primary standard.
  - The use of the graphite cavity chamber is based on the Bragg–Gray cavity theory.

PRIMARY STANDARD: IONIZATION CHAMBER



Farmer type ionization chamber is used for the determination of the ***absorbed dose to water,*** D_w **,** which is the basic measurand in clinical operation at ***the primary standard level***

Note:

Ideally, the primary standard for absorbed dose to water should be a water calorimeter that would be an integral part of a water phantom and would measure the dose under reference conditions.

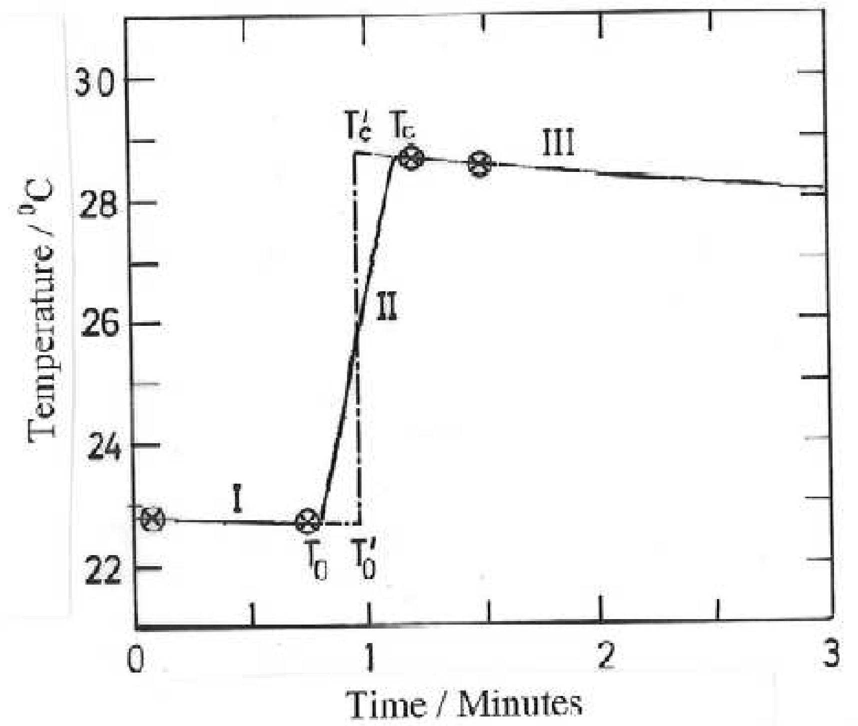
PRIMARY STANDARD: CALORIMETER

- ✓Calorimetry is the most fundamental method of realizing the primary standard for absorbed dose, since temperature rise is the most direct consequence of energy absorption in a medium.
- ✓Calorimetry is absolute method of dosimetry where almost all radiation energy absorbed in the thermally isolated mass is converted into heat which is measured.
- ✓ Measured energy per unit mass or ***the average dose*** to the medium assuming no heat loss is:

$$\overline{D} = \frac{h \cdot \Delta T}{1-\delta}$$

h is specific heat capacity of the medium; **δ** is the thermal defect (this small fraction of the energy that does not appear eventually as thermal energy – because of chemical reaction)

CALORIMETRY PRINCIPLE



NOTE—Regions I, II, and III are before, during and after irradiation, respectively. The temperature curves were extrapolated from T_o and T_c to the midpoint of the irradiation time at T_o' and T_c', respectively. $\Delta T = T_c' - T_o'$ is used for dose calculation.

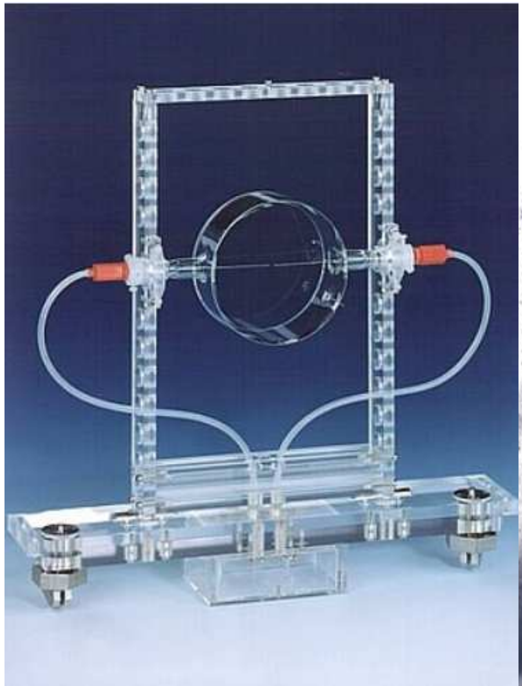
Figure is taken from H. Attix, 1986

PRIMARY STANDARD: WATER CALORIMETER
(gamma, proton, neutron beams)

Temperature- calibrated thermistors which are fused into the tip of thin, tapered glass pipettes are used for radiation induced temperature increase measurement

PTB water calorimeter

Calorimeter construction consists of cubic water phantom coated by polystyrene (due to operational temperature of 4°C) and calorimetric detector filled with highly purified water



Calorimetric detector

PRIMARY STANDARD: GRAPHITE CALORIMETER

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- The graphite calorimeter is used by several PSDLs as a primary standard to determine the absorbed dose to graphite in a graphite phantom.
- Graphite is in general an ideal material for calorimetry, since it is of low atomic number *Z* and all the absorbed energy reappears as heat, without any loss of heat in other mechanisms (such as the heat defect).
- The specific heat capacity in graphite is 7.1×10^2 J/kg°C, 710 Gy!!! will be required for the increasing the temperature by 1°C.
- Sensitive measurement devices are needed: thermistors are small and can measure a temperature in the order of a few μ°C

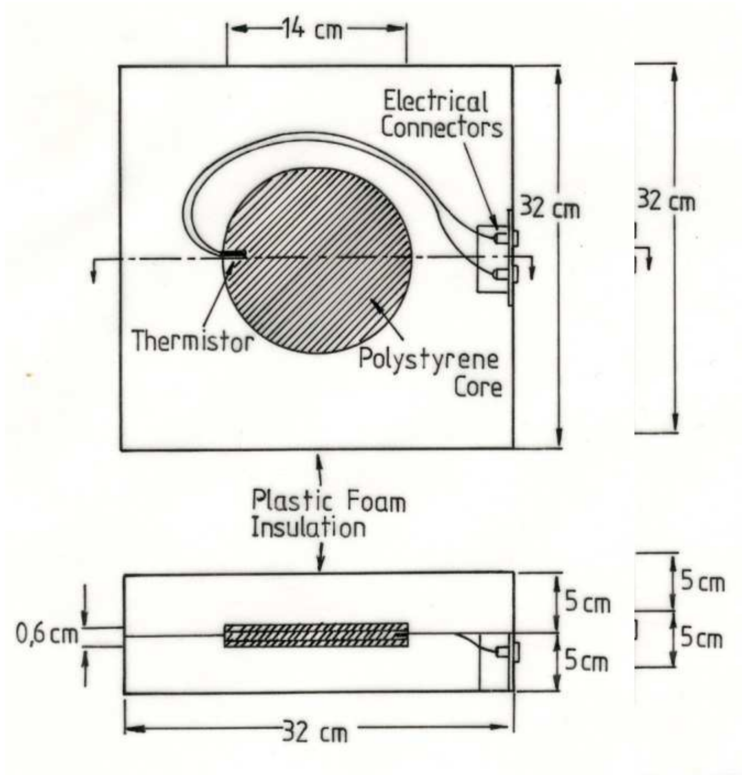
PROCESS CALORIMETERS

- The process calorimeters are classified as **Type II dosimeters** (ASTM/E2628).
- Process calorimeters may be used as internal standards at an electron beam irradiation facility, including being used as **transfer standard dosimetry systems** for calibration of other dosimetry systems, or they may be used as routine dosimeters.
- Two types of calorimeter are used in radiation dosimetry: total energy absorption calorimeters (e.g. to determine the energy or power of a particle beam) and thin calorimeters that are partially absorbent and are used to measure absorbed dose.
- Semi-adiabatic calorimeters have been designed for dosimetry at high energy electron accelerators (1–10 MeV) both for calibration and for routine process control and also for low energies between 100–500 keV.

PROCESS CALORIMETERS

Three types of calorimeters are used :
graphite, water and polystyrene

- A typical process calorimeter is a disc of material (graphite, polystyrene) or a sealed polystyrene Petri dish filled with water, which is placed in a thermally-insulating material such as foamed plastic.
- A calibrated thermistor or thermocouple is embedded inside the disc or placed through the side of the dish into the water.
- The advantage of using graphite instead of water is the lack of thermal defects. Graphite calorimeters can measure lower doses (1.5–15 kGy). Graphite calorimeters are used for calibration purposes



Calorimeter design (INTERNATIONAL **ISO/ASTM** STANDARD **DIS 51631**

PROCESS CALORIMETERS

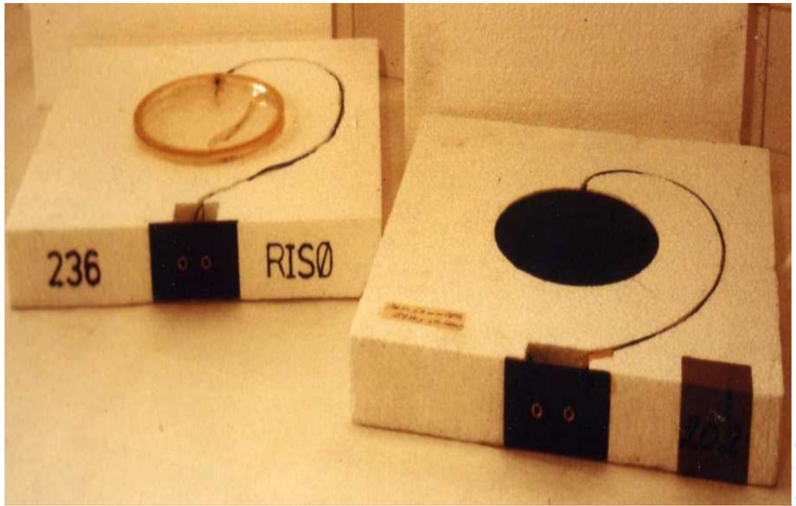
4 – 10 MeV:

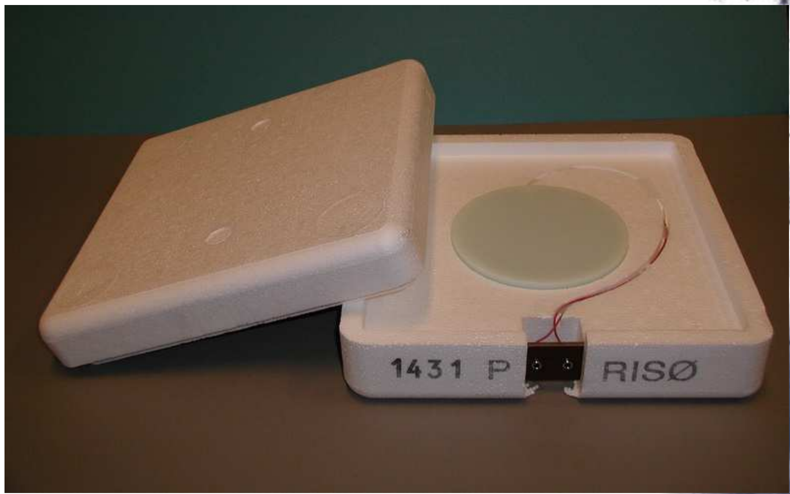
- graphite, water, polystyrene calorimeters (1.5 – 60 kGy);
- Calibration, nominal dose measurements;
- Reproducibility: less than 1%
- Sensitivity (approx):

 - water calorimeter - 3.4 kGy /°C;

 - polystyrene calorimeter - 1.4 kGy /°C;

 - graphite calorimeter - 0.75 kGy /°C.





For use at electron accelerators

PROCESS CALORIMETERS

- **1.5 – 4 MeV:**

  PS calorimeter

  - calibration,
  - nominal dose determination

Ongoing development

- **80 – 120 keV:**

  Graphite calorimeter

  - primary standard system;
  - calibration;

TABLE 2 Measurement uncertainties of routine polystyrene calorimetric dosimetry systems from Risø high dose reference laboratory (in percent, at *k* = 2) (9)

NOTE—At doses higher than 10 kGy, (2) and (3) are reduced to 0.2 %.

	Sources of Uncertainty	Type B	Type A
1	Calibration		3.2
2	Temperature measurement of calorimeter (at 3 kGy)	1.0	
3	Temperature extrapolation of calorimeter (at 3 kGy)	1.0	
4	Change of temperature sensitivity of specific heat of polystyrene		0.5
5	Heating effects	0.5	
	Quadratic sum	1.5	3.2
	Combined	3.6 %	

PRIMARY STANDARDS: Chemical dosimetry standard for absorbed dose to water

- In chemical dosimetry systems the dose is determined by measuring the chemical change produced by radiation in the sensitive volume of the dosimeter.
- The most widely used chemical dosimetry standard is **Fricke dosimeter**

Solution is sensitive to UV radiation and heat.

FRICKE DOSEMETERS

- The **Fricke dosimeter** is a solution of the following composition in water:
  - 1 mM/dm^3 $FeSO_4$ $(7H_2O)$ or $Fe(NH_4)_2(SO_4)_2$ $(6H_2O)$
  - plus 0.4 M/dm^3 H_2SO_4 , air saturated
  - plus 1 mM NaCl
- Irradiation of a Fricke solution oxidizes ferrous ions Fe^{2+} into ferric ions Fe^{3+}
- ferric ions Fe^{3+} exhibit a strong absorption peak at a wave-length 304 nm, whereas ferrous ions Fe^{2+} do not show any absorption at this wavelength.

FRICKE DOSEMETERS

When the solution is irradiated, water decomposition occurs and hydrogen atoms produced react with oxygen to produce the hydroperoxy radical:

$$H\bullet + O_2 \rightarrow HO_2\bullet \qquad (1)$$

Various reactions subsequently lead to the conversion of ferrous to ferric ions:

$$Fe^{2+} + OH\bullet \rightarrow Fe^{3+} + OHFe^{2+} + HO_2\bullet \rightarrow Fe^{3+} + HO^{2-}\ ; \qquad (2)$$

$$HO^{2-} + H_3O+ \rightarrow H_2O_2 + H_2O;$$

$$and\ Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH\bullet + OH.$$

The quantity of Fe^{3+} produced depends on the energy absorbed by the solution.

FRICKE DOSEMETERS

- The Fricke dosimeter response is expressed in terms of its sensitivity, known as the

radiation chemical yield, G value

- The G value is defined as the number of moles of ferric ions produced per joule of the energy absorbed in the solution.
- Specifically, the change in ferric ion concentration is related to the radiation dose (energy per unit mass)

FRICKE DOSEMETERS

- The average dose to Fricke solution is given by a change in optical density at 304 nm, ΔOD :

$$\overline{D_F} = \frac{\Delta OD}{\varepsilon G \rho L}$$

where ε is the molar extinction coefficient (217.4 $lmol^{-1}cm^{-1}$ at 25^oC), G is the yield of ferric ions (1.617x10^{-6} $molJ^{-1}$), ρ is the density of Fricke solution (1.023 kgl^{-1} at 25^oC), L is the path length over which the optical signal was red (typically 2-4 cm).

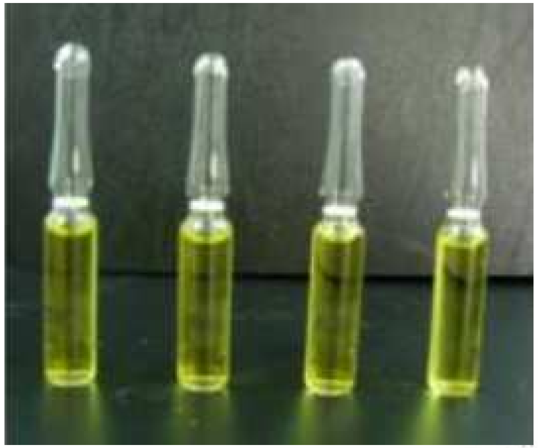
- System response is nearly independent of the photon and electron energy in the range of 5-16 MeV;
- Absorbed dose range : 40-400 Gy;
- Reproducibility: 1-2%.

CHEMICAL DOSEMETERS:
Ceric sulphate (ceric-cerous)

- **Dose range: 1–200 kGy.**

- **Radiolytic reduction** of ceric ions to cerous ions in an aqueous acidic solution;

- **Concentration** of ceric sulphate
  (or ceric ammonium sulphate):
  2×10^{-4} mol/dm^3 - 5×10^{-2} mol/dm^3
  in an aqueous solution containing
  0.4 mol/dm^3 sulphuric acid.



Usually used in radiation sterilization and food irradiation applications

CHEMICAL DOSEMETERS:
Ceric sulphate (ceric-cerous)

The evaluation: **spectrophotometry or potentiometry**.

Spectrophotometry

- Change of absorbance of the ceric ions at 320 nm (approximately linear with the dose);
- The molar linear absorption coefficient for the ceric ions is 561 m^2/mol
- Reproducibility: +/- 3 – 5 %;

Redox potential measurement

- Doses within the range 0.5-5 kGy and 5-50kGy

CHEMICAL DOSEMETERS:
Ceric sulphate (ceric-cerous)

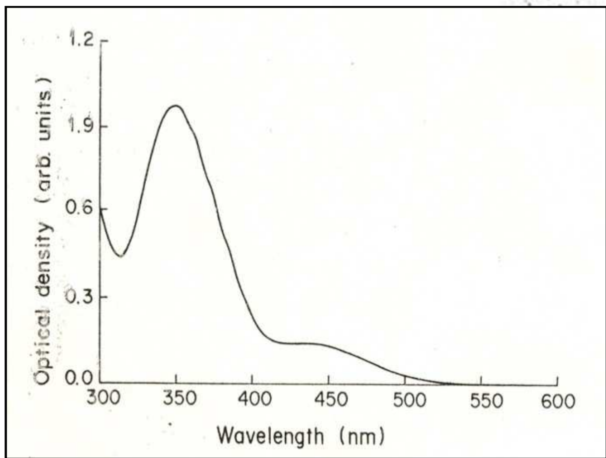
- ***Radiolytic reduction*** of the dichromate ion $(Cr_2O_7)^{2-}$ to a chromic ion in aqueous perchloric acid solution :

2×10^{-3} mol/dm^3 K_2Cr2O_7 and 5×10^{-4} mol/dm^3 Ag_2Cr_2O
in 0.1 mol/dm^3 perchloric acid.

- **Color change** (decrease of dichromate ion concentration) at 440 nm**;**
- **Dose range: 10 – 50 kGy;**
- **Reproducibility < 0.5 %;**
- **Measurement of Low doses**

  (down to 2 kGy at 350 nm)

  5×10^{-4} mol/dm^3 $Ag_2Cr_2O_7$ in 0.1 mol/dm^3 perchloric acid.



ETHANOL-MONOCHLOROBENZENE DOSIMETER (ISO/ASTM 51538)

- **EBC** contains monochlorobenzene (C_6H_5Cl) in an aerated ethanol–water solution.
- The concentration of monochlorobenzene may vary between 4 and 40 vol. % upon request.
- In ***radiation processing*** practice a solution containing 24 vol. % of monochlorobenzene is used.
- **Principle:** the formation of hydrochloric acid (HCl) upon irradiation via dissociative electron attachment, since monochlorobenzene, as a good electron scavenger.
- **Dose range:** 0.05-100kGy.
- Nearly independent of irradiation temperature.

ETHANOL-MONOCHLOROBENZENE DOSIMETER (ISO/ASTM 51538)

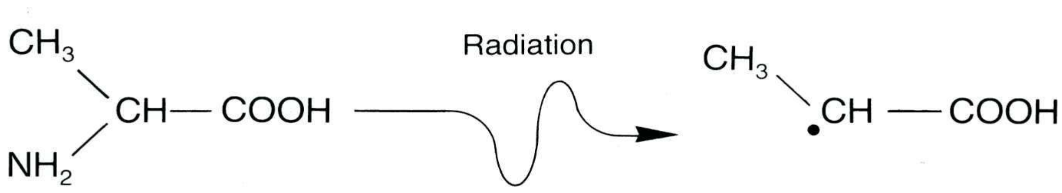
Dose evaluation:

The mercurimetric method to determine the concentration of chloride ions.

- the addition of ferric nitrate and mercuric thiocyanate to the irradiated ethanol-monochlorobenzene solution is required;
- The radiolytically generated Cl– ions react with the mercury(II) thiocyanate,
- the liberated thiocyanate ions react with ferric ions and produce the red coloured ferric thiocyanate complex, which has an absorbtion peak at 485 nm.

ALANINE DOSIMETRY (ISO/ASTM 51607)

- An ***alanine dosimeter*** is an ***amino acid*** that forms stable free radicals when irradiated.



- Dose range: 10 Gy – 100 kGy;
- Reproducibility < 0.5 %;
- The response depends on environmental conditions (humidity, temperature)
- Alanin is tissue equivalent

Used in both: medical and industrial applications

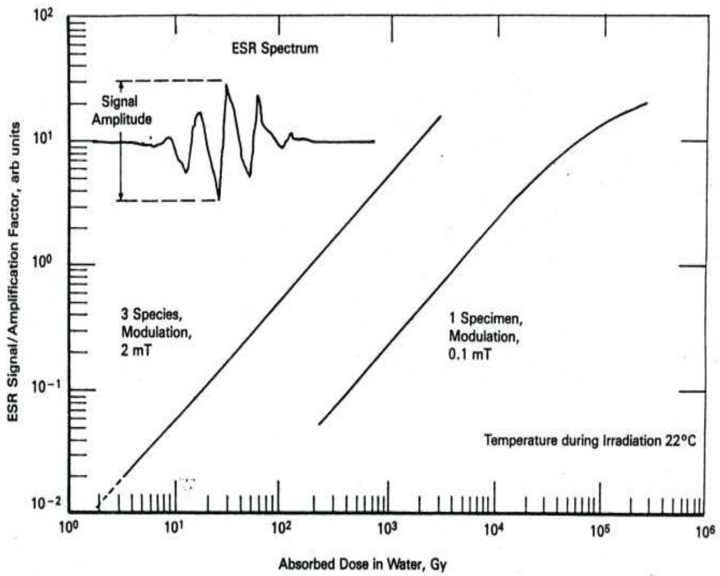
ALANINE DOSIMETRY (ISO/ASTM 51607)

Evaluation principle :
The concentration of the radicals is measured using an electron paramagnetic resonance (EPR) spectroscopy and is proportional to absorbed dose:

- In magnetic field unpaired electrons are split into two discrete energy levels;
- Separation between the levels is given by electron – spin factor g:

$$\Delta E = \frac{e\hbar}{2m_e} gB$$

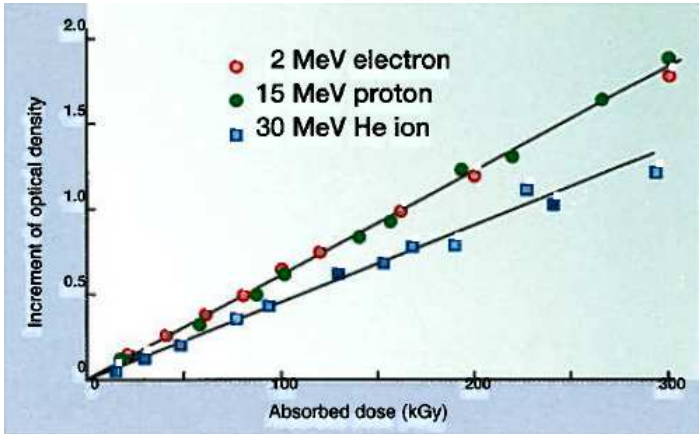
- The intensity is measured as the peak to peak height of the central line in the EPR spectrum.
- The readout is non-destructive.



CELLULOSE TRIACETATE FILM (CTA)

CTA dosimeter, FTR-125:

- Used for routine dosimetry,
- **Composition**: cellulose triacetate, triphenyl phosphate;
- **Dose range:** 5-300kGy (nearly linear);
- Radiation induced **absorbance change** at 280 nm,
- **Response**: 5%, lower for electron beams due to O_2 diffusion

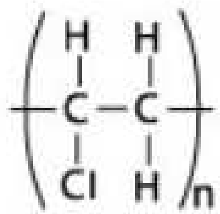


The film is used : in electron beam, gamma-ray and ion beam irradiations; at electron irradiation facilities - mainly for dose mapping

POLYVINYL CHLORIDE FILM

The colourless polyvinyl chloride (PVC) foils

- Radiation induces formation of unsattured chemical bonds;
- New species correspond to the absorbance changes at 395 nm;
- Dose range of 0.5 – 60 kGy



Polyvinyl Chloride

Polymer

Note

PVC films **are not considered** for the use as **dosemeters**, but only as **dose indicators** at electron accelerators to monitor the irradiation process and the accelerator parameters (scan width, beam spot, etc.) due to the significant influence of various factors (environmental effects on the response, dose rate ffects, batch-to-batch variation, etc.) The irradiated films have to be heat treated (60°C, 20 min) after irradiation in order to stabilize the post-irradiation response.

POLYMETHYLMETHACRYLATE (PERSPEX) DOSIMETERS (ISO/ASTM 51276)

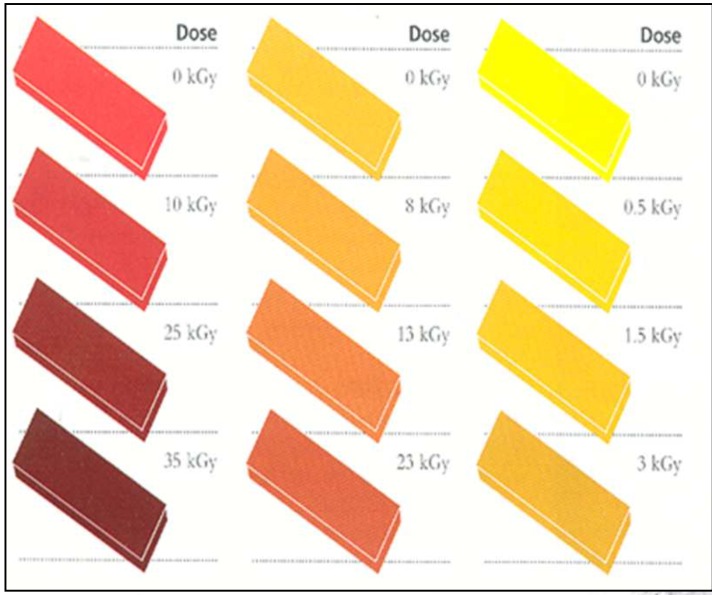
Dye containing polymethylmethacrylate dosimeters:

• *red Perspex, amber Perspex and the Gammachrome YR system*

Colour changes due to irradiation;

Dose range:

- *red Perspex*, 5-50 kGy, at 640 nm;
- *Amber Perspex*, 3-15 kGy, at 603 nm or 651 nm;
- *Gammachrome YR system,* 0.1-3 kGy, at 530 nm (for food irradiation mainly);



Reproducibility < 3 %;

Post irradiation change of signal

Used in gamma radiation processing

RADIOCHROMIC FILMS

Radiochromic film is a new type of self-developing film, containing a special dye that is polymerized and develops film specific color upon exposure to radiation.

Advantages:

- No quality control on film processing needed;
- Radio-chromic film is grainless $\Rightarrow$ very high resolution;
- Dose rate independendent;
- Radiochromic type GafChromic film, has nearly tissue equivalent composition (9.0% hydrogen, 60.6% carbon, 11.2% nitrogen and 19.2% oxygen) $\Rightarrow$ very important in medical applications.

Disadvantage:

GafChromic films are generally less sensitive than radiographic films

Principle:

Similarly to the radiographic film, the radiochromic film dose response is determined with a suitable densitometer.

RADIOCHROMIC FILMS: FTW -60 DOSEMETER (ISO/ASTM 51275)

FTW film

- Colourless film containing hexa(hydroxyethyl) pararosaniline cyanide in a nylon matrix;
- Radiation induced **colour change** to **deep blue**;
- **Dose range**:

3-30 kGy, if spectrophotometric measurement is carried out at 605 nm

30-150 kGy, if spectrophotometric measurement is carried out at 510 nm

- **Film response** is independent of the energy and type of the radiation (electron, gamma or X ray radiation) and of the dose rate up to about 10^{13} Gy/s

Used for process control for gamma as well as for low and high energy electron irradiation.

RADIOCHROMIC FILMS: B3 (GEX) FILM (ISO/ASTM 51275)

B3 (GEX) film

- Colourless Polyvinyl butyral film containing the leucocyanide of pararosaniline;
- Radiation induced colour change to pink;
- Dose range: 2–100 kGy, measured at 554 nm.
- Widely used in gamma and electron beam radiation processing.

RADIOCHROMIC FILMS: B3 (GEX) FILM (ISO/ASTM 51275)

B3 (GEX) film

- Some other versions of the same film are avalable (e.g. film is provided with ***adhesive backing and a UV protective cover;*** used for reflected light measurement with the potential for label dosimetry applications).

- Application in electron dose mapping has unique prospects, due to a small thickness.

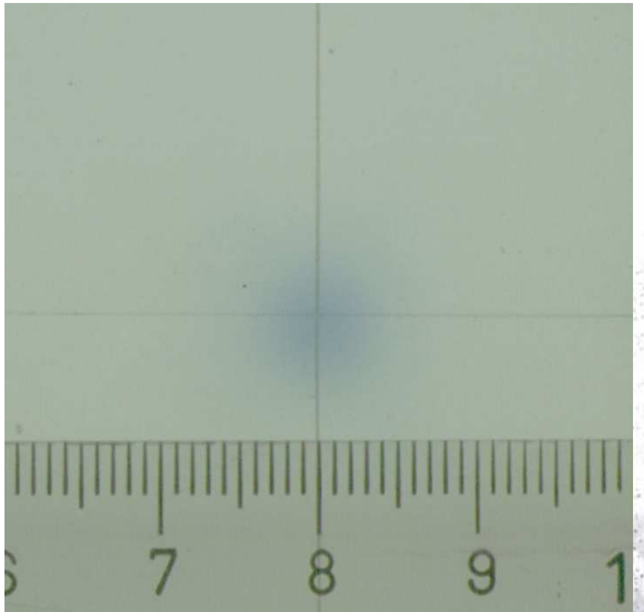
- New perspectives with a new software development at Risø National Laboratory for the scanning and evaluation of images on films used for example in dose distribution measurements.

RADIOCHROMIC FILMS:
GAFCHROMIC FILM (ISO/ASTM 51275)

Gafromic film

- Radiochromic film consisting of colourless transparent coatings of polycrystalline substituted diacetylene sensor layers on a clear polyester base.

- Radiation induced colour change to ***deep blue***
- Dose range: 1 Gy - 40 kGy, read out at different wavelengths (670, 633, 600, 500 and 400 nm) depending on the absorbed dose



Applicable in medicine and industrial radiation processing and food irradiation

RADIOCHROMIC FILMS: (ISO/ASTM 51275)

- **Spectrophotometric readout;**

	Gafchromic	GEX(B3)	FWT
Dose range, kGy:	3 – 150	3 – 150	0.001 - 40
Wavelength, nm:	554	510, 605	670, 633, 580, 400

- **Stability: heat treatment after irradiation; packaging (UV);**

SYSTEMS BASED ON OPTICAL ABSORPTION (TETRAZOLIUM SALTS)

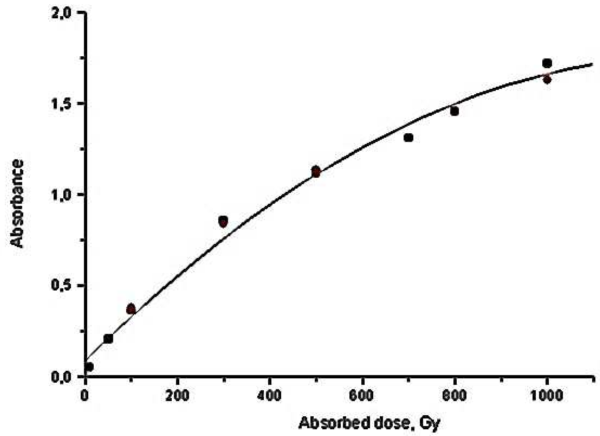
Tetrazolium salts :

Heterocyclic organic compounds, yielding highly coloured water insoluble formazans due to radiolytic reduction

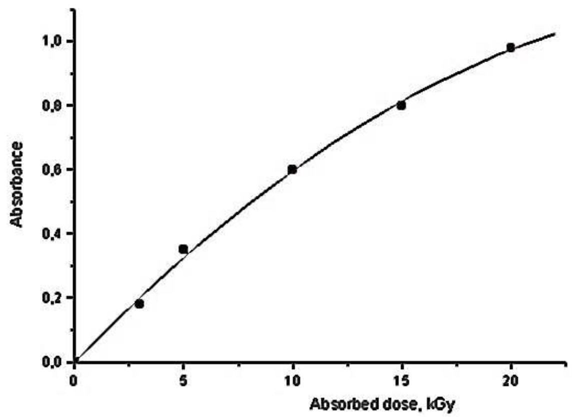
Compound / Product	$\lambda_{max.}$	Dose range:
• tetrazolium violet (TV)	525 nm	0.01 – 30 kGy
• tetrazolium red (TTC)	490 nm	0.01 – 100 kGy
• tetrazolium blue (TB)	520 nm	0.01 – 10 kGy
• nitro blue tetrazolium (NBT)	522 nm, 612 nm	0.01 – 25 kGy

DOSIMETRY APPLICATION OF NBT SOLUTION

- Dose dependence of monoformazan (522 nm) and diformazan (612 nm) radiolysis products formed in aqueous NBT solution



522 nm



612 nm

OSL: THE SUNNA DOSIMETER

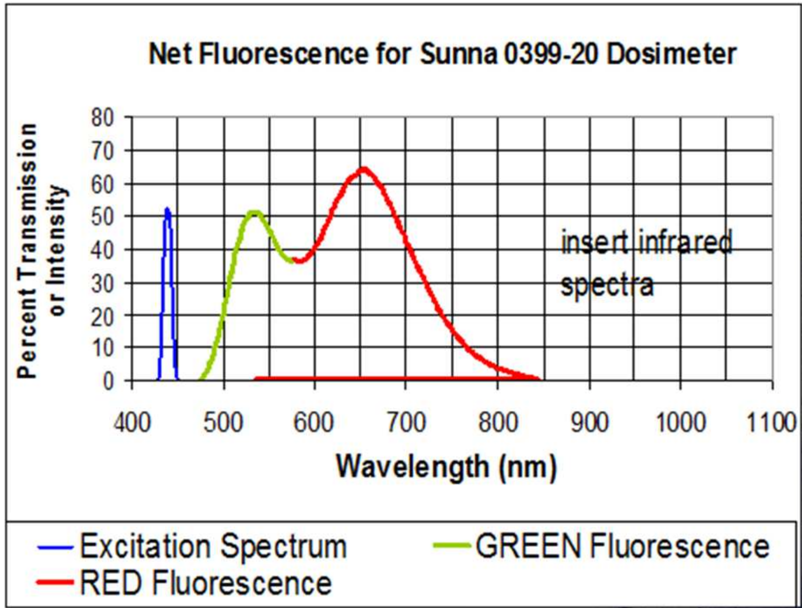
The Sunna dosimeter: LiF dispersed uniformly in PE matrix

- **Principle:**

➢ Formation of colour centers (F-, M-, N-, R centers) = (discrete optical absorption bands) in the near UV and visible spectrum due to irradiation;

➢ Excitation of the irradiated crystal with light at the wavelength of the colour centre absorption;

➢ Characteristic luminescence at a significantly higher wavelength.

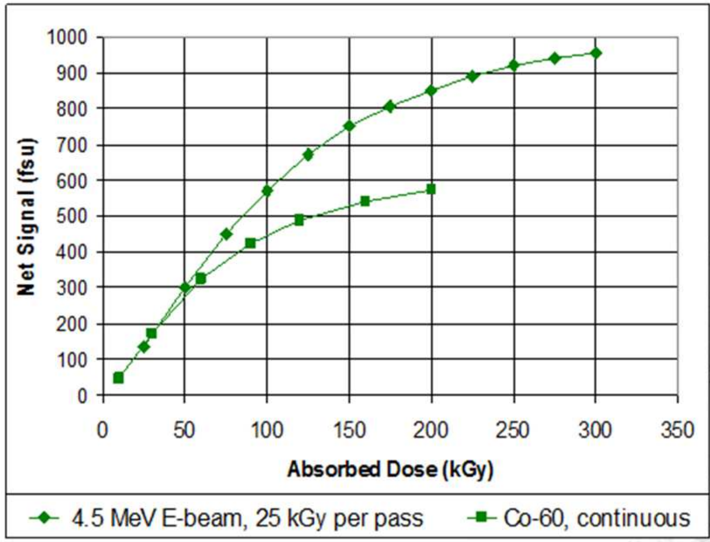


OSL: THE SUNNA DOSIMETER

- Red, green or IR OSL or UV absorption is used for dosimetry

Dose range:

- **5 – 100 kGy ,** evaluation of UV absorbance at 240 nm;
- **200 Gy – 250 kGy,** evaluation of green OSL at 530 nm;
- **10 Gy – 10 kGy,** evaluation of IR OSL at 670 nm and 1100 nm



The Sunna film is applied in both gamma and electron processing for dose distribution measurements, as well as for routine process control.

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 (comparing absorbed dose measurements)⇒dosimetry intercomparison exercise;
- These systems require calibration;
- Dosimetry systems:

  - alanine;

  - ethanol-chlorobenzene (ECB);

  - potassium dichromate;

  - ceric-cerous,

DOSIMETRY SYSTEMS IN RADIATION PROCESSING

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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 - Gex, Gafchromic, Sunna);***
  - ***- ECB, ceric-cerous solutions;***
  - ***- Process calorimeters (water, graphite, polystyrene);***

Dosimeter system	Method of analysis	Useful dose range, Gy	Nominal precision limits	References
Fricke solution	UV – spectro-photometry	$3x10 – 4x10^2$	1 %	ASTM E 1026 - 04
Ceric – cerous sulphate	UV – spectro-photometry	$10^3 – 10^6$	3 %	ISO/ASTM 51205
Potassium dichromate	UV-VIS spectrophoto	$5x10^3 – 4x10^4$	1 %	ISO/ASTM 51401
Ethanol-mono-chlorobenzene	Titration,or HF oscillometry	$4x10^2 –3x10^5$	3 %	ISO/ASTM 51538
L - alanine	EPR	$1 – 10^5$	0.5 %	ISO/ASTM 51607
Perspex systems	VIS - spectro-photometry	$10^3 – 5x10^4$	4 %	ISO/ASTM 51276
Suna film	OSL	$50-3x10^5$	3	ASTME2304
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
Cellulose triacetate	UV – spectro-photometry	$10^4 - 10^6$	3 %	ISO/ASTM 51650
Calorimetry	Resistance/ temperature	$1.5x10^3 – 5x10^4$	2 %	ISO/ASTM 51631

ENVIRONMENTAL EFFECTS ON DOSIMETRY SYSTEMS

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

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;

NEW APPROACHES – NOVEL DOSIMETRY SYSTEMS

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- **New type low energy calorimeters**
  0.08 – 0.12 MeV and 1.5 – 4 MeV systems

- **Systems based on conductivity analysis**
  Alanine solution, conducting plastics

- **Systems based on colour change**
  B3, FWT-60, GafChromic and Tetrazolium films

- **Systems based on fluorimetry analysis**
  - Sunna film (green and IR OSL; OD)

SYSTEMS BASED ON CONDUCTIVITY EVALUATION

- Aqueous – alanine solution (1 – 100 kGy)

- Polyaniline based polymer composites

  (5 – 150 kGy)

  (in research phase)

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:

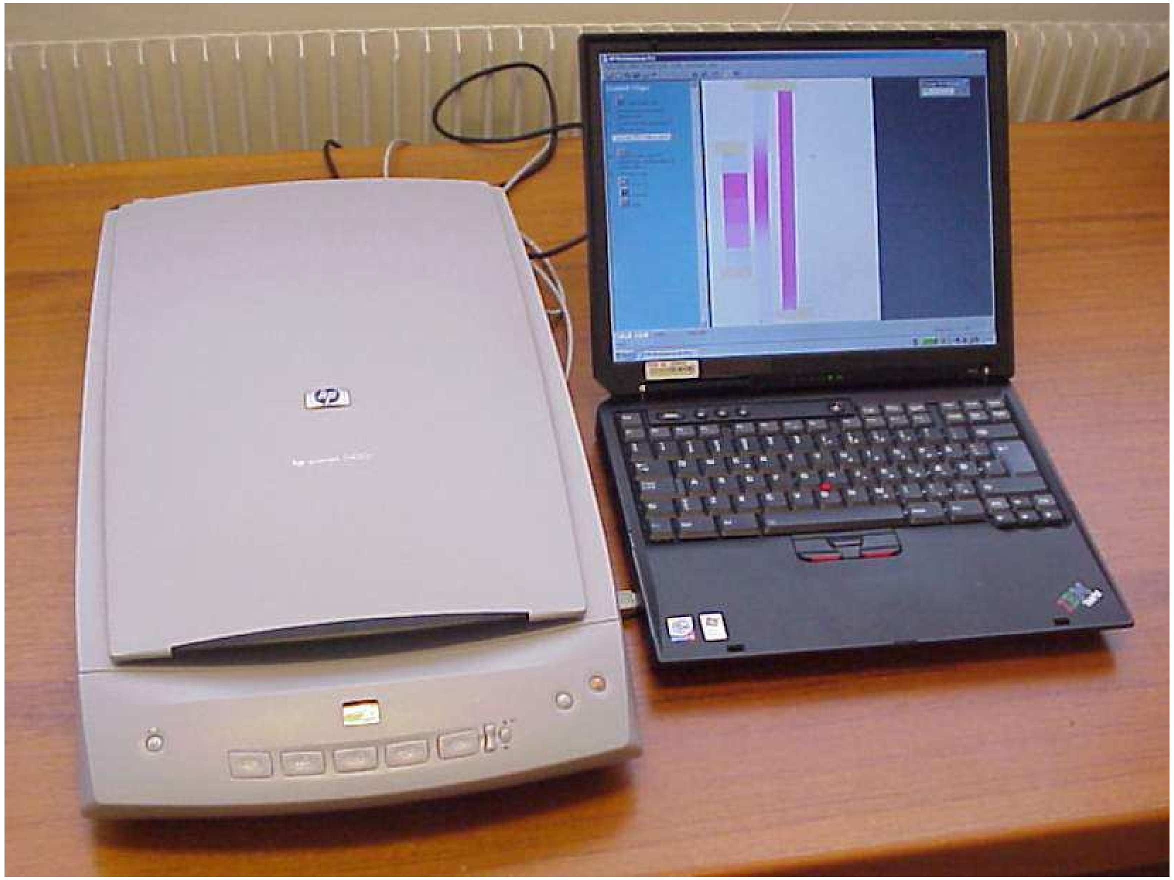
- Wide dynamic range;
- High sensitivity;
- Passive and real time dosimetry;
- Variable geometries;
- Inexpensive detectors;
- Multipurpose applications (medical diagnostic, radiation processing, radiation protection, space studies, etc);

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);

DOSE MAPPING: RISØSCAN



NOVEL DOSIMETRY SYSTEMS

SYSTEM	FORM	SENSOR COMPONENTS	METHOD OF ANALYSIS	USEFUL DOSE RANGE, kGy
Radiochromic poly-diacetylene films	coated film	substit. polydiactylene coated on polyester base	vis. spectrophot. or densitometry	0.001 – 50
Tetrazolium salt in polymer	thin film	tetrazolium chloride in polyvinyl alcohol	vis. spectrophot. or densitometry	1 – 50
Tetrazolium salt solution	aq. alcohol solution	tetrazolium chloride in solution	vis. spectrophot.	1 – 100
Bleachable dye solution	aq. alcohol solution	methylene blue, congo red, etc.	vis. spectrophot.	0.05 – 30
Optically stimulated luminescent film	photolumin. crystals in polymer	inorganic salts in polymer	spectro-fluorimetry	0.001 – 100
Polyethylene	plastic film	low- or high-density polyethylene	Fourier-transform IR spectrophot.	0.01 – 1000
Inorganic crystals	crystalline powder in polymer	SiO_2, Al_2O_3, $CaSO_4$:Dy	EPR spectrometry	0.01 – 10,000
p-FET or bipolar transistors	semiconductor transistor chips	silicon-base devices	electrical load (voltage) signal	0.1 – 60
MOSFET transistors	semiconductor transistor chips	silicon or GaAs devices	electrical load (voltage) signal	0.001- 1000
Diamond	small chip or sintered film	diamond (carbon) crystals	electrical load (current) signals	dose-rate meas. up to 10^3 Gy/min

PERSONAL DOSIMETRY

A big variety of different dosimeters:

- Film dosimeter
- TLD
- OSL
- Semiconductor devices

LUMINESCENCE DOSIMETRY

- There are two types of luminescence:
  - ***fluorescence***
  - ***phosphorescence***

- The difference depends on the **time delay** between the stimulation and the emission of light:

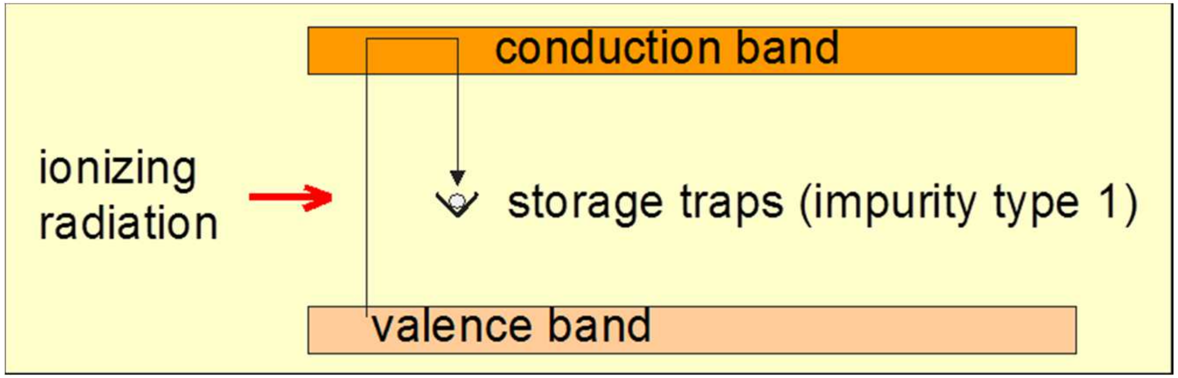
Fluorescence has a time delay between 10^{-10} to 10^{-8} s

Phosphorescence has a time delay exceeding 10^{-8} s

LUMINESCENCE DOSIMETRY

Principle:

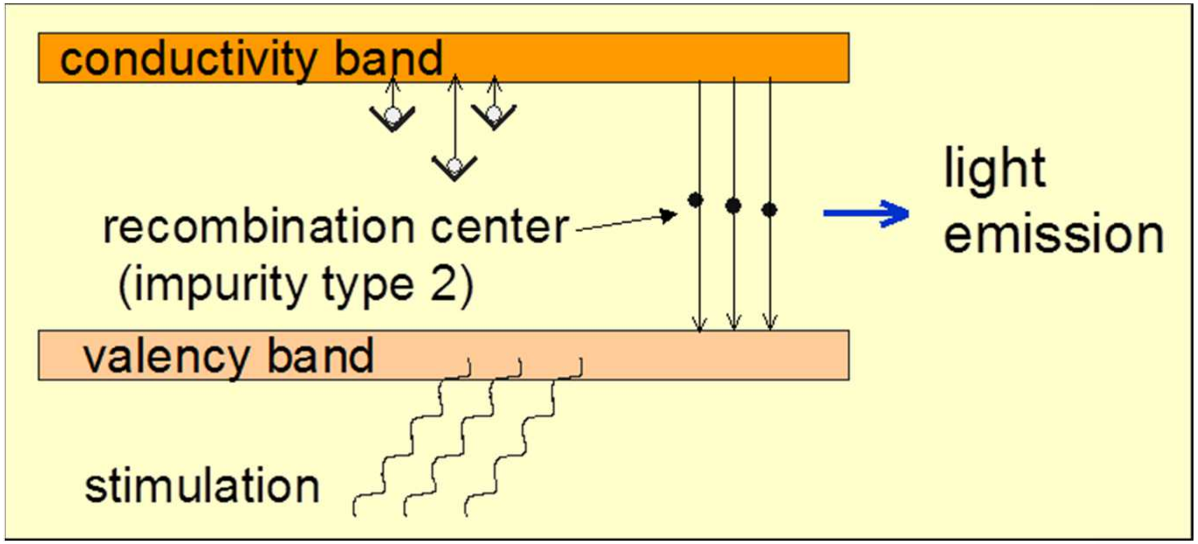
- Upon radiation, free electrons and holes are produced



- In a luminescence material, there are so-called *storage traps*
- Free electrons and holes will either recombine immediately or become trapped (at any energy between valence and conduction band)

Principle (cont.)

- Upon stimulation, the probability increases for the electrons to be raised to the conduction band …
- and to release energy (light) when they combine with a positive hole (needs an impurity of type 2)



LUMINESCENCE DOSIMETRY

- The process of luminescence can be accelerated with a suitable excitation in the form of heat or light.
- If the exciting agent is **heat**, the phenomenon is known as

  thermoluminescence

- When used for purposes of dosimetry, the material is called a
  - thermoluminescent (**TL**) material
  - or a thermoluminescent dosimeter (**TLD**).

LUMINESCENCE DOSIMETRY

- The process of luminecence can be accelerated with a suitable excitation in the form of heat or light.

- If the exciting agent is **light**, the phenomenon is referred to as

***optically stimulated luminescence* (OSL)**

THERMOLUMINESCENT DOSIMETER SYSTEMS

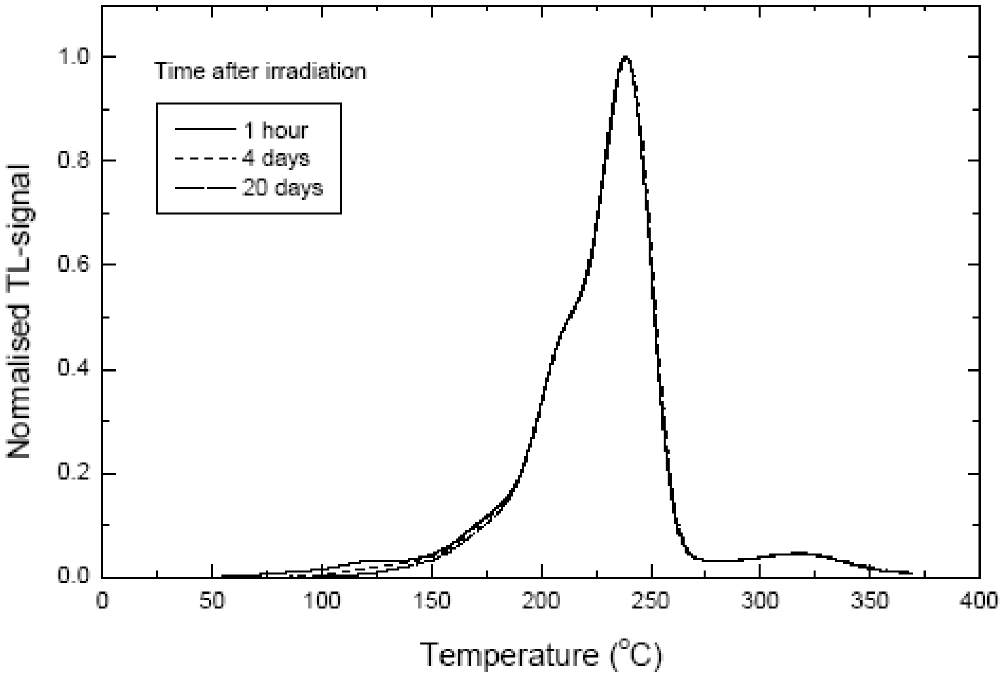
- TL dosimeters most commonly used in medical applications are (because of their tissue equivalence):
  - LiF:Mg,Ti
  - LiF:Mg,Cu,P
  - $Li_2B_4O_7$:Mn
- Other TLDs are (because of their high sensitivity):
  - $CaSO_4$:Dy
  - Al_2O_3:C
  - CaF_2:Mn
- TLDs are available in various forms (*e.g*., powder, chip, rod, ribbon, etc.).

Before use, TLDs have to be annealed to erase any residual signal.

THERMOLUMINESCENT DOSIMETER SYSTEMS

The TL intensity emission is a function of the TLD temperature *T*

TLD glow curve
or thermogram



Keeping the heating rate constant makes the temperature *T* proportional to time *t* and so the TL intensity can be plotted as a function of *t*.

THERMOLUMINESCENT DOSIMETER SYSTEMS

- The main dosimetric peak of the LiF:Mg,Ti glow curve is between 180° and 260°C; this peak is used for dosimetry.
- TL dose response is linear over a wide range of doses used in radiotherapy, however:
  - In higher dose region it increases exhibiting supralinear behaviour;
  - at even higher doses it saturates'
- To derive the absorbed dose from the TL-reading after calibration, correction factors have to be applied:
  - energy correction
  - fading
  - dose-response non-linearity corrections

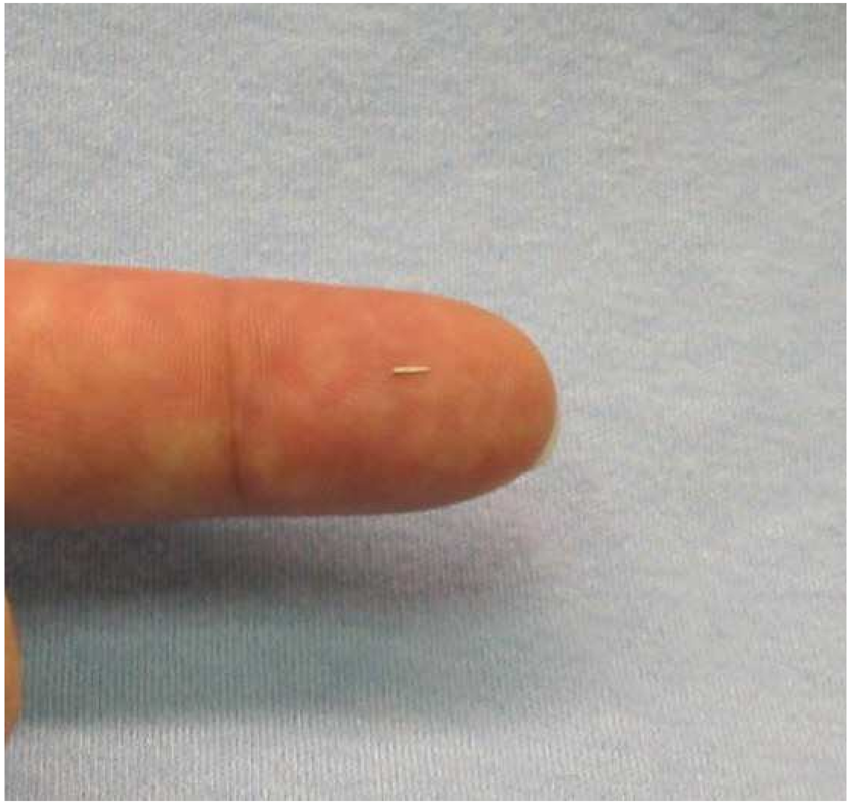
THERMOLUMINESCENT DOSIMETRY SYSTEM

OPTICALLY STIMULATED LUMINESCENCE SYSTEMS

- Optically-stimulated luminescence (OSL) is based on a principle similar to that of the TLD. Instead of heat, light (from a laser) is used to release the trapped energy in the form of luminescence.
- OSL is a novel technique offering a potential for *in vivo* dosimetry in radiotherapy.
- A further novel development is based on the excitation by a pulsed laser (POSL)
- The most promising material is Al_2O_3:C
- To produce OSL, the chip is excited with a laser light through an optical fiber and the resulting luminescence (blue light) is carried back in the same fiber, reflected through a 90° by a beam-splitter and measured in a photomultiplier tube.

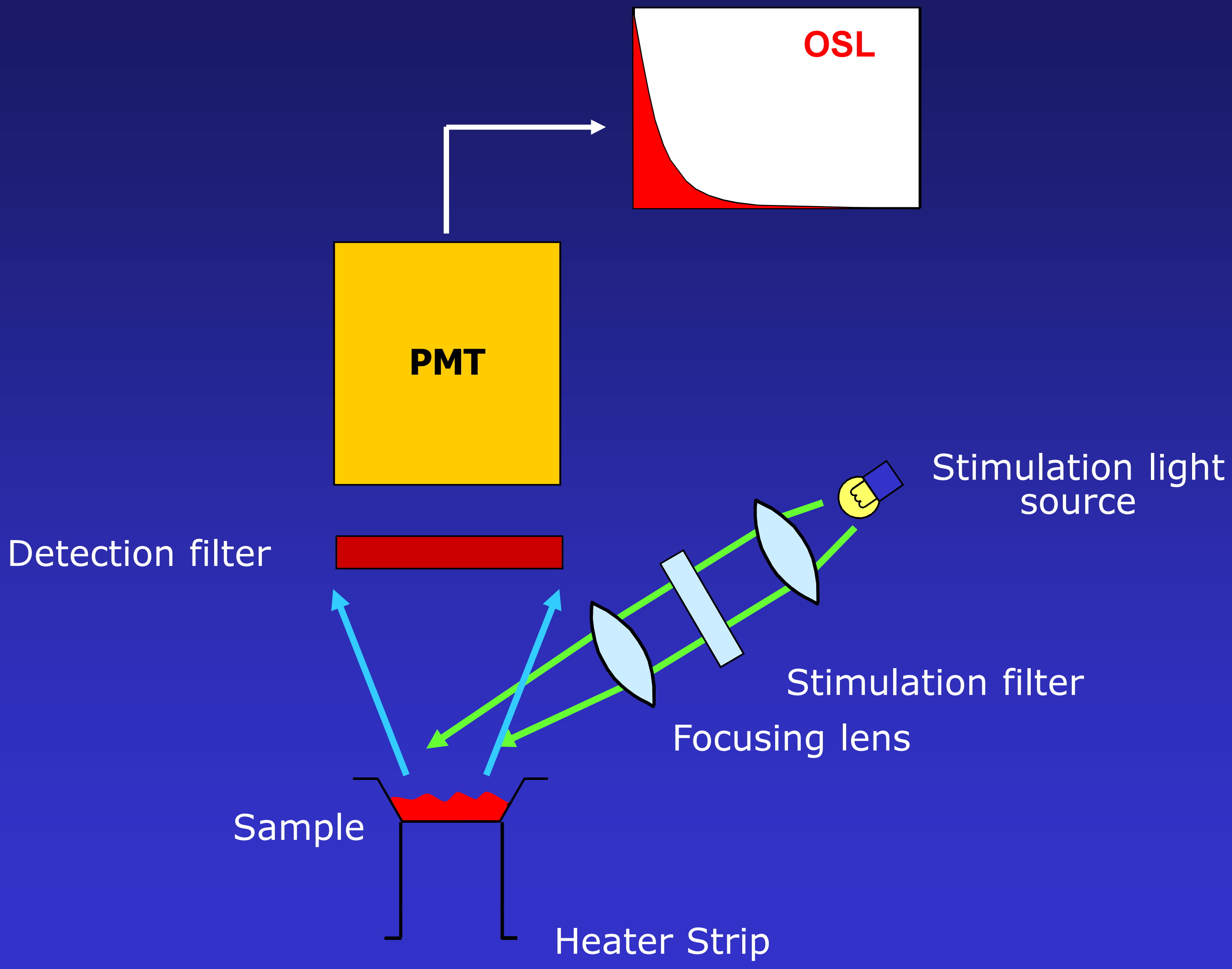
OPTICALLY STIMULATED LUMINESCENCE SYSTEMS

Crystal: 0.4 mm x 3 mm

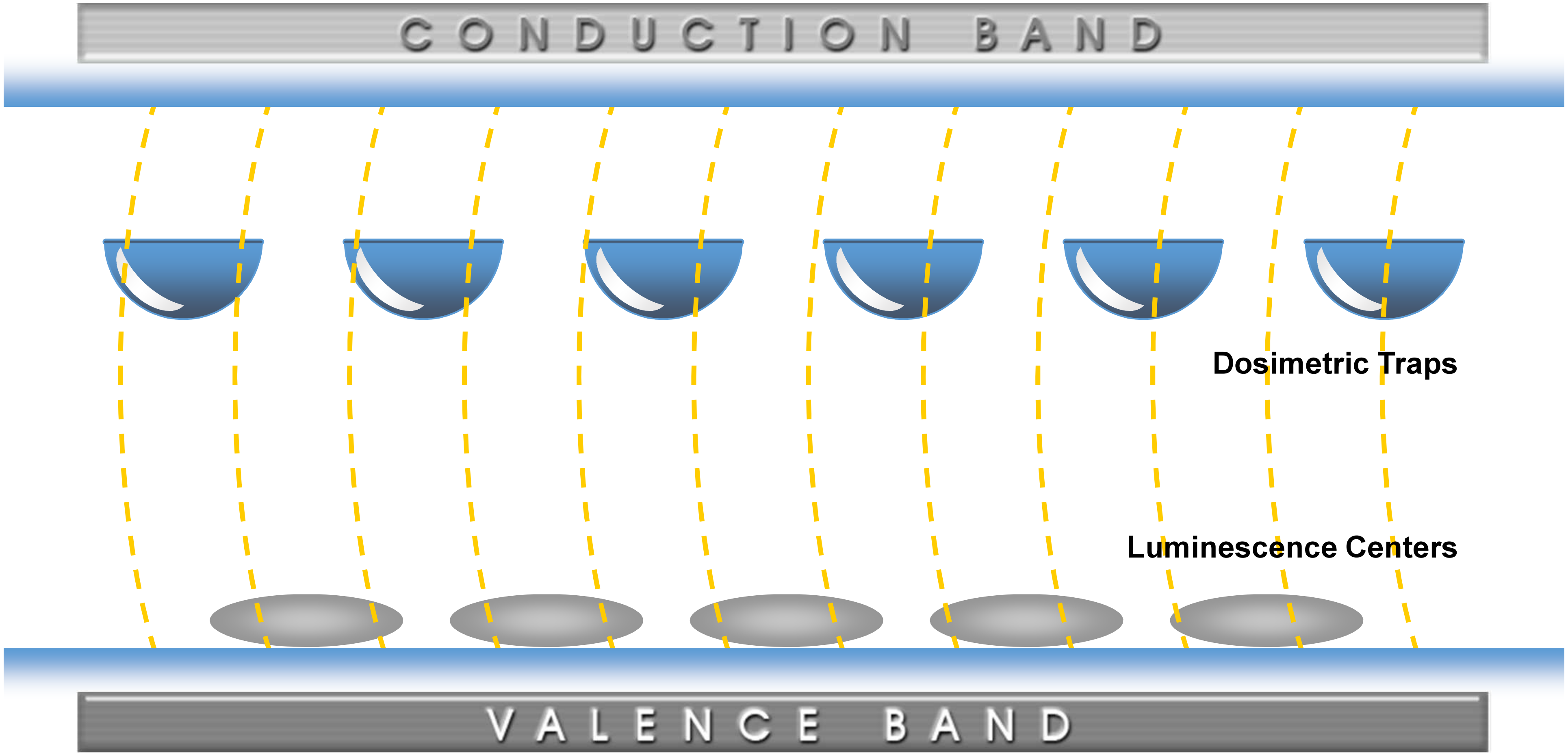


Optical fiber read out

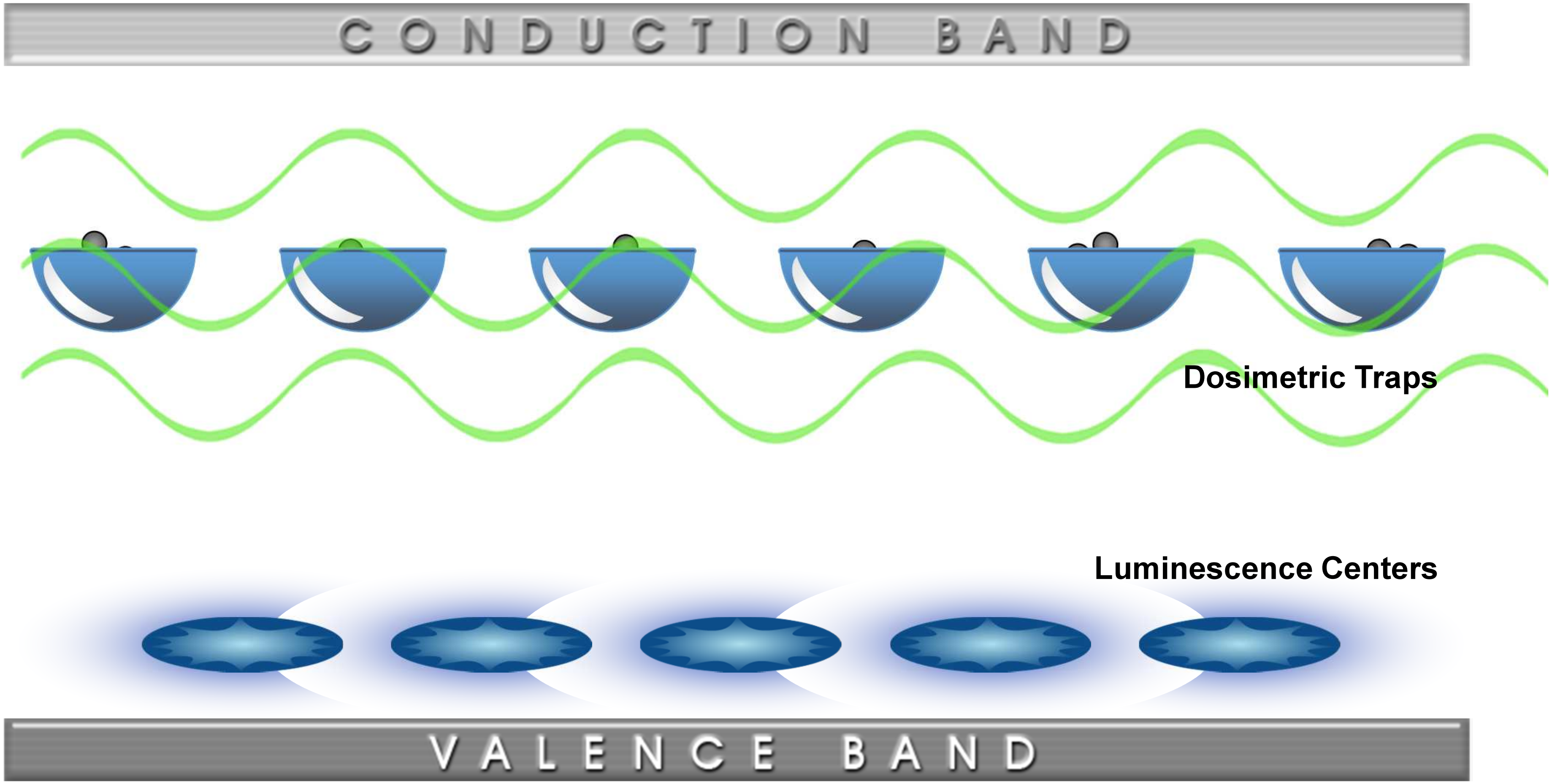
Simple OSL system



Conceptual Energy Diagram After Irradiation

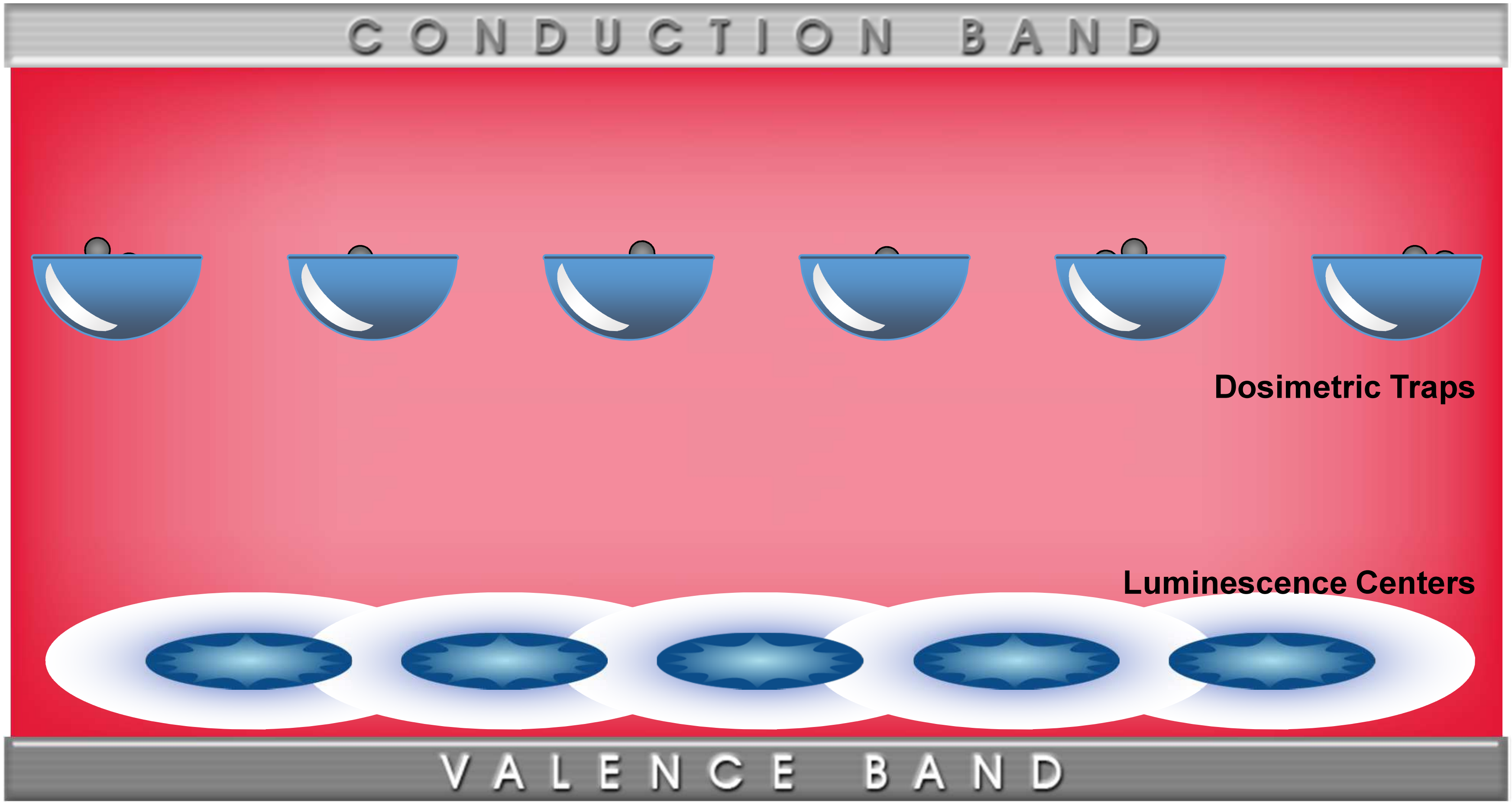


Optically Stimulated Luminescence (OSL)



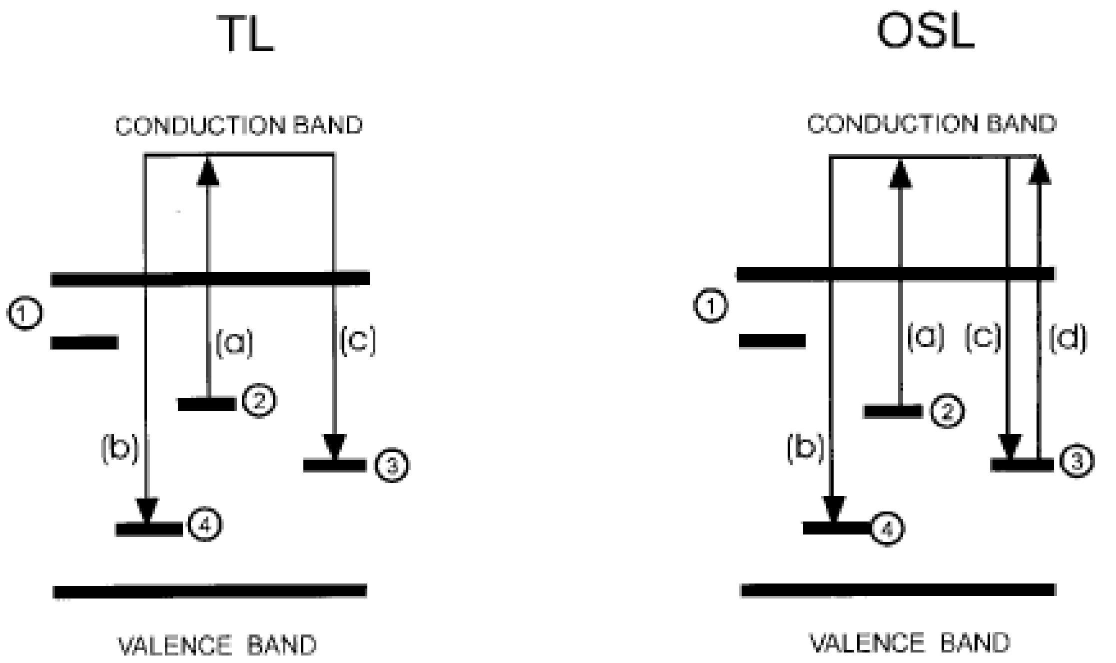
LANDAUER

Thermoluminescence Dosimetry (TLD)



LANDAUER

TL / OSL



1. Shallow trap
2. TL/OSL trap
3. Deep trap
4. Radiative recombination center

ADVANTAGES OF USING OSL OVER TL

1) OSL is normally measured at room temperature and is thus a non-destructive method (e.g. TL suffers from thermal quenching)

2) OSL is theoretically more sensitive than TL

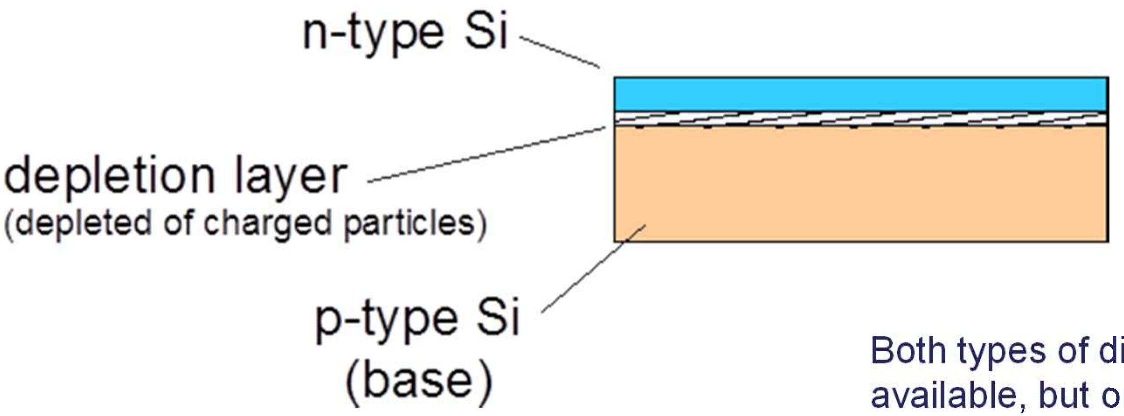
3) Parts of the OSL signal can be measured multiple times on same sample (short shine). TL requires total erasing of signal

4) The TL signal can usually be measured after OSL readout on same sample (not same traps)

5) Heating a sample (TL) will release luminescence from the whole sample

SEMICONDUCTOR DOSIMETRY

- A silicon diode dosimeter is a positive-negative junction diode.
- The diodes are produced by taking n-type or p-type silicon and counter-doping the surface to produce the opposite type material.



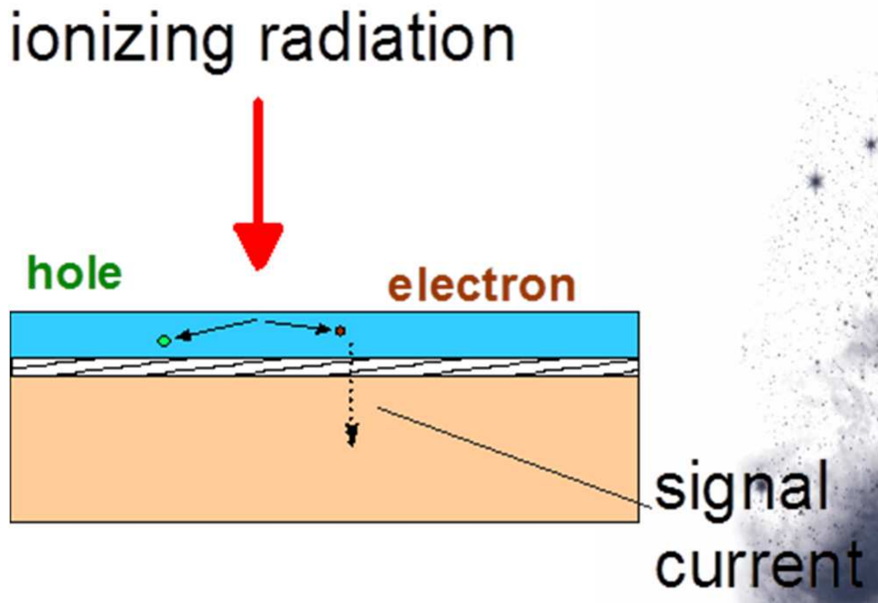
These diodes are referred to as n-Si or p-Si dosimeters, depending upon the base material.

Both types of diodes are commercially available, but only the p-Si type is suitable for radiotherapy dosimetry, since it is less affected by radiation damage and has a much smaller dark current.

Principle

The depletion layer is typically several μm thick. When the dosimeter is irradiated, charged particles are set free which allows a signal current to flow.

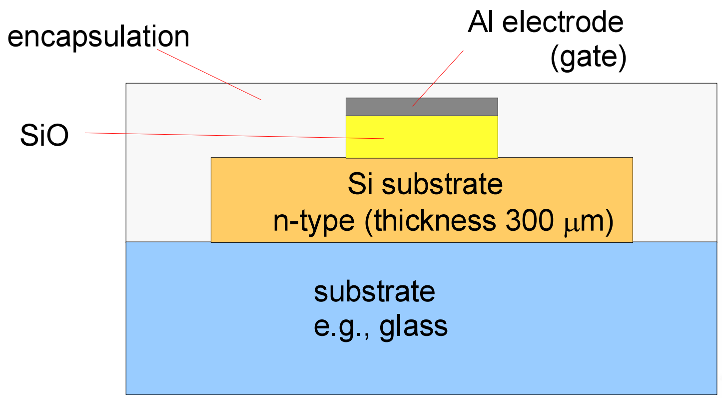
Diodes can be operated with and without bias. In the photovoltaic mode (without bias), the generated voltage is proportional to the dose rate.



SILICON DIODE DOSIMETRY SYSTEMS

MOSFET DOSIMETRY SYSTEMS

A MOSFET dosimeter is a Metal-Oxide Semiconductor Field Effect Transistor.



Physical Principle:

- Ionizing radiation generates charge carriers in the Si oxide.
- The charge carries moves towards the silicon substrate where they are trapped.
- This leads to a charge buildup causing a change in threshold voltage between the gate and the silicon substrate.

MOSFET DOSIMETRY SYSTEMS

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Measuring Principle:

- MOSFET dosimeters are based on the measurement of the threshold voltage, which is a linear function of absorbed dose.
- The integrated dose may be measured during or after irradiation.

Characteristics:

- MOSFETs require a connection to a bias voltage during irradiation.
- They have a limited lifespan.
- The measured signal depends on the history of the MOSFET dosimeter.

MOSFET DOSIMETRY SYSTEMS

Advantages

- MOSFETs are small
- Although they have a response dependent on radiation quality, they do not require an energy correction for mega-voltage beams.
- During their specified lifespan they retain adequate linearity.
- MOSFETs exhibit only small axial anisotropy (±2% for 360º).

Disadvantages

- MOSFETs are sensitive to changes in the bias voltage during irradiation (it must be stable).
- Similarly to diodes, they exhibit a temperature dependence.

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Joint innovative training and teaching/ learning program in enhancing development and transfer knowledge of application of ionizing radiation in materials processing

Thank you for your attention!