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Standard Guide for Dosimetry In Radiation Processing of Fluidized Beds and Fluid Streams[1]

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1. Scope

1.1 This guide describes several dosimetry systems and methods suitable for the documentation of the irradiation of product transported as fluid or in a fluidized bed.

1.2 The sources of penetrating ionizing radiation included in this guide are electron beams, X-rays (bremsstrahlung) and gamma rays.

1.3 Absorbed doses from 10 to 100,000 gray are considered, including applications such as disinfestation, disinfection, bioburden reduction, sterilization, crosslinking and graft modification of products, particularly powders and aggregates.

1.4 *This guide does not purport to address the safety concerns, if any, associated with the use of fluidized beds and streams incorporating sources of ionizing radiation. It is the responsibility of the user of this guide to establish appropriate safety and health practices and to determine compliance with regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E170 Terminology Relating to Radiation Measurements and Dosimetry

E666 Practice for Calculating Absorbed Dose From Gamma or X Radiation

E1026 Practice for Using the Fricke Dosimetry System

E2232 Guide for Selection and Use of Mathematical Methods for Calculating Absorbed Dose in Radiation Processing Applications

F1355 Guide for Irradiation of Fresh Agricultural Produce as a Phytosanitary Treatment

F1885 Guide for Irradiation of Dried Spices, Herbs, and Vegetable Seasonings to Control Pathogens and Other Microorganisms

2.2 *ISO/ASTM Standards:*

51204 Standard Practice for Dosimetry in Gamma Irradiation Facilities for Food Processing

51261 Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing

51275 Practice for Use of a Radiochromic Film Dosimetry System

51310 Practice for the Use of a Radiochromic Optical Waveguide Dosimetry Systems

51400 Practice for Characterization and Performance of a High-Dose Radiation Dosimetry Calibration Laboratory

51431 Practice for Dosimetry in Electron and X-Ray (Bremsstrahlung) Irradiation Facilities for Food Processing

51538 Practice for Use of the Ethanol-Chlorobenzene Dosimetry System

51540 Practice for Use of a Radiochromic Liquid Dosimetry System

51607 Practice for Use of the Alanine-EPR Dosimetry System

51608 Practice for Dosimetry in an X-Ray (Bremsstrahlung) Facility for Radiation Processing

51649 Practice for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies between 300 keV and 25 MeV

51702 Practice for Dosimetry in a Gamma Irradiation Facility for Radiation Processing

51707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing

51818 Practice for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies Between 80 and 300 keV

51956 Practice for Application of Thermoluminescence Dosimetry (TLD) Systems for Radiation Processing

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

2.3 *International Commission on Radiation Units and Measurements Reports*[3]

ICRU Report 14 Radiation Dosimetry: X-Rays and Gamma Rays with Maximum Photon Energies Between 0.6 and 50 MeV

ICRU Report 17 Radiation Dosimetry: X-Rays Generated at Potentials of 5 to 150 kV

ICRU Report 30 International Comparison of Radiological Units and Measurements: Quantitative Concepts and Dosimetry in Radiobiology

ICRU Report 34 The Dosimetry of Pulsed Radiation

ICRU Report 35 Radiation Dosimetry: Electron Beams with Energies Between 1 and 50 MeV

ICRU Report 37 Stopping Powers for Electrons and Positrons

ICRU Report 60 Fundamental Quantities and Units for Ionizing Radiation

2.4 *National Committee for Radiation Protection*

NCRP Report 69 Dosimetry of X-Ray and Gamma-Ray Beams for Radiation Therapy in the Energy Range 10 keV to 50 MeV

3. Terminology

3.1 *Definitions:*

3.1.1 *absorbed dose D*—quantity of ionizing radiation energy imparted per unit mass of a specified material. The SI unit of absorbed dose is the gray (Gy), where 1 gray is equivalent to the absorption of 1 joule per kilogram of the specified material (1 Gy = 1 J kg^{-1}). The mathematical relationship for dose is the quotient of dε by d*m*, where dε is the mean incremental energy imparted by ionizing radiation to matter of incremental mass d*m* (see ICRU 60).

3.1.1.1 *Discussion*—discontinued unit for absorbed dose is the rad (1 rad = 0.01 Gy). Absorbed dose is sometimes referred to simply as dose.

3.1.2 *absorbed dose mapping*—measurement of absorbed dose within a process stream using dosimeters transported at specified locations to produce a one or two-dimensional distribution of absorbed dose, thus rendering a map of absorbed-dose values.

3.1.3 *absorbed dose rate*—absorbed dose in a material per incremental time interval, i.e. the quotient of d*D* by d*t* (see ICRU 60) Unit: Gy s^{-1}

3.1.3.1 *Discussion*—absorbed dose rate can be specified in terms of the average value of d*D* by d*t* over long-time intervals, for example, in units of Gy min^{-1} or Gy h^{-1}

3.1.4 *areal density*—thickness of an object normalized by density. The SI unit is kg m^{-2}.

3.1.4.1 *Discussion*—the abbreviation gsm is also used in referring to areal density in grams per square meter in some technical literature.

3.1.5 *bed control*—technique used for determining the fluidized bed thickness and maintaining it between the limits required for controlled application of the process.

3.1.6 *bed thickness*—total thickness of the fluidized bed, which includes the product being processed and the carrier medium, both normalized by density. The SI unit is kg. m^{-2}.

3.1.6.1 *Discussion*—thickness is typically quoted in g. m^{-2} due to its numerical equivalence to thickness in micrometers for unit density matter.

3.1.7 *Bremsstrahlung*—broad-spectrum electromagnetic radiation (X-rays) emitted when an energetic electron is influenced by strong electric field or magnetic field such as that in the vicinity of an atomic nucleus.

3.1.7.1 *Discussion*—bremsstrahlung is produced when an electron beam strikes any material (converter). The bremsstrahlung spectrum depends on the electron energy, the converter material and its thickness, and contains energies up to the maximum kinetic energy of the incident electrons (see ISO/ASTM Practice 51608).

3.1.8 *calibration curve*—graphical representation of the dosimetry system's response function.

3.1.9 *depth-dose distribution*—variation of absorbed dose with depth from the incident surface of a material exposed to a given radiation.

3.1.10 *dose uniformity ratio*—ratio of the maximum to the minimum absorbed dose within the irradiated object or process stream.

3.1.10.1 *Discussion*—the concept is also referred to as the max/min dose ratio and is significantly influenced by the turbulence of the product flow.

3.1.11 *dosimeter*—device that, when irradiated, exhibits a quantifiable change in some property of the device which can be related to absorbed dose in a given material using appropriate analytical instrumentation and techniques.

3.1.12 *dosimeter response*—reproducible, quantifiable radiation effect on a dosimeter produced by a given absorbed dose.

3.1.13 *dosimetry system*—system used for determining absorbed dose, consisting of dosimeters, measurement instruments and their associated reference standards, and procedures for the system's use.

3.1.14 *electron energy*—kinetic energy of the accelerated electrons. The electron energy at the product is equal to its accelerated energy in vacuum less its energy losses in the accelerator's window and the air gap separating the product and the window.

3.1.15 *electron fluence*—amount of electronic charge traversing a unit area of the target, usually expressed in microcoulombs per square centimeter. It is the integral of flux over total exposure time

3.1.16 *fluidized bed or stream*—means by which the product is transported and presented to the radiation source. The carrier medium may be gaseous or liquid. The product distribution within the carrier medium may not be uniform.

3.1.17 *primary-standard dosimeter*—dosimeter of the highest metrological quality, established and maintained as an absorbed dose standard by a national or international standards organization.

[3] Available from the International Commission on Radiation Units and Measurements, 7910 Woodmont Avenue, Suite 800, Bethesda, MD, 20814,USA

3.1.18 *quality assurance*—all systematic actions necessary to provide adequate confidence that a calibration, measurement, or process is performed to a predefined level of quality.

3.1.19 *real time dose monitor*—instrument capable of continuously providing measured data on dose delivered during processing.

3.1.20 *reference-standard dosimeter*—dosimeter of high metrological quality, used as a standard to provide measurements traceable to and consistent with measurements made using primary standard dosimeters.

3.1.21 *response function*—mathematical representation of the relationship between dosimeter response and absorbed dose for a given dosimetry system.

3.1.22 *routine dosimeter*—dosimeter calibrated against a primary, reference, or transfer standard dosimeter and used for routine absorbed dose measurement.

3.1.23 *self-shielded system*—product transport-irradiation unit with integral shielding.

3.1.23.1 *Discussion*—this type of conformal shielding is typically used at lower radiation energies where rather thin layers of lead can protect the surrounding environment from virtually all of the radiation generated by the irradiator.

3.1.24 *simulated product*—mass of material with attenuation and scattering properties similar to those of the product, material or substance to be irradiated, sometimes called a dummy product.

3.1.25 *surface dose*—absorbed dose at the surface of the product.

3.1.25.1 *Discussion*—This definition becomes particularly important where low energy radiation is used to treat only the surface of particulates.

3.1.26 *target dose*—absorbed dose delivered to the surface of the bed which will produce the required absorbed dose distribution within the remainder of the product irradiated in the fluidized bed.

3.1.27 *traceability*—ability to demonstrate by means of an unbroken chain of comparisons that a measurement is in agreement within acceptable limits of uncertainty with comparable nationally or internationally recognized standards.

3.1.28 *transfer-standard dosimeter*—dosimeter, often a reference standard dosimeter, suitable for transport between different locations, used to compare absorbed-dose measurements.

3.1.29 *uncertainty*—parameter associated with the result of any measurement that characterizes the dispersion of the values that could reasonably be attributed to the measured or derived quantity.

3.1.30 *validation*—establishment of documented evidence, which provides a high degree of assurance that a specified process will consistently produce a product meeting its predetermined specifications and quality attributes.

3.2 Definitions of other terms used in this standard that pertain to radiation measurement and dosimetry may be found in ASTM Terminology E170. Definitions in E170 are compatible with ICRU 60; that document, therefore, may be used as an alternative reference.

4. Significance and Use

4.1 *Dosimetric Techniques*—The processes addressed here utilize a variety of techniques for the dynamic presentation of the product to the radiation source. This may involve gravitational flow or simple pneumatic transport about or past the radiation source. In the case of fluidized beds, the product may be presented to the radiation source while supported in a gaseous or liquid stream moving at relatively high velocities. This document provides a guide to the dosimetric techniques suitable for these processes.

4.2 *Food Products*—Food products may be treated with ionizing radiation, such as energetic electrons from accelerators or gamma rays from ^{60}Co or ^{137}Cs sources, or X-rays, for numerous purposes, including control of parasites and pathogenic microorganisms, insect disinfestation, growth and maturation inhibition, and shelf-life extension.

NOTE 1—Food irradiation specifications usually include upper and lower limits of absorbed dose: a minimum to ensure the intended beneficial effect and a maximum to avoid product degradation. For a given application, one or both of these values may be prescribed by regulations that have been established on the basis of available scientific data. Therefore, it is necessary to determine the capability of an irradiation facility to process within these absorbed-dose limits prior to the irradiation of the food product. Once this capability is established, it may be necessary to monitor and record the dose range delivered to the product during each production run to verify compliance with the process specifications within a predetermined level of confidence.

4.3 *Randomized Flow*—In a stream of randomized flow; i.e. turbulent instead of laminar, variations occur which lead to a dose distribution for the particles entrained in the stream. The "idealized" maximum and minimum doses possible can be calculated based upon knowledge of the applied dose rate, the product dwell time in the irradiation cell and the product or bed thickness. The experimentally determined maximum and minimum doses delivered to each particle, should not be confused with these idealized dose limits.

4.4 *Treatment range*—The location of the product (or of the dosimeter) in the fluidized bed or stream will determine its absorbed dose during passage through the radiation field. The experimental dose measurements in the fluidized bed or stream will define the range of product dose. The desired effect imparted to the product by irradiation will then be based upon this range of product dose and not upon maximum or minimum dose.

NOTE 2—In situations where a randomized mixing within the fluidized bed occurs with the intention that the particles or fluid elements pass through several radiation zones and accumulate a total dose with different dose rates, maximum and minimum dose values are difficult to determine and must be based on the results for the experimental dosimetry irradiated with the product . In the case of fluids, stirring after processing results only in effective treatment at a mean dose; no max and min dose measurement. For example, lethality curves will be determined as a function of this range of product treatment to the product in the fluidized bed or stream as determined by dosimetric techniques.

5. Types of Facilities, Source Characteristics and Fluidized Bed Parameters

5.1 Conventional gamma-ray sources (^{60}Co or ^{137}Cs), due to their low intrinsic dose rates, are useful for fluidized bed processing only when the irradiator is designed for the application.

5.2 The high dose rates typical of bremsstrahlung and electron beam sources are most suitable for fluidized bed treatment of product. Electron energies in the 0.3 to 3 MeV range are largely used for these applications, often in self-shielded systems under 0.5 MeV. Selection of the energies used will depend upon whether bulk or surface treatment of the particles carried in the fluidized bed is desired.

5.3 *Fluidized Bed Parameters—*

5.3.1 *Thickness*—The areal densities or bed thicknesses are typically in the range of 5 kg m^{-2} (5000 g m^{-2}) or less.

NOTE 3—Uniformity of product distribution in the stream is not critical as long as efficient product transport results at an acceptable bed thickness (see ISO/ASTM Guide 51261).

NOTE 4—Continuous (dc) electron beam systems are typically operated with accelerator current (at preset voltage or beam energy) coupled to stream velocity to achieve the desired dose.

5.3.2 *Velocity*—In the use of pulsed or scanned sources of energetic electrons for stream processing, care must be exercised. Limitations on product/stream velocity may be imposed by the pulse repetition or scanning frequencies of the source to ensure uniform product treatment. A generalized calculation formula for dose uniformity as a function of the product/stream velocity in scanned sources of energetic electrons for processing has been described **(1)**.

5.3.3 *Product flow rates*—Processing systems are also designed to limit product flow rates to levels compatible with the fixed source dose rate, as in the case of radioisotope sources. Areal density of the bed is controlled to ensure that the penetration of the radiation is sufficient to yield acceptable stream treatment uniformity.

6. Dosimetry Systems and Methods Suitable for Dose Measurements in Fluidized Beds and Fluid Streams.

6.1 *Description of Dosimeter Classes*

6.1.1 Dosimeters may be divided into four basic classes according to their relative quality and areas of application, primary-standard, reference-standard, transfer-standard, and routine dosimeters. ISO/ASTM Guide 51261 provides information about the selection of dosimetry systems for different applications. All classes of dosimeters except the primary-standards require calibration before their use.

6.1.1.1 *Primary-Standard Dosimeter*—Primary-standard dosimeters are established and maintained by national standards laboratories for calibration of radiation environments (fields) and other classes of dosimeters. The two most commonly used primary-standard dosimeters are ionization chambers and calorimeters.

6.1.1.2 *Reference-Standard Dosimeters*—Reference-standard dosimeters are used to calibrate radiation environments and routine dosimeters. Reference-standard dosimeters may also be used as routine dosimeters. Examples of reference-standard dosimeters along with their useful dose ranges are given in ISO/ASTM Guide 51261.

6.1.1.3 *Transfer-Standard Dosimeters*—Transfer-standard dosimeters are specially selected dosimeters used for transferring absorbed-dose information from an accredited or national standards laboratory to an irradiation facility in order to establish traceability for that facility. These dosimeters should be carefully used under conditions that are carefully controlled by the issuing laboratory. Transfer-standard dosimeters may be selected from either reference-standard dosimeters or routine dosimeters taking into consideration the criteria listed in ISO/ASTM Guide 51261.

6.1.1.4 *Routine Dosimeters*—Routine dosimeters may be used for process quality control, dose monitoring and dose mapping. Proper dosimetric techniques, including calibration, shall be employed to ensure that measurements are reliable and accurate. Examples of routine dosimeters, along with their useful dose ranges, are given in ISO/ASTM Guide 51261.

6.2 *Calibration of the Dosimetry System*

6.2.1 Prior to use, the dosimetry system (consisting of a specific batch of dosimeters and specific measurement instruments) shall be calibrated in accordance with the user's documented procedure that specifies details of the calibration process and quality assurance requirements. This calibration procedure shall be repeated at regular intervals to ensure that the accuracy of the absorbed dose measurement is maintained within required limits. Calibration methods are described in ISO/ASTM Guide 51261.

NOTE 5—At the time of publication of this document, no reference standard dosimeter was available from an accredited calibration laboratory to perform full *in situ* calibrations or *in situ* laboratory calibration verification for low electron beam energy. Also there is no low energy (80-300 kV) source of electron beam laboratory calibration available. Therefore users must perform a laboratory calibration using a high energy beam or gamma ray source and include an appropriate component of uncertainty in the estimate of overall uncertainty. It should also be noted that calibration under high energy electron beam conditions provided good agreement with a low energy in-line calorimeter.

6.2.2 Irradiation is a critical component of the calibration of the dosimetry system.

6.2.3 *Calibration Irradiation of Reference- or Transfer-Standard Dosimeters*—Calibration irradiations shall be performed at an accredited calibration laboratory, or in-house calibration facility meeting the requirements of ISO/ASTM Practice 51400, that provides an absorbed dose (or absorbed-dose rate) having measurement traceability to nationally or internationally recognized standards.

6.2.4 *Calibration Irradiation of Routine Dosimeters*—Calibration irradiations may be performed per 6.2.3, or at a production or research irradiation facility together with reference- or transfer-standard dosimeters that have measurement traceability to nationally or internationally recognized standards. This clause also applies when reference-standard dosimeters are used as routine dosimeters.

6.2.5 *Measurement Instrument Calibration and Performance Verification*—For the calibration of the instruments, and for the verification of instrument performance between calibrations, see ISO/ASTM Guide 51261, the corresponding

ISO/ASTM or ASTM standard for the dosimetry system, and/or instrument-specific operating manuals.

6.3 *Fluidized bed considerations:*

6.3.1 *Dose Mapping*—It should be noted that there is an important difference between dose mapping in a filled container or bin, and in a fluidized bed. In the former case, there may be no mixing, while in the latter case, turbulent flow usually exists. Once a bed of particulate matter comes into motion, the voids expand allowing individual particles to change position. This may change the bed's effective areal density, primarily through these changes in the product distribution in the bed. Hence, a dose measurement in a resting bed of bulk solids can be quite different from the results from a bed in motion.

NOTE 6—The application of mathematical methods for modeling the transport of electrons and photons in fluidized beds and fluid streams can provide valuable insight into process effectiveness. This offers an efficient complement to dosimetry and can provide guidance in irradiator design **(2)**. A guide for the selection and use of such methods is available in ASTM E2232.

6.3.2 *Bed Thickness*—Dosimetry must be conducted over the limits of bed thickness considered acceptable for the source energy provided by the processor. The degree of control of the bed thickness between these ranges will then determine the D_{max}/D_{min} ratios maintainable in the process with beam current (dose rate) slaved to bed velocity. In a similar manner, film dosimeters are used for the determination of dose with depth in bulk and packaged products **(3)**.

6.3.3 *Fluidized bed velocity*—Air velocity meters are commercially available which are well suited to the determination of fluidized bed velocity in air. Such instruments **(4)** can provide an accuracy of 3 % in velocity measurement for speeds up to 30 m s^{-1}. Volumetric flow rates up to 195,000 L s^{-1} can be measured. Probe access diameter is 6 mm, which can be easily accommodated in most ducts **(5)**.

6.4 *Dosimetry methods used in fluidized beds and streams.* ISO/ASTM Guide 51261 provides information for the selection of dosimetry systems applicable to the diagnosis of irradiated fluid streams and fluidized beds over the dose range of interest; i.e. 10^1 to 10^5 Gy. Other review articles **(6)** may be helpful in system selection.

6.4.1 *Alanine*—For most fluidized bed products, for example, fine powders, alanine EPR dosimetry provides a preferred technique for process validation. See ISO/ASTM Practice 51607 for details of its use. Alanine powder **(7,8)** is mixed homogeneously with the product at known low concentration. The EPR response of the mixture as a function of dose is then determined and the response curve can then be used, with small samples, to determine system performance under known fluidized bed conditions; i.e. velocity and dose rate. Operating conditions are normally continuously monitored through machine parameters. This technique typically renders dose values comparable to those received by the moving product since the alanine powder integrates the dose absorbed in a certain volume. With certain products, the EPR signal induced in the product itself has been used to monitor delivered dose **(9,10)**.

6.4.1.1 In addition to the standard technique of EPR free radical determination induced in α-alanine **(11)**, electrochemical potentiometric measurements of NH_3 produced in irradiated alanine powder **(7)** dissolved in water provide a broad range (0.1–1000kGy) dosimetry system for stream use.

6.4.2 *Hydrocarbon chemical dosimeters*—A number of hydrocarbon chemical dosimetry systems such as dyed cellulose acetate or dyed polymethylmethacrylate are practicable, in that they have densities and atomic constituents similar to biological systems, foods and water **(6,12)**. This advantage provides energy independent response to ionizing photons and electrons.

6.4.3 *Thin film dosimeters*—Radiochromic film dosimeters **(13,14)** may be used to determine the fluidized bed dose. See ISO/ASTM Practice 51275 for details of this dosimetry system. Thin alanine-polyethylene dosimeters may also be used **(15)**. They are inserted directly into the fluidized bed or stream, or may be rolled into product exemplars, or protective capsules, where sufficient penetration is provided by the source for such "protected" dosimetry - as in fluid streams for example. The literature describes recovery techniques suitable for continuous stream use, involving magnetic extraction of the capsule **(12,13,14)** or screen/filter recovery of film **(8)**.

6.4.4 *Semiconductor detectors*—The monitoring of the dose, and dose distribution in pourable products has utilized small diameter semiconductor detectors **(16)**. These devices can approximate the properties and dimensions of many bulk materials and are readily transported with them. Since their response has a linear relation with the electron fluence or dose received, such a measurement provides a cheap, reusable, convenient dosimeter for "pourable" products.

6.4.5 *Thermoluminescent dosimetry*—This has been widely used in the determination of the dose delivered by gamma rays and electron beams in fluid beds, particularly for sludge treatment. These may range from Ag doped low phosphate glass **(17)** to encapsulated $Li_2 B_4O_7$ and LiF dosimeters **(18)** to sand **(19)** or silica **(20)** separated from the sludge. A cleaning process using H_2O_2 or HF to prepare 5 mg samples of sand for readout in a conventional TLD reader has been described. All samples were subjected to a 120°C × 20 minute post-irradiation anneal to eliminate the influence of low temperature thermoluminescence peaks **(19)**. ISO/ASTM Practice 51956 addresses thermoluminescent dosimetry.

6.4.6 *Dyes*—Dimethylaminothiazine dye, methylene blue, whose bleaching by ionizing radiation is known to be stable in the 50-500 Gy region (measured at 664 nm), has been described for fluid treatment **(21,22)**. This range can be extended to 5 kGy with the addition of 0.1 % ethanol and to 10 kGy with 5 % ethanol **(23)**. Because of the relatively low cost of the dye, it is a useful dosimeter for quality control of electron beam processing of large volumes of wastewater where doses in the 5-30 kGy range are used. The solutions are usually sealed in small glass ampoules or pouches and readout within 24 hours of irradiation to avoid oxidative decoloration **(24)**. ISO/ASTM 51310 addresses Optical Waveguide Dosimetry and ISO/ASTM 51540 addresses Radiochromic Liquid dosimetry.

6.4.7 *Ethanol-monochlorobenzene dosimeter system*—An ethanol-monochlorobenzene dosimeter (ECB) system for 10

MeV electron irradiation systems has been described **(23)**. The solution was encapsulated in glass ampoules with a wall thickness of 0.5 mm. When used at lower energies (e.g. 4 MeV) double layer mylar pouches can be used over a wide dose range (1-50 kGy). Readout is accomplished via titration or conductivity measurements in order to determine its Cl^- content and hence absorbed dose.

6.5 *Specific applications:*

6.5.1 *Disinfestation*—One of the most widely studied processes utilizing fluidized bed treatment is the disinfestation of spices, leafy herbs and cereal grains. For grains **(25,26)**, encapsulated LiF is used to provide good agreement of dosimeter and product motion in the irradiator, with recovery by sieving. Thin film microdosimetry **(27,28)** using radiochromic or cellulose triacetate (CTA) films is also practicable. In this case, the film is inserted into a section of the grain and a microspectrophotometer used to evaluate the internal dose variation. This type of dosimetry is only of interest if bulk rather than surface dose is of concern. For the low doses involved in disinfestation, chemiluminescence in glutamine and salt added to the product, is appropriate **(29)**. Review articles have been presented for methods of dose determination in bulk particulate foodstuffs **(24,30)** (see ASTM F1355 and F1885), as well as in the use of the foodstuffs themselves as active dosimeters **(30)**.

6.5.2 *Blood*—There has been considerable experience at the lower end of this dose range (10 Gy) for the irradiation of blood **(31)**. Radiochromic dye solution and suspension of thermoluminescent lithium borate in water, both calibrated with standard Fricke dosimetry, have been used for calibration of a blood irradiator unit**(32)**. Although primarily gamma based, these dye techniques have also been used with 10 MeV electrons **(32,33)**.

6.5.3 *Aqueous streams*—For medium dose levels (5-10 kGy) in aqueous streams where the stream purity (specific heat) is known, calorimetric techniques can be employed to determine the average absorbed dose. Several resistance temperature devices at the inlet and outlet ducts of an electron beam wastewater treatment facility utilizing 1.5 MeV electrons have been used for this purpose **(34,35)**. Because a dose of 10 kGy results from 2.4 cal/g specific energy absorption, quite precise average delivered dose determinations can be made in this way with the short transit times in which very little conductive or convective cooling can take place between irradiation and measurement; e.g. 100 ms. Because of the relatively uniform behavior of wastewater disinfection with dose after removal of aqueous contaminants, the 4 % agreement of calculated and measured average doses reported in such continuous stream applications is quite adequate. Other examples of such calorimetric techniques are available in the literature **(33,34–36)** and have been well developed for dose distribution determinations in ducts used in the electron beam treatment of flue gases **(37)**.

6.5.4 *Industrial waste streams*—Among the developing application areas are sewage sludge hygienization and treatment of polluted wastes gases. For designing the treatment system using electron beam irradiation, dosimetry in the transported gases is effective to evaluate the average dose delivered. Dosimetric studies have been conducted using thin film dosimeters for measuring depth-dose distribution **(38)** and high-dose gas phase dosimetry can be accomplished by measuring the concentration of ozone formed in an irradiated pure oxygen flow system **(39)**.

7. Measurement Uncertainty

7.1 To be meaningful, a measurement of absorbed dose shall be accompanied by an estimate of uncertainty.

7.2 Components of uncertainty shall be identified as belonging to one of two groups:

7.2.1 Type A - those evaluated by statistical methods, or

7.2.2 Type B - those evaluated by other means.

7.3 Other ways of categorizing uncertainty have been widely used and may be useful for reporting uncertainty. For example, the terms *precision* and *bias* or *random* and *systematic* (non-random) are used to describe different categories of uncertainty.

NOTE 7—The identification of Type A and Type B uncertainties is based on methodology for estimating uncertainties published in 1995 by the International Organization for Standardization (ISO) in the Guide to the Expression of Uncertainty in Measurement **(40)**. The purpose of using this type of characterization is to promote an understanding of how uncertainty statements are arrived at and to provide a basis for the international comparison of measurement results.

NOTE 8—ISO/ASTM Guide 51707 defines possible sources of uncertainty in dosimetry performed in radiation processing facilities and offers procedures for estimating the magnitude of the resulting uncertainties in the measurement of absorbed dose using a dosimetry system. The document defines and discusses basic concepts of measurement, including estimation of the measured value of a quantity, "true" value, error and uncertainty. Components of uncertainty are discussed and methods are provided for estimating their values. Methods are also provided for calculating the combined standard uncertainty and estimating expanded (overall) uncertainty.

NOTE 9—If this practice is followed, the estimate of the expanded uncertainty of an absorbed dose determined by a radiochromic film dosimetry system, for example, could be less than 10 % for a coverage factor k = 2 (which corresponds approximately to a 95% level of confidence for normally distributed data).

8. Certification

8.1 *Documentation.* General articles as helpful guides appear in the bibliography **(41,42)**.

8.1.1 Establish a record and documentation system, which documents all dosimetry data from the time of facility installation, including testing procedures, process validation, and system maintenance history.

8.1.1.1 Record the measurements of performance, which qualify the dose delivering characteristics of the equipment. Record the date, time, value of the critical process parameters and the name of the machine operator.

8.1.1.2 Record dosimetry results and the values of the processing parameters affecting absorbed dose together with sufficient information identifying these parameters with specific production runs.

8.1.1.3 Record or reference the calibration and maintenance of equipment and instrumentation used to control or measure the absorbed dose delivered to the product. (See ISO/ASTM Guide 51261)

8.1.2 *Facility Records*

8.1.2.1 Record the dates and times of any facility maintenance, including specific components replaced. Record all equipment failures, the nature of the problem which caused the outage, and any corrective action taken.

8.2 *Review and Approve*

8.2.1 Review and approve all dosimetry records in accordance with an established quality control program.

8.2.2 Audit all documentation periodically to assure that records are accurate and complete.

8.3 *Retention of Records*

8.3.1 Retain all records at the facility and have them available for inspection as needed. Keep the files for a period of time specified by relevant authorities.

9. Keywords

9.1 Absorbed dose; electron beam; gamma ray; dosimetry; food processing; fluidized bed irradiation; fluid stream irradiation; electron disinfection/disinfestation/sterilization

ANNEX

(Informative)

A1. TYPICAL DOSIMETRY PROCEDURE FOR FLUIDIZED BED PROCESSING

A1.1 *Performance Verification With Thin Film Dosimeters.* Radiochromic dosimeters **(13)** may be used for performance verification of an electron beam fluidized bed system handling powders, seeds or aggregates. The dosimeters are fed through the processor with the product during the run, if recovery permits, or they may be run in the air bed without product to ease recovery before the run, if desired. Performance verification before a run is conducted with four to eight dosimeters, fed sequentially through the processor at the desired velocity and beam current combination. The dosimeters are then recovered, cleaned to remove any product contamination, annealed and read out. Investigators **(8)** have used a 3σ criterion for rejection of any of the data points in determining average dose. Five determinations, each taken with 8 radiochromic film dosimeters, over a 9 day period, on a system running at approximately the same current at a nominal 750 m min^{-1} bed velocity, are shown in Table A1.1. In this case, similar data

TABLE A1.1 Fluidized Bed Pilot Reproducibility

Date	Current (mA)	Dose (kGy)	Calculated Velocity (m min^{-1})	Average Velocity (m min^{-1})
15/7/1998	13.3	6.8 ± 1.3	757	761 ± 27
16/7/1998	13.3	7.2 ± 0.6	714	
21/7/1998	14.2	7.2 ± 0.7	763	
24/7/1998	13.4	6.7 ± 1.1	774	
24/7/1998	14.2	6.9 ± 1.3	796	
10/8/1999	14.0	3.2 ± 0.2	1693	1873 ± 99
01/9/1999	11.2	2.4 ± 0.4	1806	
07/9/1999	14.0	3.0 ± 0.6	1806	
19/10/1999	15.0	2.9 ± 0.2	2001	
21/10/1999	15.0	3.0 ± 0.5	1935	
25/10/1999	15.0	3.0 ± 0.3	1935	
14/1/200	15.0	3.0 ± 0.2	1935	

taken at different currents (dose rates) at higher velocities (1900 m min^{-1}) are shown in the second part of the table. In these runs the bed carried winter wheat at a similar loading.

A1.1.1 In Table A1.1, the first set of data (5 runs) taken in 1998 was run using a 5 cm x 30 cm unscanned beam from an ESI Electrocurtain® at an acceleration voltage of 230 kV. All runs were recorded at a product (hulled sesame seed) flow rate of 100 g s^{-1}. The treatment chamber is 76 mm wide and 54 mm deep for a cross sectional area of 41 cm^2. At the calculated bed velocity of 761 m min^{-1}, this yields an average product thickness of 100 g m^{-2} in an air bed thickness 5.4 cm × 12 g m^{-2} cm-1 = 65 g m^{-2}. If one assumes a bulk density for the seed of unity, the 100 cm^3 of product is moving in a bed volume of 41 × 12.7 x 100 cc or 5.2 x10^4 cm^3 for a product occupied volume in the bed of 0.2 %.

A1.1.2 The second set of data (7 runs) was recorded in 1999 in the same processor at an acceleration voltage of 225 kV. All of these runs were recorded at a product (hard winter wheat) flow rate of 70 g s^{-1}. At the bed velocity measured of 31 m s^{-1}, this yields an average product thickness 29 g m^{-2}. Using a bulk density of 1.4 g cm^{-3}, the 50 cm^3 of product is now in a bed volume of 41 × 31 ×10^2 or 1.3×10^5 cm^3 for a product occupied volume of 0.04 %

A1.1.3 For these two cases, the bed loading factors; i.e. for the sesame seeds and wheat, were 160 % and 45 % respectively. The loading factor is defined as the ratio of the weight of product transported to the weight of the carrier gas moved through the system per unit of time.

NOTE A1.1—The role played by the bed loading in affecting the transport velocity of these 5 milligram dosimeters was found to be quite significant. Increasing the bed loading results in decreased bed velocity and hence dosimeter velocities, and must be measured for each set of production conditions.

A1.2 *Real Time Radiation Monitoring.* This dosimetry procedure provides results with a standard deviation as shown in Table A1.1 of 6 to 20 percent, adequate for bulk processing application for process control. This facility also uses a real time radiation monitor **(43)** for detection and analysis of the bremsstrahlung generated in the window foil and its support frame, in order to log the performance of the electron source

during a run. With it, a continuous log of both machine operating voltage and beam current at the preset bed velocity is available for process quality assurance. This type of monitor is capable of providing dose delivery information with a much improved standard deviation and provides important real time verification of system performance, traceable to national standards through the use of the same film dosimetry.

A1.3 *Throughput.* The dosimetry used in these fluidized bed systems is critical for optimization of the irradiation duct geometry. Their relatively good processing power efficiency for high velocity product transport, provides excellent production capacity at modest power levels. For example, a 25 mA × 1 MeV system using a 1 meter (longitudinal) irradiation duct, can deliver 10 kGy at a product velocity of 500 m min^{-1}. When handling 500 g s^{-1}, such a 25 kW system will treat fluidized bed product at 1800 kg h^{-1} at this dose. For grain disinfestation at 0.8 kGy, the processor, now with a transverse irradiation duct, can handle 22,500 kg h^{-1}, now at a feed rate of 6.25 kg s^{-1}. Robust film dosimetry in the 0.1-10 kGy region is important for the control and monitoring required for the varied industrial uses of this process.

BIBLIOGRAPHY

(1) Tanaka, R., "Calculation of Longitudinal Dose Non-uniformity with Simultaneous Product Movement and Beam Scanning in Industrial Electron Irradiation", *Appl. Radiat. Isot.* **35**, 1089-1094 (1984).

(2) Weiss , D.E., Kalweit, H.W., Kensek, R.P., "Low Voltage electron-beam simulation using the ITS Monte Carlo code and calibration through radiochromic dosimetry," *Irradiation of Polymers Fundamentals and Technological Applications,* Clough, R.L., and Shalaby, S.W., (eds.), ACS Symposium Series 620, American Chemical Society, Washington, DC. (1996).

(3) Kovacs, A., Hargittai, P., Kaszanyiczki, L. and Foldiak, G., "Evaluation of Multipurpose Electron Irradiation of Packaged and Bulk Spices", *Appl. Radiat. Isot.* **45**, 783-788, (1994).

(4) TSI Inc., P.O. Box 64394, St. Paul, MN. 55164-0394. Model 8345/8346 Velociacalc®.

(5) Cleghorn, D.A., Nablo, S.V. and Ferro, D.N., "Performance of the Electron Beam Fluidized Bed Process for Disinfection and Disinfestation of Stored Products", Application of Accelerators in Research and Industry, ed J.L. Duggan and I.L. Morgan, American Institute of Physics Conference Proceedings, **576**, pp. 779-782 (2001).

(6) McLaughlin, W.L., "Novel Radiation Dosimetry Systems", Proc. of an Int'l. Symp. on High Dose Dosimetry for Radiation Processing, IAEA, Vienna, SM-314/39, (1990).

(7) Van Laere, K., Buysse, J., Berkven, P., "Alanine in High Rate Dosimetry: Spectrophotometric and Electrochemical Readout Procedures Compared with ESR", *Appl. Radiat. Isot.* **40**, 885-895, (1989).

(8) Nablo, S.V., Wood, J.C., Desrosiers, M.F. and Nagy, V.Y., "A Fluidized Bed Process for Electron Sterilization of Powders," *Radiat. Phys. Chem.* **52**, 479-485 (1998).

(9) Munoz , E., Adem, E., Burillo, G., Gleason, R. and Murrieta, H., "ESR Studies of Irradiated Ground Corn as a Dosimeter", *Radiat. Phys. Chem.* **43**, 311-313, (1994).

(10) Ehlermann , D.A.E., "The Suitability of Intrinsic and Added Materials as Dose Meters for Radiaiton Processing of Particulate Foods". *Radiat. Phys. Chem.* **36**, 609–612, (1990) *Appl. Radiat. Isot.* **40**, 885-895, (1989).

(11) ESR Dosimetry and Applications, ed. D.F. Regulla, A. Scharmann and W.L. McLaughlin, Pergamon Press, NY (1989).

(12) McLaughlin, W.L., "Solid Phase Chemical Dosimeters", pp. 219-252, *Sterilization by Ionizing Radiation*, Gaughran, ERL and Goudie, A. J., eds., Multiscience Publication Ltd., Montreal, Quebec (1974).

(13) Gehringer, P., Eschweiler, H. and Proksch, E., "Dose and Humidity Effects on the Radiation Response of Nylon-Based Radiochromic Film Dosimeters", *Appl. Radiat. Isot*, **31**, 595-605 (1980).

(14) McLaughlin , W.L., Humphreys, J.C., Hocken, Dene and Chappas, W.J., "Radiochromic Dosimetry for Validation and Commissioning of Industrial Radiation Processes", *Radiat. Phys. Chem.* **31**, 505-514, (1988).

(15) Janovsky , I., Hansen, J.W. and Cernoch, P., "A Polymer-Alanine film for measurement of radiation dose distributions", *Appl. Radiat. Isot.* **39**, 651 (1991).

(16) Grunewald, T. and Rudolf, M., "Semiconductor Diode Aided Dosimetry of the Irradiation of Pourable Bulk Material", *Food. Irr. Newsletter* **11**, 1, 42-47, (1987).

(17) Regulla , D. F., Schurmann, A. and Suess, H., "Dosimetry at a High Activity ^{60}Co Waste Treatment Facility", Proc. Int. Symp. on Radiation for a Clean Environment 465-476, IAEA-SM-194 (1975). See also Suess , A., Rosopulo, A., Borchert, H. Beck, T., Bauchhens, J. and Schurmann, G., "Experience with a Gamma-Ray Pilot Plant for the Irradiation of Sewage Sludge" ibid, 503-533.

(18) Schurman, G. and Regulla, D.F., "Determination of Dose and Irradiation Homogeneity in a Sewage Sludge Irradiation Plant", *Kerntechnik* , **20**, 323 (1978).

(19) Benny, P. G. and Bhatt, B.C., "Investigation of TL Properties of Sand Collected from Sludge as an "in situ" Dosimeter", *Appl. Radiat. Isot* **47**, 115-121, (1996). See also Benny, P.G., Bhatt, B.C. and Shah, M.R., "TL Dosimetry Using Extracted and Cleaned Sand to Measure Gamma-Ray Dose Rate at a Liquid Sludge Irradiation Facility", *Radiat. Phys. Chem.* **49**, 377-381 (1997).

(20) McKeever, S.W.S., " Thermoluminescence in Quartz and Silica", *Radiation Protection Dosimetry* **8**, 81-98 (1984).

(21) Kovacs , A., Wojnarovits, L., Kurucz, C., Al-Sheikhly, M. and McLaughlin, W.L., "Large Scale Dosimetry Using Dilute Methylene Blue Dye in Aqueous Solution", *Radiat. Phys. Chem.*, **52**, 539-542 (1998).

(22) Day , M.J. and Stein, G., "The Action of Ionizing Radiation on Aqueous Solutions of Methylene Blue", *Radiation Research* **6**, 666 (1975).

(23) Kovacs, A. and Miller, A., "Use of Ethanol-Monochlorobenzene Dosimeters at Electron Accelerators", *Radiat. Phys. Chem.* **36**, 709-713 (1990).

(24) Ehlermann, D.A.E., "Dose Distribution and Methods for its Determination in Bulk Particulate Food Materials" Health Impact, Identification and Dosimetry of Irradiated Foods, (Boegl, K.W., Regulla, D.F. and Suess, M. I. eds) Bundesgesundheitsamt, Berlin, ISH-Heft 125, 415-419 (1988).

(25) Tilton , E.W. and Brower, J.H., " Status of US Department of Agriculture Research on Irradiation Disinfestation of Grain and Grain Products", pp. 295-309, *Radiation Preservation of Food*, IAEA-SM-166/49, IAEA, Vienna (1973).

(26) Uribe, R.M., de la Piedad, B.A., Adem. E., and Reyes, L. J., "Dosimetry in a pilot Plant for bulk Disinfestation of Grain by Electron Irradiation Lithium Fluoride in Powdered Form", *Revista Mexicana de Fisica*, **26**, 421-427, (1980).

(27) Zakladnoy , G.A., Menshenin, A. Pertsovsky, E.S., Slimov, R.A.,

Cherepkov, V. G., Bogolyubov, B.F., Stanev, I.S., "Radiation Disinfestation of Grain in a Port Elevator with a Capacity of 400t/h", *Radiat. Phys. Chem.* **34**, 991-994, (1989).

(28) Tanaka , R. Uribe, R.M., de la Piedad, A. and Adem, E., "Basic Study of Electron Irradiation Technique in Disinfestation Process of Maize", JAERI-M, 83-239, (1984).

(29) Ehlermann , D.A.E. and Delincee, H., "Dosimetry and Process Control for Radiation Processing of Bulk Quantities of Particulate Foods", *Radiat. Phys. Chem.* **35**, 836-840, (1990).

(30) Ehlermann, D.A.E., "The Use of Particulate Foods as a Dosimeter in Irradiated Food", ibid, 420-424.

(31) Weeks, E., Rasmussen, I., Christensen, P., Botter-Jensen, L., "Extracorporeal Irradiation of Blood for Immuno-Suppresive Therapy: Equipment, Dosimetry and Medical Experience, Riso-M-1261 (1971).

(32) Hjortenberg, P.E. and McLaughlin, W.L., "Use of Radiochromic Dye Systems for Dosimetry", Proceedings of the Regional Conference on Radiation Protection, Yavne, Israel, (Feige, Y. and Schlesinger, T. eds) 122-140, (1973).

(33) Nickelsen , M.G., Cooper, W. J., Lin, K., Kurucz, C.N. and Waite, T.D., "High Energy Electron Beam Generation of Oxidants for the Treatment of Benzene and Toluene in the Presence of Radical Scavengers", *Water Research*, **28**, 1227-1237 (1994).

(34) Farooq, S., Kurucz, C.N., Waite, T.D. and Cooper, W.J., "Disinfection of Wastewaters: High Energy Electron vs. Gamma Irradiation", *Water Research*, **27**, 1177-1184, (1993).

(35) Kurucz, C.N., Waite, T.D. and Cooper, W.J., "The Miami Electron Beam Research Facility: A Large Scale Wastewater Treatment Application", *Radiat. Phys. Chem.*, **45**, 299-308 (1995).

(36) Nickelsen , M.G., Cooper, W.J. Kurucz, C.N. and Waite, T.D., "Removal of Benzene and Selected Alkyl-Substituted Benzenes from Aqueous Solution Utilizing Continuous High Energy Electron Irradiation", *Environmental Science and Technology*, **26**, 144-151, (1991).

(37) Wittig, S., Spiegel, G. Platzer, K.H. and Willibald, U., "The Performance Characteristics of the Electron Beam Technique: Detailed Studies at the (ITS) Flue Gas Facility", *Radiat. Phys. Chem.* **31**, 83-93 (1988).

(38) Hakoda , T., Sunaga, H., Takizawa, H., Hirota, K. and Kojima, T., "Absorbed Dose Rate Distribution in 300 keV Electron Beam Irradiation Field for Wasts-gas Purification", *Radioisotopes* **53**, 59-69 (2004) (in Japanese).

(39) Namba, H., Tokunaga, O. and Suzuki, N., "Oxygen Gas Dosimeter for Flow System", *Appl. Radiat. Isot.* **40**, 53-56 (1989)

(40) "Guide to the Expression of Uncertainty in Measurement", International Organization for Standardization, 1995 ISBN 92-67-10188-9. Available from the International Organization for Standardization, 1 rue de Varembé, Case Postale 56, CH-1211, Geneva 20, Switzerland

(41) *Dosimetry for Food Irradiation, Technical Report Series No. 409*, International Atomic Energy Agency, Vienna, 2002.

(42) McLaughlin, W.L., Boyd, A.W., Chadwick, K.H., McDonald, J.C. and Miller, A., *Dosimetry for Radiation Processing*, Taylor and Francis, New York, 1989.

(43) Kneeland, D.R., Nablo, S.V., Weiss, D.E. and Sinz, T.E., "Industrial Use of the Real Time Monitor for Quality Assurance in Electron Processing", *Radiat. Phys. Chem.*, **55**, 429-436 (1999)