Validation and Routine Process Control for Low-energy Electron Accelerators

Miller, Arne; Helt-Hansen, Jakob

Publication date:
2011

Document Version
Publisher's PDF, also known as Version of record

RadTech Europe 2011 – presentation 13.5

Validation and routine process control for low-energy electron accelerators

Arne Miller*, Jakob Helt-Hansen

Radiation Research Department
Risø National Laboratory for Sustainable Energy, Risø-DTU
Technical University of Denmark
DK-4000 Roskilde, Denmark

*) corresponding author e-mail: armi@risoe.dtu.dk
fax: +45 4677 4959

Abstract

Radiation curing may not operate under the same strict legal requirements as, for example, radiation sterilization, but it is essential that dose is not delivered to product outside specifications without the knowledge of the operator. This is obtained though establishing and understanding the relationships between key parameters (energy, beam current, beam width and conveying speed) of an electron accelerator facility and absorbed dose.

The relationships are established during dosimetric validation of the facility. Validation is defined as “documented procedure for obtaining, recording and interpreting the results required to establish that a process will consistently yield product complying with predetermined specifications”, and for irradiation processes the international standard for radiation sterilization EN ISO 11137 can be used as a template for the validation activities.

Using the ISO 11137 template, this presentation describes how the steps of validation: Installation Qualification (IQ), Operational Qualification (OQ) and Performance Qualification (PQ) can be applied for low-energy e-beam polymer processing. Calibration of dosimeters and routine dosimetry process control are also described.

Keywords: Low-energy, dosimetry, validation, process control.

1. Introduction

The major applications of low-energy electron accelerators (80-300 keV) have different requirements to the level of documentation for running the process. However, several low-energy applications involve health issues, and these applications are regulated by international or national health regulations, which generally require that a quality management system is in place. This implies that measurements of absorbed dose are traceable to national standards, and that the irradiation process is validated so that it is ensured that dose to product exceeds a minimum dose required for an effective process, and that a maximum dose is not exceeded that might impair the properties of the product.

The health-related issues where these principles are applied are primarily radiation sterilization of packaging materials for pharmaceutical products (Sadat and Huber, 2002), and curing of inks and lacquers on food packaging materials. However, the several other low-energy irradiation

processes may require traceability of the delivered dose, and dose measurements as documentation for a correctly executed process.

The international standard EN ISO 11137-1 (2006) has been written for radiation sterilization of medical devices, but it provides a template for documentation of almost any radiation process, including curing and crosslinking using low-energy electrons. Not all users of low-energy electron irradiations require dosimetry to be traceable to a national standard, and may find it practical to establish a reproducible dosimetry method without traceability to national standards that satisfies their own specific needs and requirements. However, EN ISO 11137-1 still provides a useful template also in these situations.

The outline for validation and routine process control given in EN ISO 11137-1 is followed by the ISO/ASTM standards for dosimetry in radiation processing, the most relevant ones for this discussion being ISO/ASTM 51649 (2005) for high energy electron accelerators (0.3 – 25 MeV) and ISO/ASTM 51818 (2009) for low energy electron accelerators (80 – 300 keV).

The elements of EN ISO 11137-1 comprises the following with chapter numbers in parenthesis:

- Calibration and measurement traceability (4)
- Equipment characterization (6)
- Product definition (7)
- Process definition (8)
- Validation (9)
    - Installation Qualification (9.1)
    - Operational Qualification (9.2)
    - Performance Qualification (9.3)
- Routine Process Control (10)

EN ISO 11137-1 defines “validation” as:

> “- documented procedure for obtaining, recording and interpreting the results required to establish that a process will consistently yield product complying with predetermined specifications.”

2. Calibration and measurement traceability

Dosimeters used in low-energy applications are thin films, such as the radiochromic film described in ISO/ASTM 51275 (2004). The response of these dosimeters is affected by various influence factors such as humidity and temperature (ASTM 2701-09, 2009). In order to minimize these effects, two calibration methods are recommended (Sharpe and Miller, 2009):

1. In-plant calibration. Routine dosimeters are irradiated at the facility where the dosimeters will be used. The routine dosimeters are irradiated together with reference dosimeters for measurement of the dose to the routine dosimeters.
2. Calibration laboratory calibration followed by in-plant verification. Routine dosimeters are irradiated at a calibration laboratory, usually in a cobalt-60 gamma cell at constant (low) dose rate and constant temperature. The calibration function that is established based on this irradiation is verified by irradiation with reference dosimeters at the facility of use at a few selected doses.

Without in-plant irradiation for calibration or verification measurement traceability cannot be obtained. The conditions of calibration irradiation and the conditions of use would be different, and without data to document the effect of the different conditions the traceability chain would be broken.

3. The concept of Dμ

The response of dosimeters irradiated at low energy will depend on the thickness of the dosimeter and on the radiation energy. This illustrated in figure 1, where dosimeters of different thickness are irradiated using 125 keV electrons. If all dosimeters were initially calibrated at high energy e-beam or at gamma, then they will measure different doses when irradiated to the same low-energy e-beam.

This problem is overcome if the surface dose Dμ is determined for each dosimeter (Helt-Hansen et al, 2010). In order to do so the depth dose curve must be measured and a general response function of the dosimeter must be known. The value of Dμ is defined as the dose to water in the first micrometer of the absorbing material.

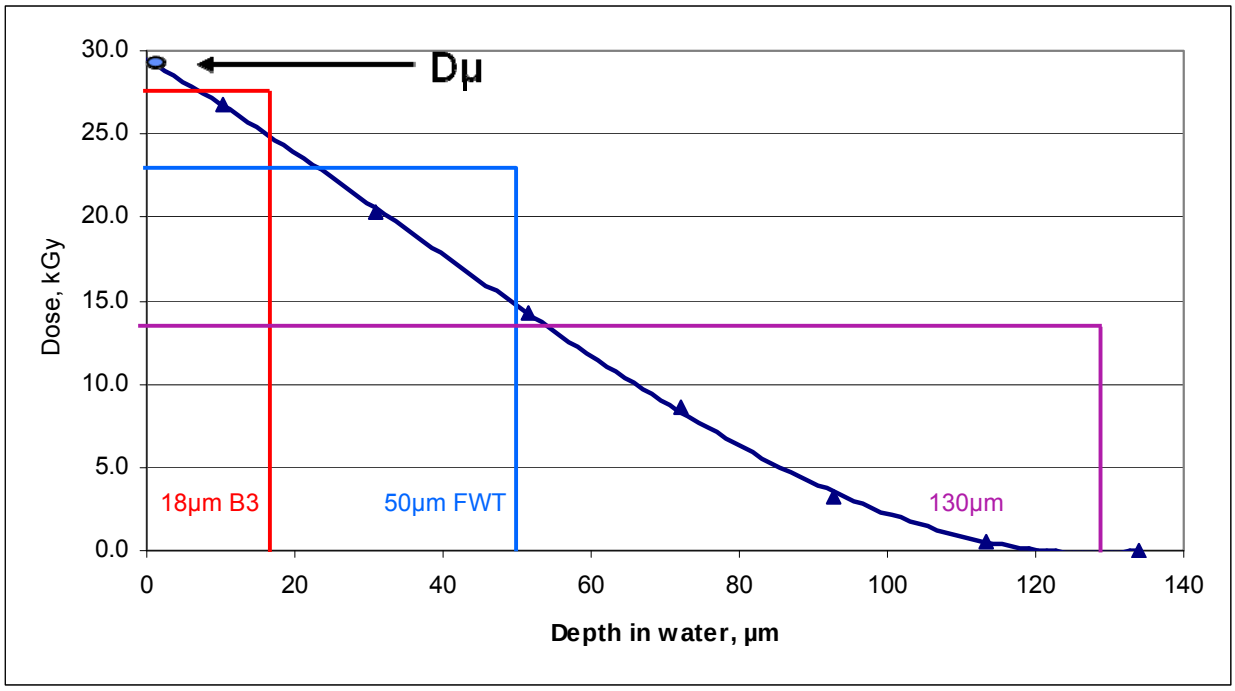

Figure 1. Typical depth dose distribution for a 125 electron accelerator. Three commonly used dosimeters (18 μm Risø B3, 50 μm FWT-60 and 130 μm alanine film) are shown, each with thickness and measured average dose for irradiation at this accelerator. The surface dose Dμ is also indicated (Helt-Hansen et al, 2010)

The user of the low-energy electron accelerator will not have to deal with this problem; it is purely for the calibration laboratory that issues the reference dosimeter, usually alanine film (ISO/ASTM 51607, 2004) for dose measurement during irradiation for calibration or verification. When this method is used, the user's dosimeters will calibrated in terms of Dμ.

4. Installation qualification (IQ) and operational qualification (OQ)

IQ is "carried out to demonstrate that the sterilization equipment and any ancillary items has been supplied and installed in accordance with their specification." (EN ISO 11137, 2006). However, whether or not data are "in accordance with specification" depends on the agreement between supplier and user. The dosimetry measurements that might be carried during IQ are

often the same as the ones carried out during OQ, in fact the IQ dose measurements might be considered as the first of the OQ measurements. IQ is therefore not described in detail, rather the user should specify his IQ requirements based on knowledge about the OQ reqiuirements.

OQ “shall demonstrate that the irradiator, as installed, is capable of operating and delivering appropriate doses within defined acceptance criteria” (EN ISO 11137, 2006). Another important aspect of OQ is that it provides baseline data to show consistent operation of the facility.

OQ dose measurements at an electron accelerator typically concerns the steps described in the table below.

Characteristics to be measured	Significance
Dose distribution in reference product	Not relevant for low energy
Beam width and homogeneity	Important
Beam energy and beam penetration	Energy: Not important Beam penetration: Important
Dose as function of speed, current and beam width	Important
Beam spot size	Not relevant for low energy – in most cases
Effect of process interruption	Not relevant for low energy – in most cases

Dose distribution in reference product.

This measurement is relevant in a penetrating electron beam (for example 10 MeV), but not in beams with penetration of only tens of micrometers.

Beam width and homogeneity

The beam width is measured by placing dosimeter strips or discrete dosimeters at selected intervals over the full beam width. Whenever possible, dosimeters should be placed beyond the expected beam width to identify the limits of the full beam width. The width of the beam is generated by extended or multiple cathodes or by scanning a narrow beam.

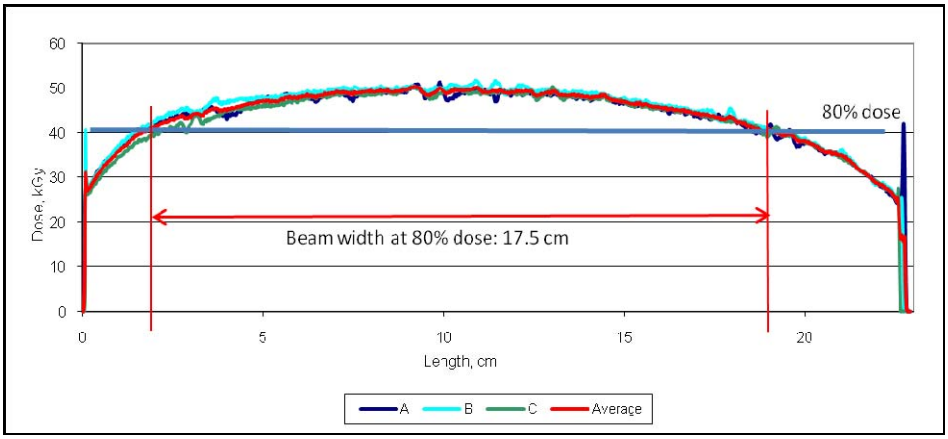

Figure 2. Example of beam width measurements at a 125 keV electron accelerator used in sterilization of pharmaceutical packaging.

Beam penetration

The beam penetration is measured using a stack of thin dosimeters or by placing a dosimeter strip under thin layers of plastic foils.

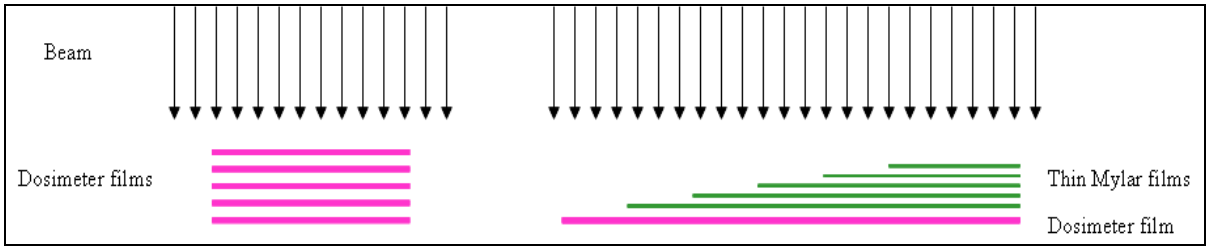

Figure 3. Arrangement for measurement of beam penetration using either a stack of dosimeter films or a “staircase” of thin films placed over a dosimeter film.

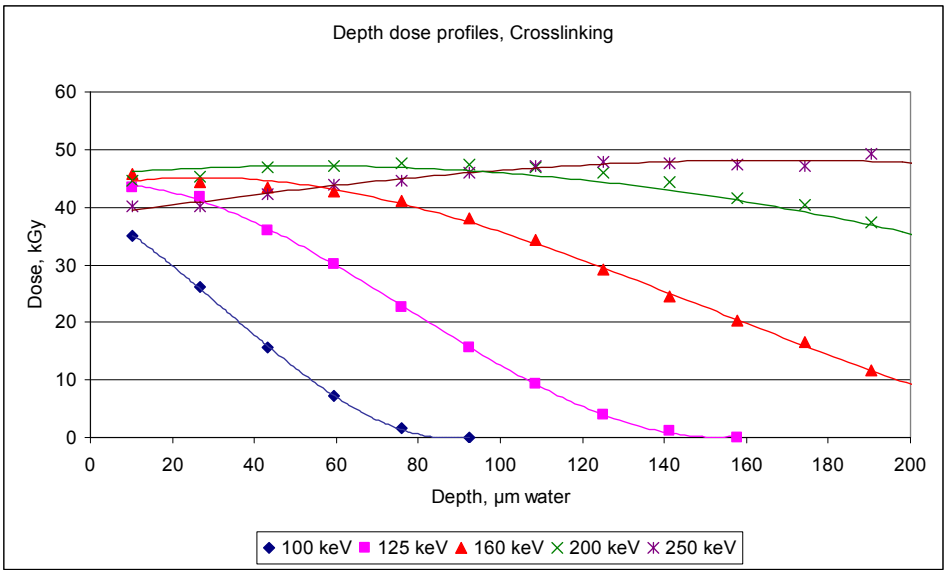

Figure 4. Depth dose curves measured at different electron beam energies.

Beam energy

Beam energy can be determined from the measured beam penetration (ISO/ASTM 51649, 2005), but such methods are not established for energies below 300 keV, where the beam energy at the surface of the absorbing material depends strongly on the energy losses in window and air gap between window and material.

Dose as function of beam current, beam width and conveying speed

Dose to product irradiated in an electron accelerator facility is proportional to beam current (I), inversely proportional to conveying speed (V_I), and inversely proportional to beam width (W_b). This is expressed as

$$\text{Dose} = K * (I/(V_I * W_b)) \qquad \text{(eq. 1)}$$

Where D = Absorbed dose (Gy)
I = Average beam current (A)

V_l = Conveying speed (m s^{-1})
W_b = Beam width (m)
K = Slope of the straight line relationship in eq. 1 (Gy * m^2) / (A * s)

This straight-line relationship should be determined for each energy selected for the operation of the facility. In order to determine the relationship, dose should be measured at a specific location using a number of selected parameter sets of beam current, conveying speed and beam width to cover the operating range of the facility.

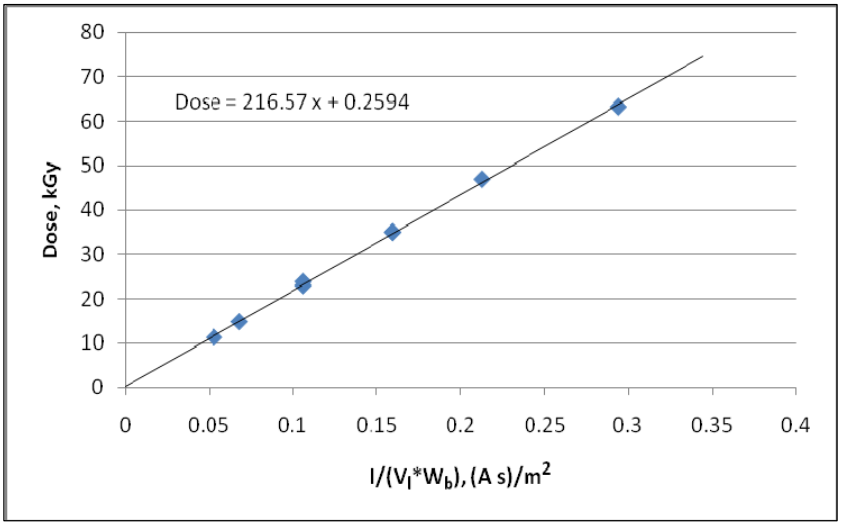

Figure 5. Example of measurement of dose as function of beam current, beam width and conveying speed. The slope of the line (K) is 216.57 (Gy * m^2) / (A * s).

Beam spot size

The shape of the beam is not of concern in most low-energy applications, but for scanned beam and high-speed product movement it must be assured that the beam overlaps from one scan to the next as the product moves through the beam zone.

Effect of process interruption

The effect of a process interruption should be determined, so that decisions about possible product disposition can be made, and for most low-energy applications product is discarded in case of a process interruption. A process interruption can be caused by, for example, failure of beam current delivery or by the conveyor stopping. An arc in the high voltage components can cause a short process interruption and its effect is normally not possible measure, as the occurrence of arcs cannot be planned. Nevertheless, the effect should be estimated.

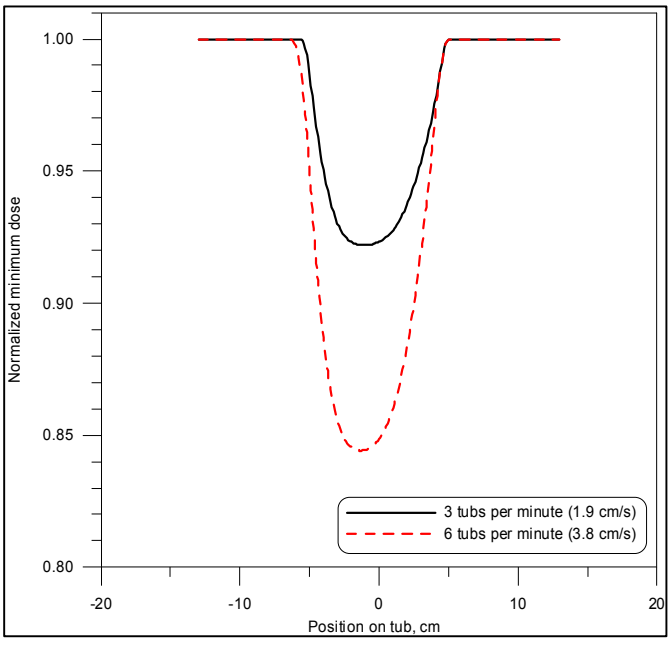

Figure 6. Calculation of the effect of an arc at a 125 keV electron accelerator at two different product speeds.

The OQ measurements should be repeated a sufficient number of times to allow determination of measurement uncertainties. Based on these uncertainties acceptable limits for variation of the operating parameters can be determined.

It may be necessary to carry out a new OQ after changes to the facility that might affect dose or dose distribution. Activities that might lead to a new OQ of the irradiation facility include, but are not limited to

- replacement of accelerator emitter,
- replacement of accelerator window,
- replacement of window support grid,
- replacement of conveyor parts,
- change in accelerator energy,
- change in distance of accelerator window to product surface.

Assessment of the change may lead to all or only parts of the OQ to be repeated.

5. Performance qualification (PQ)

PQ dose mapping is carried out to demonstrate that

- minimum dose to product exceeds the dose required for the intended effect and
- maximum dose to product does not exceed a maximum acceptable dose.

In many low-energy applications OQ and PQ are combined, because only a single product is irradiated. However, at - for example – sterilization applications detailed dose maps of complex product may have to be carried out.

Figure 7. Complex product being dose mapped for sterilization at low-energy e-beam.

6. Routine process control

The process parameters (beam energy, beam current, beam width and conveying speed) should be monitored to provide assurance that the irradiator is consistently operating within specifications.

The dose at a routine monitoring position should be measured at intervals specified by the operator of the facility. The intervals should be chosen to provide assurance that the irradiator is consistently operating within specified limits.

In order to show that a facility is consistently operating within specifications it is recommended to apply statistical process control (SPC) on the measured results. A useful approach to SPC is given by the Panel on Gamma and Electron Irradiation (Panel, 2006).

7. Summary

We demonstrate that dosimetry measurements can be carried out for validation and process control of low-energy electron accelerators following the outline given by EN ISO 11137-1. We have successfully applied these dosimetry methods for validation of low-energy electron accelerator facilities for both sterilization and curing applications.

References

ASTM E 2701-09 Guide for Performance Characterization of Dosimeters and Dosimetry Systems for Use in Radiation Processing. ASTM International, West Conshohocken, PA 19428, USA.

EN ISO 11137, 2006. Sterilization of Health Care Products – Radiation – Part 1: Requirements for development, validation and routine control of a sterilization process for medical devices. CEN, Rue de Stassart, 36, B-1050 Brussels, Belgium.

Helt-Hansen, J., Miller, A., Sharpe, P., Laurell, B., Weiss, D., Pageau, G., 2010. D_{μ} - a new concept in industrial low energy electron dosimetry. Rad. Phys. Chem.79, 66-74.

ISO/ASTM 51818, 2009. Practice for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies between 80 and 300 keV. ASTM International, West Conshohocken, PA 19428, USA.

ISO/ASTM 51649, 2005. Practice for Dosimetry in an Electron-Beam Facility for Radiation Processing at Energies between 300 keV and 25 MeV. ASTM International, West Conshohocken, PA 19428, USA.

ISO/ASTM 51275, 2004. Practice for use of a radiochromic film dosimetry system. ASTM International, West Conshohocken, PA 19428, USA.

ISO/ASTM 51607, 2004. Practice for use of an alanine/EPR dosimetry system. ASTM International, West Conshohocken, PA 19428, USA.

Panel, 2006. A method for statistical process control of radiation sterilization facilitites. Panel on Gamma and Electron Irradiation, 212 Piccadilly, London W1J 9HG, UK. http://www.irradiationpanel.org/docs/publications/Panel_SPC_Method.pdf

Sadat, T. and Huber, T., 2002. E-beam – a new transfer system for isolator technology. Rad. Phys. Chem., 63, 587-589.

Sharpe, P. and Miller, A., 2009. Guidelines for the calibration of routine dosimetry systems for use in radiation processing. NPL report CIRM 29 (2009). National Physical Laboratory, Teddington, Middlesex, UK.