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# Dosimetry for Food Irradiation



### DOSIMETRY FOR FOOD IRRADIATION

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## DOSIMETRY FOR FOOD IRRADIATION

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#### **FOREWORD**

A Manual of Food Irradiation Dosimetry was published in 1977 under the auspices of the IAEA as Technical Reports Series No. 178. It was the first monograph of its kind and served as a reference in the field of radiation processing and in the development of standards. While the essential information about radiation dosimetry in this publication has not become obsolete, other publications on radiation dosimetry have become available which have provided useful information for incorporation in this updated version.

There is already a Codex General Standard for Irradiated Foods and an associated Code of Practice for Operation of Irradiation Facilities used for Treatment of Food, issued in 1984 by the Codex Alimentarius Commission of the FAO/WHO Food Standard Programme. The Codex Standard contains provisions on irradiation facilities and process control which include, among other requirements, that control of the processes within facilities shall include the keeping of adequate records including quantitative dosimetry. Appendix A of the Standard provides an explanation of process control and dosimetric requirements in compliance with the Codex Standard. By 1999, over 40 countries had implemented national regulations or issued specific approval for certain irradiated food items/classes of food based on the principles of the Codex Standard and its Code of Practice. Food irradiation is thus expanding, as over 30 countries are now actually applying this process for the treatment of one or more food products for commercial purposes. Irradiated foods are being marketed at retail level in several countries.

With the increasing recognition and application of irradiation as a sanitary and phytosanitary treatment of food based on the provisions of the Agreement on the Application of Sanitary and Phytosanitary Measures of the World Trade Organization, international trade in irradiated food is expected to expand during the next decade. It is therefore essential that proper dosimetry systems are used to ensure the compliance of trade in irradiated food with national and international standards.

In view of the foregoing, FAO and the IAEA, through their Joint FAO/IAEA Division of Nuclear Techniques in Food and Agriculture, Vienna, considered it timely to revise the Manual of Food Irradiation Dosimetry. A Consultants' Meeting was convened in Vienna from 27 to 30 October 1998 to revise the Manual. It was attended by R. Chu, MDS Nordion, Kanata, Canada; I. de Bruyn, Atomic Energy Corporation of South Africa Ltd, Pretoria; D.A.E. Ehlermann, Federal Research Centre for Nutrition, Karlsruhe, Germany; W.L. McLaughlin, National Institute of Science and Technology, Gaithersburg, Maryland, USA; P. Thomas, Bhabha Atomic Research Centre, Mumbai, India. Subsequently, D.A.E. Ehlermann incorporated contributions from the other participants and from IAEA staff members, and finally K. Mehta revised and edited the document.

#### EDITORIAL NOTE

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An appendix, when included, is considered to form an integral part of the standard and to have the same status as the main text. Annexes, footnotes and bibliographies, if included, are used to provide additional information or practical examples that might be helpful to the user.

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#### 1. GENERAL ASPECTS OF FOOD IRRADIATION

#### 1.1. INTRODUCTION

The world food trade is entering a new era following the conclusion of the GATT Uruguay Round. In particular, the foundation of the World Trade Organization (WTO) and the Agreements on the Application of Sanitary and Phytosanitary (SPS) Measures and on Technical Barriers to Trade (TBT) will have a profound effect on trade in food and agricultural commodities [1]. The SPS Agreement is designed to protect the health and safety of humans, animals and plants and to harmonize control measures between countries. It recognizes standards, guidelines and recommendations of competent intergovernmental bodies such as the Codex Alimentarius Commission, the International Plant Protection Convention and the International Office of Epizootics, to assist the WTO in settling trade disputes. The TBT Agreement was designed to protect the quality of traded goods and the rights of consumers.

Thus, the Codex General Standard for Irradiated Foods and its associated Code of Practice [2], adopted by the Codex Alimentarius Commission in 1983, will play an even greater role in the future with regard to trade in irradiated food. In addition, the International Consultative Group on Food Irradiation (ICGFI), established under the aegis of the FAO, IAEA and WHO in 1984, has issued a number of guidelines and recommendations (see lists in the Bibliography) to strengthen control procedures in the operation of irradiation facilities based on the principles of the Codex Standard. On the basis of the Codex Standard and the relevant recommendations of the ICGFI, a Model Regulation on Food Irradiation for Asia and the Pacific was developed by regulatory officials from this region. This model was endorsed by the ICGFI and has provided the basis for harmonization of regulations in countries in Asia and the Pacific, Africa, Latin America and the Middle East [3]. Thus, international trade in irradiated food can occur with no obstacles from the regulatory point of view.

International trade in irradiated food is expanding. Spices and dried vegetable seasonings are irradiated for commercial purposes in some 20 countries, with volumes increasing from about 5000 t in 1990 to over 90 000 t in 2000, approximately half of which was done in the USA. Irradiation as a quarantine treatment of fresh fruits and vegetables and as a method to ensure the hygienic quality of food of animal origin is increasingly accepted and applied. International trade in these commodities is highly likely in the near future.

The effectiveness of processing of food by ionizing radiation depends on proper delivery of absorbed dose and its reliable measurement. For food destined for international trade, it is of the utmost importance that the dosimetry techniques used for dose determination are carried out accurately and that the process is monitored

in accordance with the internationally accepted procedures. Such dosimetry should be traceable to national or international standards, and thus the quality of the dosimetry results allows for the evaluation of the process reliability. This also provides an independent control of the process.

It was considered most timely to produce this book, which is in essence a revision of the Manual of Food Irradiation Dosimetry issued by the IAEA in 1977 [4], in view of the foregoing and considering recent developments, such as:

- (a) More experience gained with the current dosimetry systems and the development of a few new ones,
- (b) The establishment of several irradiation facilities and the experience gained from these,
- (c) The establishment of a special subcommittee of the American Society for Testing and Materials (ASTM) for dosimetry of radiation processing which has developed more than 20 relevant standards,
- (d) The publication of a monograph dealing exclusively with dosimetry for radiation processing [5].

#### 1.2. CODEX ALIMENTARIUS PROVISIONS

At present, the Codex General Standard for Irradiated Foods [2] (presently under revision) uses the concept of 'overall average dose' and the value of 10 kGy as the maximum allowable value, which is based on toxicological findings [6]. This concept refers to food in general only, and does not make any reference to the setting of specific limiting values for the lowest or highest absorbed dose or the restriction to limited food classes or to individual foodstuffs. However, the definition of 'overall average dose' as given in this standard is not suitable for measurements in production situations and, therefore, for regulation and enforcement by authorities. In the light of the findings of an FAO/IAEA/WHO Joint Study Group on High Dose Food Irradiation [7, 8], the General Standard is being modified under the Codex procedure. Thus, the terminology of 'overall average dose' and setting of the maximum dose will be eliminated from the standard. This would finally remove the justification for any regulation of this kind. However, it would not remove the need for reliable dosimetry in facility operation and process control. Furthermore, food production and handling are also governed by other regulations and standards, in particular those of the Codex Alimentarius, and the principles of the Hazard Analysis and Critical Control Point (HACCP) also apply to the technology of food processing by ionizing radiation.

The Codex General Standard also requires that an irradiation facility be licensed and registered for the purpose of food processing, be staffed with trained and competent personnel, and keep adequate records including those related to dosimetry.

To recognize irradiation facilities which operate under the proper supervision of national food control authorities, ICGFI established in 1991 an 'International Inventory of Authorized Food Irradiation Facilities'. The radiation absorbed dose is a key quantity in the processing of food by ionizing radiation, and registering a facility is a prerequisite for proficiency in operation, including dosimetry.

#### 1.3. INTERNATIONAL TRADE ASPECTS

The emergence of the WTO and the establishment of several associated agreements have also had significant consequences for international trade in food, including irradiated food [1, 9]. Globalization and consumer demands for a secure and safe food supply have placed new challenges on controlling such trade. In particular, the consumers' right to know requires informative labelling and associated regulations and quality standards, communication and readily available information. In the processing of food by radiation, dosimetry and the communication of results in a reliable and trustworthy manner become particularly important. Therefore, bilateral and international arrangements must provide for adherence to reasonable and scientifically sound principles. At present, this is achieved through several harmonization efforts [1].

Many aspects of food processing are strictly regulated because of consumer concern about the correct application of allowable processes. The processing of food by radiation is no exception. In order to establish confidence in the entire process, several national and international organizations have developed relevant recommendations, standards and guidelines. First, the Codex Alimentarius General Standard on Irradiated Foods [2] sets the general requirements. However, technical details are covered elsewhere, for example, ICGFI has developed guidelines for good irradiation practice (GIP) which summarize the practical and technological results in radiation processing of food (see Bibliography). Under the agreement of the WTO, the rules of the Codex Alimentarius Standard have now become the technical reference for the quality requirements in international trade in food, and hence mutually accepted and binding standards.

In radiation processing, the essential quantity is 'radiation absorbed dose'. Therefore, dosimetry is of crucial importance in radiation processing of food. The standards of the International Organization for Standardization (ISO) are mandatory for the members of ISO. Several ISO standards for dosimetry in radiation processing are already in effect. Such standards can help the industries concerned and the international trade in their business. An example is an ISO standard on sterilization of health care products [10], which regulates a business of great economic importance. Several standards on radiation dosimetry in food processing and related areas have been developed by the ASTM (see the Bibliography), and several of these

have now been recognized by the ISO and thus become mandatory. These elucidate the importance of reliable dosimetry in radiation processing of food.

At present, there are no easy means available at international entry points to determine whether an imported food item has received the correct irradiation treatment. Consequently, such assurance needs to be based, through shipping documents and labelling, on the dosimetry and process control records generated at the entry facility, which should be in accordance with international standards. This requires bilateral agreements to allow for such inspection and the application of mutually accepted standards. Formal accreditation of standards laboratories and of the quality control systems operated in an irradiation facility results in mutual equivalence and recognition of dose measurements, and hence contributes to international trade in radiation processed products and helps to remove trade barriers [11]. Several national regulations already allow for imports of irradiated foods provided that the requirements of the importing country have been met at the facility abroad; for example, the standards applied there for dosimetry and process control must be commensurate with the national regulations.

#### 1.4. ADVISORY TECHNOLOGICAL DOSE LIMITS

These dose limits are recommended by the ICGFI solely on the basis of technological data available in the literature. The lowest absorbed dose required to achieve a desired effect is termed in this book the lowest acceptable dose. The highest absorbed dose acceptable is determined by the sensory and functional properties of the product that must not be impaired by too high a dose. Their values are based on food irradiation experience and are specified by food technologists for each combination of process and product on the basis of the results obtained in experimental research preceding commercial scale application. It should be kept in mind, however, that the radiation effect on a product is a continuous function and not a step function of the applied dose, so that the lowest acceptable dose is not stringently defined but must be derived from other considerations. The collection of such results (see Bibliography for ICGFI publications) defines GIP in radiation processing of food, which is the same as good manufacturing practice (GMP) in other food processing. GIP is always considered an integral part of GMP. These advisory technological dose limits are given in Table I [3].

Much has been published on food irradiation, and several international conferences sponsored by FAO/IAEA/WHO have taken place. 1 It is not the aim of

<sup>&</sup>lt;sup>1</sup> At Karlsruhe in 1966, at Bombay in 1972, at Wageningen in 1977, at Washington in 1985, at Geneva in 1988, at Aix-en-Provence in 1993 and at Antalya in 1999; the proceedings of these conferences are available through the IAEA.

TABLE I. ADVISORY TECHNOLOGICAL DOSE LIMITS [3]

Classes of food	Purpose	Maximum dose (kGy)	ICGFI <sup>a</sup> document No.
Class 1: Bulbs, roots and tubers	To inhibit sprouting during storage	0.2	8
Class 2: Fresh fruits and vegetables (other than Class 1)	To delay ripening Insect disinfestation Shelf life extension Quarantine control <sup>b</sup>	1.0 1.0 2.5 1.0	6 3, 7, 17 6 7, 13, 17
Class 3: Cereals and their milled products, nuts, oil seeds, pulses, dried fruits	Insect disinfestation Reduction of microbial load	1.0 5.0	3, 20 3, 20
Class 4: Fish, seafood and their products (fresh or frozen)	Reduction of certain pathogenic micro-organis Shelf life extension Control of infection by parasites <sup>c</sup>	5.0 sms <sup>c</sup> 3.0 2.0	10 10 10
Class 5: Raw poultry and meat and their products (fresh and frozen)	Reduction of pathogenic micro-organisms <sup>c</sup> Shelf life extension Control of infection by parasites <sup>c</sup>	7.0 3.0 2.0	4 4 4
Class 6: Dry vegetables, spices, condiments, animal feed, dry herbs and herbal teas	Reduction of certain pathogenic micro-organisms <sup>c</sup> Insect disinfestation	10.0	5, 19 5, 19
Class 7: Dried food of animal origin	Disinfestation Control of moulds	1.0 3.0	9 9
Class 8: Miscellaneous foods, including, but not limited to, honey, space foods, hospital foods, military rations spices, liquid egg, thickeners	Reduction of micro-organisms Sterilization Quarantine control	>10 >10 >10	

<sup>&</sup>lt;sup>a</sup> See list of ICGFI documents in the Bibliography.

#### **Notes:**

- 1. Product grouping into classes (except Class 8) is on the basis of similarity of chemical compositions.
- 2. The maximum dose limits have been set for good irradiation practice and not from a food safety viewpoint.

b The minimum dose may be specified for particular pests. For fruit flies, the minimum dose is at least 0.15 kGy.

<sup>&</sup>lt;sup>c</sup> The minimum dose may be specified depending on the objective of the treatment to ensure the hygienic quality of food.

this book to provide a full bibliography on the subject; however, more detailed information on food irradiation and effects achieved may be found elsewhere [12–17] and by reference to some of the published food irradiation bibliographies<sup>2</sup>.

The dose ranges given in the literature should not be understood to be rigid; the lowest acceptable dose may vary in either direction depending on the conditions of production and harvesting, on the state of ripeness and on the environmental conditions. This is also true for the maximum tolerable dose, which may be quite low in some applications in order to avoid damage to radiation sensitive products. Consequently, any reference to such values is as 'advisory technological dose limits' only. Such dose data are, in principle, not suitable for regulatory purposes, but only for guidance.

The ranges of dose commonly used in food irradiation to achieve various effects can be classified as given in the following sections.

#### **1.4.1.** Applications at low dose levels (10 Gy–1 kGy)

Sprouting of potatoes, onions, garlic, shallots, yams, etc. can be inhibited by irradiation in the dose range 20–150 Gy. Radiation affects the biological properties of such products in such a way that sprouting is appreciably inhibited or completely prevented. Physiological processes such as ripening of fruits can be delayed in the dose range 0.1–1 kGy. These processes are a consequence of enzymatic changes in the plant tissues.

Insect disinfestation by radiation in the dose range 0.2–1 kGy is aimed at preventing losses caused by insect pests in stored grains, pulses, cereals, flour, coffee beans, spices, dried fruits, dried nuts, dried fishery products and other dried food products. A minimum absorbed dose of about 150 Gy can ensure quarantine security against various species of tephretid fruit flies in fresh fruits and vegetables, and a minimum dose of 300 Gy could prevent insects of other species from establishing in non-infested areas. In most cases irradiation either kills or inhibits further development of different life-cycle stages of insect pests.

The inactivation of some pathogenic parasites of public health significance such as tapeworm and trichina in meat can be achieved at doses in the range 0.3–1 kGy.

#### **1.4.2.** Applications at medium dose levels (1–10 kGy)

Radiation enhances the keeping quality of certain foods through a substantial reduction in the number of spoilage causing micro-organisms. Fresh meat and seafood, as well as vegetables and fruits, may be exposed to such treatments with

<sup>&</sup>lt;sup>2</sup> For example, Bibliography on Irradiation of Foods, Bundesforschungsanstalt für Ernährung, Karlsruhe, Germany (published at irregular intervals until No. 40 in 1996, now available on-line at <a href="http://www.dainet.de/8080/BFELEMISTW/SF">http://www.dainet.de/8080/BFELEMISTW/SF</a>).

doses ranging from about 1 to 10 kGy, depending on the product. This process of extending the shelf life is sometimes called 'radurization'.

Pasteurization of solid foods such as meat, poultry and seafoods by radiation is a practical method for elimination of pathogenic organisms and micro-organisms except for viruses. It is achieved by the reduction of the number of specific viable non-spore-forming pathogenic micro-organisms such that none is detectable in the treated product by any standard method, for which doses range between 2 and 8 kGy. The product will usually continue to be refrigerated after the radiation treatment. This process of improving the hygienic quality of food by inactivation of food-borne pathogenic bacteria and parasites is sometimes called 'radicidation'. This medium dose application is very similar to heat pasteurization, and is hence also called radiopasteurization.

#### **1.4.3.** Applications at high dose levels (10–100 kGy)

Irradiation at doses of 10–30 kGy is an effective alternative to the chemical fumigant ethylene oxide for microbial decontamination of dried spices, herbs and other dried vegetable seasonings. This is achieved by reducing the total microbial load present in such products including pathogenic organisms.

Radiation sterilization in the dose range 25–70 kGy extends the shelf life of precooked or enzyme inactivated food products in hermetically sealed containers almost indefinitely. This is valid independent of the conditions under which the product is subsequently stored as long as the package integrity is not affected. This effect is achieved by the reduction of the number and/or activity of all organisms of food spoilage or public health significance, including their spores, to such an extent that none are detectable in the treated product by any recognized method. This process is analogous to thermal canning in achieving shelf-stability (long term storage without refrigeration) and is sometimes called 'radappertization'.

#### 1.5. NEED FOR DOSIMETRY

In all the various guidelines and standards developed for food irradiation, the activities of principal concern are process validation and process control. The objective of such formalized procedures is to establish documentary evidence that the irradiation process has achieved the desired results. The key element of such activities is inevitably a well characterized reliable dosimetry system that is traceable to recognized national and international dosimetry standards. Only such dosimetry systems can help establish the required documentary evidence. In addition, industrial radiation processing such as irradiation of foodstuffs and sterilization of health care products are both highly regulated, in particular with regard to dose. Besides,

dosimetry is necessary for scaling up processes from the research level to the industrial level. Thus, accurate dosimetry is indispensable.

#### 1.6. OBJECTIVES

The purposes of this book are first to recognize the importance of radiation dosimetry in food irradiation processing and to define the role that it must fulfil for successful implementation of the technology. The book also describes how dosimetry should accomplish its role, by providing details of the dosimetry procedures for process validation and process control. These methods and procedures must be accurate, practical, easy to use, inexpensive and, above all, acceptable to the various national regulatory agencies concerned with the wholesomeness of processed foodstuffs and with the control of the production of and the trade in food.

It is expected that the dosimetry personnel at the irradiation facility have sufficient training and understanding of the dose measurement processes to be able to follow such procedures that are part of a quality assurance plan. It is also expected that the owner of the facility appreciate the importance of dosimetry and be committed to supporting financially the various dosimetry related activities commensurate with the commercial scope of the facility.

#### 1.7. SCOPE

Emphasis is laid in this book on the dosimetry techniques most commonly used at present in food irradiation. The book deals with all aspects of dosimetry related to radiation processing of food at an irradiation facility. Section 2 describes fundamentals of dosimetry, including relevant aspects of the interaction of radiation with matter, various classes of dosimeters, selection criteria for dosimetry systems and the practical use of such dosimeters. Section 3 deals with principles of the various types of irradiators suitable for food processing; this includes aspects of irradiator design for optimizing the irradiation process, the selection of the various radiation sources and the relevant operations during the irradiation process. Section 4 describes the initial qualification of an irradiation facility and the process qualification necessary for each category of product. The objective of facility qualification is to establish baseline information for a new facility over the full range of process parameters. On the other hand, the objective of process qualification is to establish values for all process parameters for a given treatment and the conditions for their control. This is best accomplished through measurement of dose and dose distribution in representative product arrangements (dose mapping). Section 5 describes the operation of irradiation facilities, including routine processing, use of dosimetry in process control and the documentation that should be maintained at an irradiation facility. It also includes some aspects of inventory control, product testing and the principles of the HACCP system. Section 6 deals with various kinds of dosimetry systems, including those that are classification based on their areas of application and their relative quality. It also refers to procedures for calibration and test services, and describes some frequently used dosimetry systems. This is followed by two Appendices: Appendix I giving detailed information on dosimetry principles for the two most commonly used dosimetry systems (Fricke and polymethylmethacrylate (PMMA)), and Appendix II describing the quality control measures for one of the most frequently used measurement instruments, namely the spectrophotometer.

Owing to the practical nature of the book, the References are not intended to cover the field of dosimetry in a complete scientific manner but to guide the reader to some relevant publications for information and for a fundamental understanding. For further information, the book also includes a Bibliography that lists available standards and GIPs related to dosimetry for food irradiation. Annex I describes a few of the currently operating food irradiation facilities, while Annex II gives a brief 'check list' of dosimetry requirements and its applications at a food irradiation facility. A glossary providing practical help to the reader completes the book.

#### 2. FUNDAMENTALS OF DOSIMETRY

#### 2.1. INTRODUCTION

The success of radiation processing of food depends to a large extent on the ability of the processor:

- (a) To measure the absorbed dose delivered to the food product (through reliable dosimetry);
- (b) To determine the dose distribution patterns in the product package (through process qualification procedures);
- (c) To control the routine radiation process (through process control procedures).

It is necessary that personnel responsible for the operation of these facilities have a basic understanding of the radiation physics/engineering and dosimetry involved [2, 5, 18, 19]. Furthermore, it is indispensable that the parties involved, namely the management of an irradiation facility and the supplier or producer of the food product requesting processing by radiation, have a thorough understanding of the specific food engineering problems involved (Section 1). This helps them to

appreciate the importance of the radiation physics/engineering, dosimetry and process control.

Since the radiation absorbed dose is the quantity which relates directly to the desired effect in a specific food, the need for suitable and accurate dose measurement techniques must not be underestimated. This is best appreciated by realizing the consequences of using inadequate techniques, causing under- or overexposure of the product and the resulting failure to administer an effective treatment. The consequences to the processor can be both legal and economic, while the consumer may not only suffer an economic loss by having to discard an inadequately treated product but also lose confidence in the irradiation process.

For food processing, intense radiation sources are used, which may be electron accelerators, X ray machines or radionuclide irradiators containing either  $^{60}$ Co or  $^{137}$ Cs sources. The source geometry is related to the method of generating the radiation; monodirectional and scanned beams for electrons and X rays, while  $\gamma$  radiation from rectangular plaque or cylindrical sources for radionuclide irradiators is emitted isotropically (see Section 3 for a description of irradiator designs). The radiation energy limits for the sources suitable for food irradiation are approximately 0.1 and 10 MeV. The approximate range of absorbed dose used in food processing is from 0.01 to 100 kGy [12–15, 18] (see also Section 1.4 and Table I). This dose range cannot be covered by a single dosimetry system, therefore more than one system may be needed at a facility (Table II). Process load<sup>3</sup> geometries are generally confined to those conventional shapes and sizes currently used in the commercial packaging of food (e.g., rectangular cartons containing cylindrical, spherical or rectangular unit packages such as food drums and cans, bulk fruit, onions or potatoes, or boxes of meats, vegetables, cereals, spices or grain).

Except for differences in dose level and package size, the dosimetry and process control methods presented here are quite similar to those used in other radiation research and processing applications [5, 20], such as polymer modification [5, 21], sterilization of health care products [5, 21–23], and those in agriculture [5, 12–15, 24].

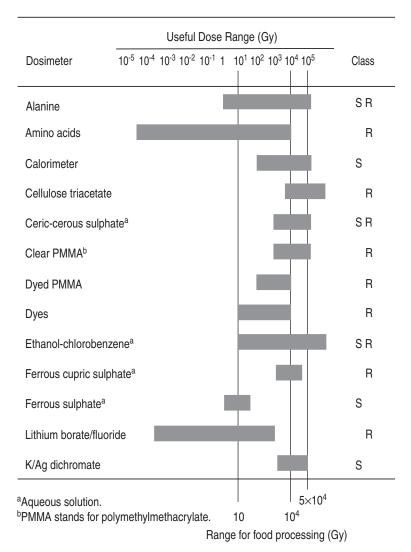
#### 2.2. ABSORBED DOSE

In the processing of foodstuffs by radiation, reliance is placed on the radiation quantity 'absorbed dose' to obtain accurate and meaningful information about the relevant radiation effects. The regulatory body or any other group responsible for the acceptance of the foodstuffs requires information that demonstrates that every

 $<sup>^3</sup>$  Process load is defined as volume of material with a specified loading configuration irradiated as a single entity.

TABLE II. USEFUL DOSE RANGES FOR VARIOUS ROUTINE AND REFERENCE STANDARD DOSIMETERS

(Classes: R, routine; S, reference standard)



part of the process load under consideration has been treated within the range of acceptable absorbed dose limits.

The absorbed dose (sometimes referred to as 'dose'), D, is the amount of energy absorbed per unit mass of irradiated matter at a point in the region of interest. It is defined as the mean energy,  $d\overline{\epsilon}$ , imparted by ionizing radiation to the matter in

a volume element divided by the mass, dm, of that volume element (for a rigorous definition see Ref. [25] or any ASTM Standard in the Bibliography):

$$D = \frac{d\overline{\epsilon}}{dm} \tag{1}$$

The SI derived unit of absorbed dose is the gray (Gy), which replaced the earlier unit of absorbed dose, the rad,

$$1 \text{ Gy} = 1 \text{ J/kg} = 100 \text{ rad}$$

The absorbed dose rate,  $\dot{D}$ , is defined as the rate of change of the absorbed dose with time:

$$\dot{D} = \frac{\mathrm{d}D}{\mathrm{d}t} \tag{2}$$

In practical situations, D and  $\dot{D}$  are measurable only as average values in a larger volume than is specified in the definitions, since it is generally not possible to measure these quantities precisely in a very small volume in the material. In this book, the absorbed dose is considered to be an average value, either as measured in the sensitive volume of the dosimeter used if it is of appreciable size or existing in its immediate vicinity if the dosimeter is very small or thin, where cavity theory is applicable [5, 26, 27].

For any given irradiation conditions, it is necessary to specify the absorbed dose in the particular material of interest because different materials have different radiation absorption properties. For food irradiation, the material of interest is generally specified as water. With regard to radiation interaction properties, most foods behave essentially as water regardless of their water content.

#### 2.3. INTERACTION OF RADIATION WITH MATTER

#### 2.3.1. Physical aspects of radiation absorption

When high energy X rays,  $\gamma$  rays or electrons are incident on a medium, multiple interactions occur that give rise to secondary particles; the interactions consist almost entirely of ionization that produces secondary electrons and photons of lower energies [27]. These particles go on to produce further interactions, thus producing the so-called cascading effect.

In the case of photons ( $\gamma$  rays from  $^{137}$ Cs and  $^{60}$ Co, and X rays) used in food irradiation, the main interactions are due to the Compton effect — the inelastic scattering of the incoming photons by atomic electrons. The photons are scattered and

secondary electrons are knocked off the atoms. Both the scattered photon and the secondary electron emerge at various angles, each with kinetic energy lower than that of the incident photon. This process predominates at photon energies between 0.05 and 10 MeV in low atomic number media, such as water. If the scattered photons have sufficient energy, they may undergo further Compton scattering.

Lower energy photons, those near the binding energy of the orbital electrons, undergo photoelectric absorption, which results in the emission of photoelectrons owing to ejection of bound orbital electrons. The kinetic energy of the ejected photoelectron is equal to the difference between the energy of the incident photon and the binding energy of the electron. Accompanying photoelectron ejection is an emission of characteristic X rays, an effect known as atomic fluorescence. In competition with this effect, especially for low atomic number materials, is the Auger effect, i.e. the ejection of other orbital electrons during the readjustment of the atomic electron orbits. For food irradiation by photons, the photoelectric effect is significant only for constituents of higher atomic number.

At high incident photon energies, greater than the sum of the rest masses of electrons and positrons (>1.02 MeV), pair production may occur. This portion (1.02 MeV) of the photon energy is converted into mass in the form of an electron and a positron, and the remaining energy appears as the kinetic energy of the two particles. The probability of such pair production interaction increases with the incident photon energy and with the square of the atomic number of the irradiated material. After slowing down, the positron recombines with an electron, resulting in annihilation radiation, i.e. the simultaneous emission of two photons each of energy 0.51 MeV. Figure 1 shows cross-sections for these three types of interactions for water as a function of photon energy [28].

In the case of electrons, their interactions with irradiated material give rise to secondary particles which are mainly lower energy electrons, mostly going forward at various angles to the primary beam direction. These arise from inelastic scattering and energy absorption processes. These secondary electrons go on to produce further electrons along their tracks until the energy is finally dissipated by molecular excitation and thermal processes at very low energies. The higher energy electrons, particularly those above several million electronvolts, can produce bremsstrahlung, which consists of photons emitted due to the loss of energy of fast electrons as they are slowed or deflected in their passage through the electric fields of absorbing atoms. The sum of the amount of energy loss due to this radiation process,  $(dE/dx)_{rad}$ , and that due to inelastic collision processes resulting in secondary electrons,  $(dE/dx)_{col}$ , is the total electron stopping power of the irradiated material, for the given incident electron energy. The ratio of  $(dE/dx)_{rad}$  to  $(dE/dx)_{col}$  is approximately proportional to the energy of the incoming electrons and the atomic number of the material. In food irradiation by electrons, bremsstrahlung production is negligible (except perhaps at electron energies approaching 10 MeV incident on materials containing higher

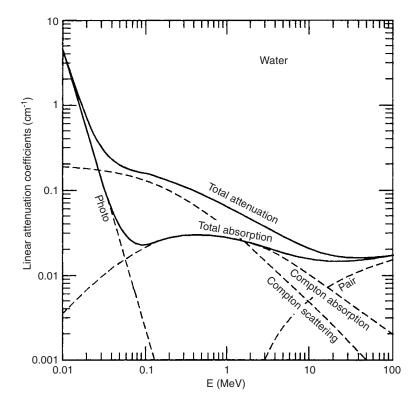


FIG. 1. Mass attenuation coefficient for photons in water [28].

atomic number constituents, as in the case of metallic containers). In most cases, energy deposition as a result of irradiation with electron beams is mainly due to secondary electron production and the result of absorption of that energy through secondary processes.

#### 2.3.2. Depth-dose distribution

The process of energy transfer from photons to the irradiated medium takes place in two distinct stages:

- (a) Interaction of photons via several processes as discussed above (mainly Compton, pair production and photoelectric effect) which set secondary (high energy) electrons in motion; this transfer takes place at the point of interaction.
- (b) Transfer of energy from these secondary electrons to the medium through excitation and ionization of the atoms of the medium; these transfers take place over a certain distance.

If the incident radiation is essentially monoenergetic and the angle of incidence at the irradiated surface is approximately perpendicular and monodirectional, there is initially a marked increase (buildup) of energy deposition (dose) near the incident surface. This region extends up to the depth corresponding to the average range of the first interaction secondary electrons. This is then followed by an exponential decay of dose to greater depths as shown in Fig. 2 [29, 30]. The approximate value of the buildup depth, in units of millimetres of water and mass per unit area<sup>4</sup>, is given below for different photon energies,

```
 \begin{array}{lll} ^{137}\text{Cs } \gamma \text{ rays } (E_{\gamma} = 0.66 \text{ MeV}) & 3 \text{ mm of water } (= 0.3 \text{ g/cm}^2, 3 \text{ kg/m}^2) \\ ^{60}\text{Co } \gamma \text{ rays } (E_{\gamma} \approx 1.25 \text{ MeV})^5 & 5 \text{ mm of water } (= 0.5 \text{ g/cm}^2, 5 \text{ kg/m}^2) \\ 4 \text{ MeV X rays} & 10 \text{ mm of water } (= 1.0 \text{ g/cm}^2, 10 \text{ kg/m}^2) \\ 6 \text{ MeV X rays} & 16 \text{ mm of water } (= 1.6 \text{ g/cm}^2, 16 \text{ kg/m}^2) \\ 10 \text{ MeV X rays} & 30 \text{ mm of water } (= 3.0 \text{ g/cm}^2, 30 \text{ kg/m}^2) \\ \end{array}
```

If the incident photon energy spectrum is fairly broad (e.g., for X rays) or the angles of incidence are widely varying (e.g., with a radionuclide plaque source of extended size close to an absorber), there is no appreciable dose buildup region. There is, instead, an essentially exponential decrease in dose with depth due to attenuation beginning at the incidence surface (in fact, the decrease is pseudo-exponential as the superposition of several exponential curves is not exactly exponential). The shape of the depth–dose distribution in the irradiated material depends on a number of factors; the most important being the source geometry, source-to-material distance and the geometry of the irradiated material. Figure 3 shows central axis depth–dose distributions in semi-infinite water targets at two distances from the source (2.5 cm and 18 cm) for  $^{60}\text{Co}\,\gamma$  ray plaque sources of two sizes (150 cm × 610 cm and 50 cm × 76 cm) [31]. No buildup effect is present for such a diffuse broad beam incidence of photons.

For the incident electron beams that are used in food processing (energies of 0.25–10 MeV), there is generally a buildup region in low atomic number materials due to the progressive cascading of secondary electrons by collisional energy losses.

<sup>&</sup>lt;sup>4</sup> Electron or photon penetration is dependent on the type of material, but as expressed as the standardized (or density normalized) depth in units of mass per unit area, it is nearly the same for all materials. To reconvert from standardized depth to linear depth for a given material, divide by the density of that material. Standardized depth is variously called thickness, area density, surface density and density thickness.

 $<sup>^5</sup>$  Ninety-nine per cent of the energy emitted by  $^{60}$ Co decay is equally divided between two photons of energies 1.17 and 1.33 MeV; for food irradiation purposes, an average energy value of 1.25 MeV is assumed.

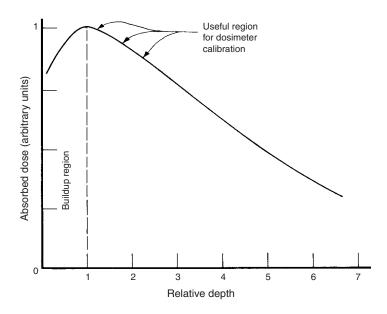


FIG 2. Radiation energy deposition as a function of thickness (depth) in an absorber showing the buildup region, the thickness required to achieve electron equilibrium (the dashed vertical line) and the useful region for dosimeter calibration. The thickness is expressed here in multiples of 'thickness for maximum dose'.

This buildup region extends up to a depth of approximately one third to two thirds of the electron range, except when the angle of incidence is greatly increased (away from the normal) or when a scattering material is placed between the source and the irradiated material. Figure 4 shows that for 2 MeV electrons the depth of maximum buildup of dose in polystyrene decreases as the angle of incidence increases [32]. With a scanned beam (an electron beam facility has a scanning horn of typically 2 m length and a scanning width around the perpendicular incidence point of  $\pm 0.5$  m), the angle of incidence can vary by about ±15° and thus the resulting depth-dose distribution is a superposition of a series of such curves. On the other hand, as shown in Fig. 5 for 10 MeV electrons in water, the buildup region is flattened by the presence of a higher atomic number material in front of the irradiated material (curve 3), and the attenuation region is also flattened somewhat by the positioning of a higher atomic number backscattering medium at a greater depth (curve 2) [33-35]. Typically, the electron beam is generated in vacuum and emerges through a metallic window (e.g. 0.1 mm thick tantalum), thus causing similar scattering effects to those described here.

There are experimental and theoretical data for depth–dose distributions for 10 MeV electrons in several absorbing materials. Table III lists the material densities,

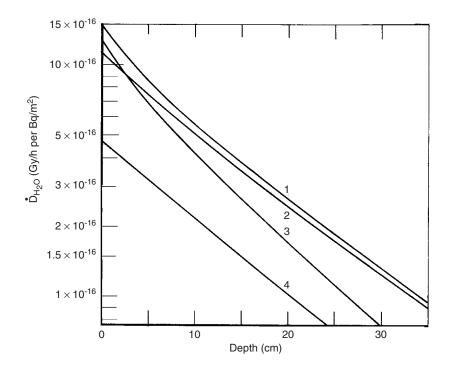


FIG. 3. Calculated central axis depth—dose curves in a semi-infinite water slab, irradiated by a rectangular  $^{60}$ Co  $\gamma$  ray plaque source in two sizes and at two different distances from the slab. The ordinate is absorbed dose rate per area load of activity. Plaque source of 150 cm  $\times$  610 cm area at a distance of 2.5 cm (curve 1) and 18 cm (curve 2); and a plaque source of 50 cm  $\times$  76 cm area at a distance of 2.5 cm (curve 3) and 18 cm (curve 4) [31].

TABLE III. DEPTH–DOSE PARAMETERS FOR VARIOUS SEMI-INFINITE ABSORBERS FOR 10 MeV ELECTRONS [36, 37]

A lala	Density	Z(max	x. dose) <sup>a</sup>	Z(optimum) <sup>b</sup>	Ratio (ma	x./surface)c
Absorber	(g/cm <sup>3</sup> )	(cm)	(g/cm <sup>2</sup> )	(cm)	(exp.)	(calc.)
Graphite	1.73	1.7	2.94	2.3	1.39	1.38
Aluminium	2.67	1.0	2.67	1.4	1.51	1.53
Polyethylene	0.96	3.1	2.96	3.9	1.27	
Polystyrene	1.05	3.2	3.36	4.0	1.35	1.33
Water	1.00	3.1	3.1	3.9	1.30	1.32

<sup>&</sup>lt;sup>a</sup> The depth at which the dose is maximum, given in two units (cm and g/cm<sup>2</sup>).

<sup>&</sup>lt;sup>b</sup> The depth at which the dose is equal to the entrance dose.

<sup>&</sup>lt;sup>c</sup> The ratio of maximum dose to the dose at the entrance surface, measured experimentally and calculated (calculations by Spencer [38]).

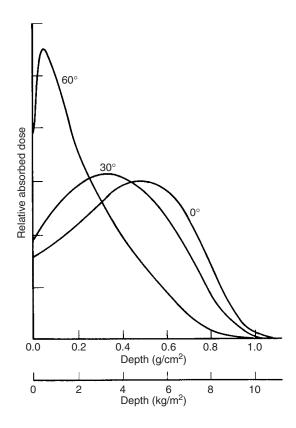


FIG. 4. Experimental central axis depth—dose curves for broad beam monodirectional 2 MeV electrons incident on a semi-infinite polystyrene slab at perpendicular incidence (0°), and at 30 and 60° angular incidence (data obtained with thin radiochromic films to achieve the required resolution) [32].

the depths where the maximum doses occur, the depths at which the doses on the descending portions of the depth—dose curve equal the entrance doses, and both experimental [36, 37] and theoretical [38] values of the peak to entrance dose ratio, for 10 MeV scanned electron beams for semi-infinite carbon (graphite), aluminium, polyethylene, polystyrene and water media.

#### 2.3.3. Electron equilibrium

For photons, the distribution of dose close to the entrance surface for a monoenergetic, monodirectional beam is likely to be non-uniform due to dose buildup. The exact value of dose in this region is sensitive to the surface conditions. It is easier to ascertain the value of dose beyond this region where the secondary

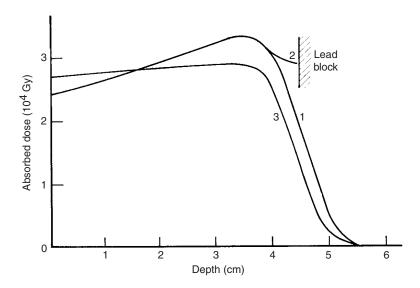


FIG. 5. Experimental central axis depth—dose distributions for a broad beam scanned 10 MeV electron beam incident on a semi-infinite water slab: curve 1, unmodified depth—dose distribution; curve 2, depth—dose modified due to the presence of a thick lead block at ≈4.5 cm depth; curve 3, depth—dose modified due to scattering by a thin copper sheet in front of the water slab [33–35].

electron spectrum is fairly constant; at this depth the conditions for electron equilibrium are satisfied (see the Glossary for the definition of electron equilibrium). The buildup region approximately corresponds to the range of the highest energy secondary electrons produced by photon interactions in the medium [39]. When the conditions for electron equilibrium exist, the absorbed dose can be expressed more accurately by its definition in terms of energy deposited per unit mass of a particular medium, because most secondary electrons (which are responsible for energy deposition) result from interactions in the same medium.

The shape of the depth–dose curve for  $\gamma$  radiation in a homogeneous material is shown in Fig. 2. It is determined by the  $\gamma$  absorption coefficients and the electron stopping power of the material for the energy of the incident  $\gamma$  rays. This distribution is rigorously valid for narrow beam, normal incidence and monoenergetic  $\gamma$  radiation. In the case of material irradiated with an extended  $\gamma$  ray plaque source or a diffused X ray beam, electron equilibrium conditions prevail essentially up to the surface (Fig. 3). For the reasons given above, the portion of the curve just beyond the buildup region is considered the most useful region for dosimeter calibration. In addition, the shape of this part of the curve is more predictable than the portion in the buildup

region, particularly in the case of complex radiation spectra and beam geometries and of mixed radiation fields.

When a thin dosimeter is being irradiated in a given medium (for the purpose of calibration), it is relatively easy to position the dosimeter between layers of the material of interest (e.g., low atomic number plastics) thick enough to establish electron equilibrium conditions. Figure 6 gives the approximate electron equilibrium thickness for water for various incident photon energies important for food irradiation [29, 30]. It is more difficult to position a thick dosimeter with a relatively small surface to volume ratio (such as an ampoule containing a chemical solution) in this way. This is partly because of self-absorption by the sensing material and its container giving a non-uniform dose distribution within the dosimeter. The problem becomes greater when the dosimeter material is considerably different from its surroundings.

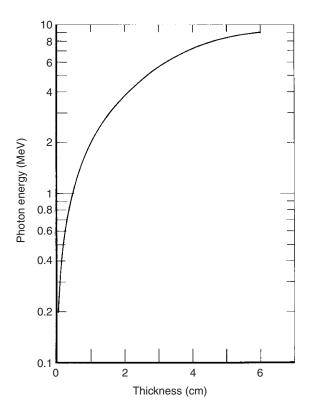


FIG. 6. Electron equilibrium thickness in water for different photon energies [29, 30].

#### 2.4. DOSIMETRY SYSTEMS

The measurement of absorbed dose involves the use of a dosimetry system which consists of not only well established physical or chemical dosimeters but also the instrument which measures the relevant radiation induced effect in the dosimeter (e.g., spectrophotometers, electron paramagnetic resonance (EPR) spectrometers) and their associated reference standards (such as wavelength and absorbance standards), and the procedure for using the system. The measuring instrument must be well characterized, so that it gives reproducible and accurate results. Any radiation induced effect (also called the dosimeter response) which is reproducible and can be quantified can, in principle, be used for dosimetry.

#### 2.4.1. Classes of dosimeter

Dosimetry systems can be classed on the basis of their intrinsic accuracy and applications. There are four classes of dosimeter [40]:

- Primary standard dosimeters
- Reference standard dosimeters
- Transfer standard dosimeters
- Routine/working dosimeters.

Primary standard dosimeters enable an absolute measurement of absorbed dose to be made with reference only to the SI base units (mass, length, time, electric current, etc.) and fundamental physical constants. They do not need to be calibrated. This type of dosimetry system is generally maintained and operated by national standards laboratories and is used to provide the basic standard for use in the country. There are two types of primary standard dosimeters: ionization chambers and calorimeters.

Reference standard dosimeters are dosimeters of high metrological quality that can be used as reference standards to calibrate other dosimeters. In turn, they need to be calibrated against a primary standard, generally through the use of a transfer standard dosimeter. They must have a radiation response that is accurately measurable, and this response must have a well defined functional relationship with the absorbed dose. The effect on the dosimeter response of various parameters, such as irradiation temperature and post-irradiation stability, must be well characterized and capable of expression in terms of simple correction factors. Commonly used reference dosimeters include Fricke, ceric-cerous, dichromate, ethanol-chlorobenzene (ECB) and alanine dosimeters.

Transfer standard dosimeters are used for transferring dose information from an accredited or national standards laboratory to an irradiation facility in order to

TABLE IV. CLASSES OF DOSIMETER

Class	Calibration necessary?	Uncertainty $(k = 1)$	Examples
Primary	No	1%	Calorimeter, ionization chamber
Reference	Yes	2–3%	Calorimeter, alanine, dichromate, ceric-cerous, ECB, Fricke,
Transfer	Yes	3–5%	Alanine, Fricke, dichromate, ceric-cerous, ECB
Routine	Yes	≈5%	PMMA, radiochromic films, CTA, ceric-cerous, ECB

establish traceability to that standards laboratory. They should be used under conditions specified by the issuing laboratory. They are normally reference standard dosimeters that have characteristics meeting the requirements of the particular application. For example, they have to be transported from one place to another; there is also generally a time delay between their preparation and irradiation, as well as between irradiation and analysis. Similar to reference standard dosimeters, they need to be calibrated.

Routine (or working) dosimeters are used in radiation processing facilities for dose mapping and for process monitoring for quality control. They must be frequently calibrated against reference or transfer dosimeters, as they may not be sufficiently stable and independent from environmental or radiation field conditions. In addition, they may show significant variations from batch to batch. Commonly used routine dosimeters include PMMA, radiochromic and cellulose triacetate (CTA) films, cericcerous and ECB dosimeters.

Table IV lists these four classes of dosimeter. Various dosimetry systems are discussed in more detail in Section 6.

#### 2.4.2. Characterization of dosimetry systems

The reliability of a dosimetry system increases with increasing understanding of its behaviour. The user's confidence in the interpretation of its behaviour and its response also increases with more experience. Thus, a thorough characterization is quite essential before using any dosimetry system for dose measurement. Characterization consists of:

- (1) Calibrating the dosimetry system,
- (2) Establishing traceability,

- (3) Determining batch homogeneity,
- (4) Determining uncertainty in the measured dose value,
- (5) Understanding and quantifying the effects of the influence quantities on the performance of the dosimetry system.

#### 2.4.2.1. Calibration

Calibration is the relationship between the absorbed dose and the radiation induced effects in dosimeters determined using the measurement instrument. The calibration procedure of a dosimetry system mainly consists of [40]:

- (a) Irradiation of dosimeters to a number of known absorbed doses over the useful dose range,
- (b) Analysis of the irradiated dosimeters using the calibrated measurement instrument,
- (c) Generation of a calibration relationship (curve).

Such a calibration must be traceable to a national laboratory, which means that the measurements are certified by a national laboratory (this is discussed further in Section 2.4.2.2).

Calibration must be carried out on each new batch of dosimeters. The calibration curve supplied by the manufacturer/supplier of the dosimeters should be considered as general information and should not be used without further verification of its applicability. Different lots of dosimeters purchased at different times from a batch identified by the manufacturer as the same batch should be cross-checked to ensure equivalent response. This could be achieved through a calibration verification exercise, where dosimeters from both lots are irradiated together in such a way that they receive the same dose. This should be repeated at several doses spread over the calibration dose range. A statistical test, such as a *t* test, should then be used to determine if there is any significant difference between the two lots. The calibration of the existing batch should be checked approximately annually to confirm its continued validity. This check could take the form of a calibration verification exercise.

Calibration needs to be performed for the entire dosimetry system, not just for the dosimeters. The measurement instrument is an integral part of the dosimetry system, thus the calibration of a dosimetry system should be regarded as being specific to a particular instrument. Calibration that is established with one instrument is not valid for another one. The effect of any changes, or repairs, to the measurement instrument should be assessed. A major repair may require either a calibration check (e.g., a calibration verification exercise) or a complete recalibration of the dosimetry system. In addition, the calibration needs to be checked if the procedure is altered.

For many dosimeters, the response is influenced by the environmental conditions, such as the temperature during and after irradiation, the humidity, the dose rate and the analysis time relative to the time of irradiation. Since the calibration relationship is valid strictly only for the conditions present during the calibration procedure, it is necessary to have the calibration conditions as similar as possible to those present during normal dose measurements in order to limit errors due to these effects. Two methods are possible for calibration irradiation of the dosimeters:

- (1) Irradiate them in the production facility ('in-house' calibration),
- (2) Irradiate them in a calibration laboratory followed by a calibration verification in the production facility.

More details of these methods and other general descriptions of calibration procedures may be found in ASTM E1261 [40] and Ref. [41].

It is necessary to convert the measured calibration data into some form of smooth function that will enable dose to be determined from a measured dosimeter response. This could be as simple as a hand drawn graph of response (y axis) versus dose (x axis), but in practice a mathematical fitting procedure (regression analysis) of some form is generally used to obtain the relationship between the dosimeter response and the dose. Strictly, dose should be used as the independent variable (x variable). However, this results in an expression which is difficult to solve for dose, which is the quantity required. In practice, it is more convenient to have dose as the dependent variable (y variable). This will not result in an appreciable error provided the dose range is not greater than one decade. If the response is directly proportional to the dose, the calibration relationship is linear. If there is no direct proportionality, the relationship is non-linear. This is the case when a dosimeter undergoes a saturation effect as, for example, in dye systems in plastic materials. Figure 7 gives examples of typical linear and non-linear response functions. In general, there is no recommended type of mathematical expression to represent the non-linear relationship between response and dose. In many cases, a polynomial function (e.g., dose =  $a + b \times \text{response} + c \times \text{response}^2 + ...$ ) will adequately describe the relationship. In selecting a function, the main consideration is to use the lowest order of polynomial that will adequately represent the data.

#### 2.4.2.2. Traceability

A system of calibration should exist within each country to ensure that all measurements can be related to the national standard through an unbroken chain. Such a chain is known as a *traceability chain*. Traceability may be defined as the ability to demonstrate by means of an unbroken chain of comparisons that a measurement is in agreement within acceptable limits of uncertainty with comparable

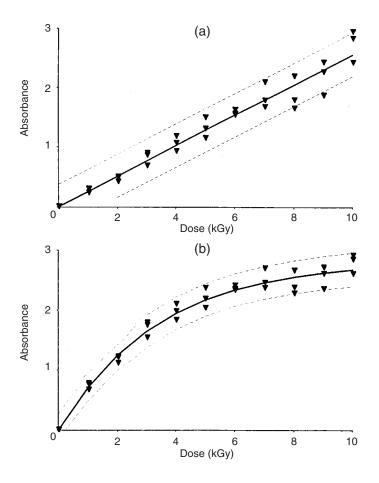


FIG. 7. Results of typical regression analyses for (a) linear and (b) non-linear response curves; at each dose point three independent replicates were used. The dashed curves indicate the 95% confidence limits for individual values.

nationally or internationally recognized standards. This is a very important requirement; the measurements do not have much validity without such traceability.

The International Bureau of Weights and Measures (BIPM), Sèvres, France, has an important role in acting as a focal point for the intercomparison of standards held by individual countries. The relationships between different laboratories and the end user (such as an industrial facility) are shown in Fig. 8 [11]. End users derive their calibrations either directly from the national standards laboratory or from a secondary calibration laboratory. Figure 9 shows how different classes of dosimeter are used in the traceability chain [11].

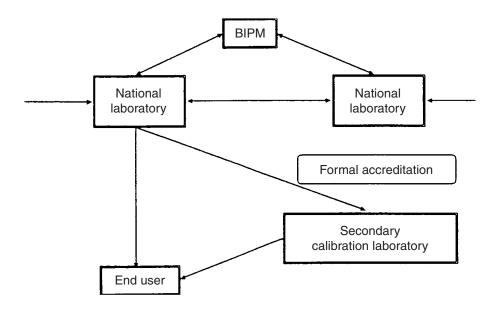


FIG. 8. Relationships between different bodies within the International Measurement System [11].

It is essential that *all* measurements be traceable to a national standards laboratory, i.e. every aspect of the dosimetry system should be traceable. Thus, every piece of equipment/instrument that forms part of a dosimetry system should be calibrated and compared against a standard supplied by a national standards laboratory. This exercise should be done regularly.

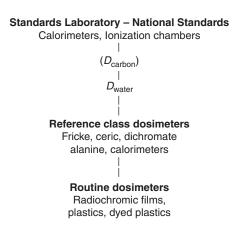


FIG. 9. A typical national traceability chain for high dose dosimetry [11].

## 2.4.2.3. Batch homogeneity

It is essential to determine the extent of variability of the response from dosimeters of a given batch. To determine this, 10–30 dosimeters are irradiated to the same dose under the same irradiation conditions. These dosimeters are then analysed under similar conditions by the same technician over a short time period. This is generally referred to as conditions of repeatability. The standard deviation of the distribution of the response values is the measure of uncertainty due to non-homogeneity of the batch of dosimeters. The coefficient of variation,  $CV(\%) = 100 \times (\text{standard deviation/mean})$ , should be less than 2% for routine dosimeters.

## 2.4.2.4. Uncertainty in dose measurement

The objective of a measurement is to determine the value of the measurand, i.e. the value of the particular quantity to be measured. In general, the result of a measurement is only an approximation or estimate of the value of the measurand (e.g. dose) and thus is complete only when accompanied by a statement of the uncertainty of that estimate. Uncertainty (of measurement) may be defined as a parameter that characterizes the distribution of the values that could reasonably be attributed to the measurand. Thus, the uncertainty of the result of a measurement reflects the lack of exact knowledge of the value of the measurand (dose) or, in other words, it reflects the degree of accuracy in the measured value.

This is discussed further in Section 2.6.

### 2.4.2.5. *Influence quantities*

The response of nearly every type of dosimeter is influenced by various external parameters to a varying degree. This effect should be carefully studied and the impact minimized or corrected for. For example, if the dosimetry system is used for a dose measurement at a temperature different than the one for which it was calibrated, it is necessary to correct the dosimeter response before using it to determine the value of the dose from the calibration relationship. Some of the most common quantities of influence are: temperature, humidity (water content of the dosimeter), oxygen content of the dosimeter, dose rate and light. The radiation type ( $\gamma$  rays or electrons), the energy of radiation and geometrical factors can also affect the response of a dosimeter to a lesser degree. The response of a dosimeter after irradiation quite often varies with time.

It is important that these effects be understood and their influences be taken into account to reduce any uncertainty in the response (and therefore dose) measurement. For more details of how this can be achieved, see Refs [5, 42].

#### 2.5. SELECTION CRITERIA FOR ROUTINE DOSIMETERS

In selecting a routine dosimetry system for use in radiation processing of food, the following criteria must be fulfilled to ensure suitable dosimetry. The dosimetry system should have:

- (a) A suitable dose range;
- (b) Ease of calibration over the dose range of interest;
- (c) Good reproducibility of dose response and its measurement;
- (d) Limited variation in response when used with different radiation spectra (i.e. limited energy dependence of response);
- (e) Insignificant variation in response within the expected dose rate range;
- (f) Product equivalence characteristics (approximately equivalent to water)<sup>6</sup>;
- (g) Total uncertainty suitable for the application;
- (h) Limited variation in response to environmental conditions (the effects of light, temperature, ambient atmosphere, humidity, impurities and storage) or amenability to easy correction;
- (i) A well developed and proven standard measurement procedure (e.g., refer to the ASTM standards in the Bibliography);
- (j) An extended stable readout period (e.g., from one hour to a few days);
- (k) A physical size suitable for the required spatial resolution of the dose measurement;
- (1) Low cost and simple handling and readout procedures;
- (m) Long pre-irradiation shelf life and ruggedness.

It is essential to thoroughly understand the dosimetry requirements and the conditions of use so as to be able to make a suitable selection of the routine dosimetry system. It is almost impossible that any single dosimetry system will have all of these desirable qualities; thus, a compromise is often necessary.

#### 2.6. UNCERTAINTY IN DOSIMETRY

As stated earlier, the result of a measurement is only an approximation or estimate of the value of the measurand (e.g., the absorbed dose) and thus is complete only when accompanied by a statement of the uncertainty of that estimate. An

 $<sup>^6</sup>$  It should be noted that most foods, independent of their actual water content, are almost water equivalent with regard to radiation interaction and have an effective atomic number of about 7.5. They have a density in general between 0.8 and 1.1 g/cm<sup>3</sup>.

interesting way of viewing the concept of uncertainty that also provides an insight into the situation is as follows:

- (a) The person with one dosimeter thinks (s)he knows the dose value accurately.
- (b) The person with two dosimeters has only a rough estimate and begins to wonder.
- (c) The person with three dosimeters realizes (s)he does not know, but can make a better judgement than the other two, since (s)he can estimate the uncertainty.

This is what ISO states regarding uncertainty and its determination [43]: "When reporting the result of a measurement of a physical quantity, it is obligatory that some quantitative indication of the quality of the result be given so that those who use it can assess its reliability. Without such an indication, measurement results cannot be compared, either among themselves or with reference values given in a specification or standard. It is therefore necessary that there be a readily implemented, easily understood, and generally accepted procedure for characterizing the quality of a result of a measurement, that is, for evaluating and expressing its *uncertainty*."

Uncertainty in any measurement is a fact of life and unavoidable. First, the sources of uncertainty should be identified, and their effects minimized as much as possible. The remaining sources of uncertainty should then be evaluated. This is most easily done by considering in turn each step in the calibration and use of a dosimetry system, and assessing what uncertainties are likely to be associated with each step. The uncertainty associated with a dose measurement can then be calculated by combining the individual components together. The philosophy used is to ascribe to each component of uncertainty an effective standard deviation, known as a standard uncertainty, and these standard uncertainties are then combined to produce the total uncertainty. The methodology for estimating uncertainties and their components is well developed and available guidelines should be used [41, 43, 44]. This is of importance for dosimetry in food irradiation for both scientific research and commercial radiation processing (see ASTM Standard E1900 [45] and the Bibliography). Examples of estimating uncertainties of dose measurements and deriving the total uncertainty associated with the reported values can be found elsewhere [46–48].

There are two categories of uncertainty based on their *method of evaluation*: Type A and Type B [43]. Type A uncertainties are those that are evaluated by statistical analysis of series of observations. While Type B uncertainties are those that are evaluated by means other than the statistical analysis of observations; this is based on scientific judgement and information that may include previous measurement data, general knowledge about the instruments, and manufacturer's specification and calibration certificates. At the same time, components of uncertainty may also be categorized as random or systematic depending on the type of the associated effects.

However, it should be realized that this type of categorization can be ambiguous when generally applied, since the category can change depending on the application. For example, a random component of uncertainty in one measurement may become a systematic component in another measurement in which the result of the first measurement is used as an input data.

The combined or total standard uncertainty associated with a particular measurement is generally obtained by summing the individual component standard uncertainties following the law of propagation of uncertainty. If u is defined as the *relative* standard uncertainty, then generally the relative combined standard uncertainty,  $u_c$ , may be determined by taking the square root of the sum of the squares of the individual components (for a more rigorous treatment, refer to Ref. [43]):

$$u_{\rm c} = (u_1^2 + u_2^2 + u_3^2 + ...)^{1/2}$$

In reporting the uncertainty associated with a particular measurement, the value given should imply a high level of confidence that the correct result will lie within the reported range. Historically, uncertainties have been reported as expanded uncertainty, U, based on either a 95 or a 99% probability that the correct value is within the range. The accurate calculation of such values is, however, complex, and current practice is to report combined standard uncertainty multiplied by a coverage factor, k, of either 2 or 3. For most situations

 $U = 2u_c$  implies a 95% level of confidence and  $U = 3u_c$  implies a 99% level of confidence.

Interpretation of dosimetry data is an essential part of the validation and control of irradiation processes. Knowledge of the sources and magnitudes of the various components of dosimetry uncertainty can be used both to assess the significance of individual measurements and to establish a statistical control regime for the process. The specific components of dosimetry uncertainty that need to be considered will depend on the use to which a particular dose measurement is being put. The combined uncertainty  $u_{\rm c}$  is the overall uncertainty of a single dose measurement. However, in the case of relative dose mapping for example, the absolute value of a dose measurement is not required, and only those components of uncertainty that affect the random scatter of the dose readings need to be considered. Possible areas of application of dosimetry uncertainty data include

- (a) Interpretation of dose mapping data establishing the significance of small variations in measured dose, and identification of low and high dose regions;
- (b) Interpretation of routine dosimetry data establishing the origin of observed dose variability;

(c) Establishment of routine operating parameters to ensure dose delivery within defined confidence limits.

#### 2.7. USE OF DOSIMETERS

A convenient method of determining the absorbed dose experimentally in a given medium is to use a routine dosimeter by inserting it into that medium [5, 26]. It should be small enough to ensure that the photon or electron spectrum is not disturbed appreciably by the presence of the dosimeter material. Accuracy of the dose measurement is improved by using a dosimeter with an effective atomic number similar to that of the host material. This ensures that both their photon absorption coefficients and their electron stopping powers are very similar over a wide radiation energy range. For example, if one wants to measure the absorbed dose in a typically hydrogenous food substance (such as meat, vegetable or fruit) or in a plastic material such as polyethylene  $(CH_2)_n$ , a dosimeter made of PMMA or of thin dyed plastic film of low atomic number will be useful as a dosimeter when tightly incorporated in the host material.

In principle, the dosimeters used for electron irradiation should be calibrated in an electron beam. However, the response for a large majority of dosimeters is similar for the two radiation types, at least over a limited dose range. However, before using dosimeters that are calibrated in a  $\gamma$  field for electron dose measurements, it is necessary to confirm that its behaviour is similar in the dose range of interest. Since dose gradients are much steeper for electron beams than for  $\gamma$  fields, the dosimeters should be thin for precise measurements. A somewhat thicker dosimeter may be used at the position of the peak of the depth–dose distribution, since here the dose changes only slightly with depth in the product. The response of the dosimeter must be relatively independent of the spectral energy because the energy spectrum also changes with depth in the material.

For food irradiation applications, water is considered the reference material. Thus, 'dose' generally means 'dose in water'. A large majority of dosimeters used for food irradiation are water equivalent, such as water calorimeters, Fricke dosimeters, PMMA dosimeters or other organic systems. If the absorbed dose is measured in a material whose radiation absorption characteristics (photon mass energy absorption cross-sections or electron mass stopping power) differ appreciably from those of water, appropriate corrections have to be made in order to convert the dose in one material to that in the other. This is rarely the case with food irradiation.

If more precise dose measurement is required (i.e. if the uncertainty in the value is larger than that required for the application), using several dosimeters at one point would help. The uncertainty in the mean of the measured dose values is then reduced; generally the random component of the uncertainty is reduced by  $\sqrt{n}$ , where n is the number of dosimeters used.

# 3. IRRADIATOR DESIGN CONCEPTS

#### 3.1. INTRODUCTION

The design and construction of a food irradiator are important considerations in the treatment of the product, since they affect the dose distribution in the product and the dose range attainable. Irradiators are generally designed either specifically for food irradiation or for multipurpose applications which may include some foods. Many of the irradiator concepts discussed in this book also apply to other processing applications, for example radiation sterilization, waste treatment and processing of various materials. Detailed descriptions of such irradiators may be found elsewhere [49]. Some examples of typical irradiators suitable for food processing are given in Annex I.

There are design principles for irradiation facilities which are linked to the physical nature of the radiation emission (Fig. 10) [50]. Radionuclide sources emit radiation equally in all directions; thus, in order to absorb the emitted radiation energy most effectively the process loads must be distributed around the source leaving as small gaps as possible. Machine sources — on the contrary — emit a unidirectional beam, and even when converted into bremsstrahlung the forward direction is

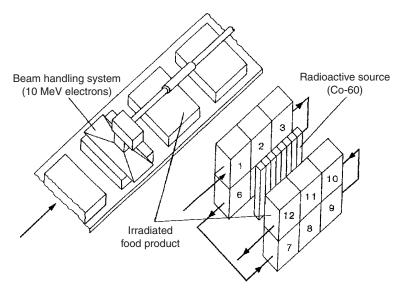


FIG. 10. Schematic diagram of an irradiation facility. For an electron facility, process loads are brought into the radiation field one at a time. For a radionuclide facility, several process loads surround the radiation source and are thus irradiated together.

predominant. Consequently, the process loads to be irradiated have to be brought before the beam exit window of the accelerator. Besides, an electron beam must be widened by technical means (scanning or scattering) in order to obtain a homogeneous dose distribution over process loads of extended size. However, both types of irradiation facility have in common the need for movement of the process loads in order to compensate for differences in dose rate at different locations and thus to make the resulting dose distribution more homogeneous. These physical characteristics of radiation emission and absorption, and the consequent design principles for irradiation facilities have consequences for the performance characterization and the appropriate dosimetry procedures in radiation processing (see ASTM Standards E1204 [51] and E1431 [52] for food processing, and ASTM Standards E1608 [53], E1649 [54] and E1702 [55] for processing in general).

With regard to treatment capacities and achievable throughput, the radionuclide and the machine sources may be compared in the following way. Consider an industrial irradiation facility with a  $^{60}$ Co source of activity 3.7 ×  $10^{16}$  Bq (1 MCi). Multiplying the activity by the energy per disintegration (1.17 MeV plus 1.33 MeV) results in an emitted power of 14.8 kW. Under optimal conditions, an overall energy absorption efficiency of 25% can be achieved for both kinds of irradiator, resulting in an effective power of about 3.7 kW. Rearranging the units, this can be converted to a product throughput of 3.7 kGy·kg/s. Thus, a typical treatment at 10 kGy would allow for a product throughput of 0.37 kg/s or about 1.3 t/h. X ray (bremsstrahlung) conversion from an electron beam under optimal conditions may reach a conversion efficiency of about 10%; consequently, an X ray facility needs a 150 kW electron accelerator in order to achieve a throughput similar to a  $^{60}$ Co facility of 1 MCi.

The major considerations in the design of an irradiator are the uniformity of the absorbed dose in the irradiated product, efficient utilization of the radiation energy and cost effectiveness based on minimizing the combined capital and operating costs. In the case of food irradiation, generalization is difficult, since only a few facilities are designed specifically to handle only one food item, whereas the majority of irradiation facilities are designed for multipurpose applications and to handle many different items. The physical nature and the quantity of the product to be treated determine the general design principles; and the desired effect determines the magnitudes of the minimum and maximum doses to the product.

### 3.2. CRITERIA FOR IRRADIATOR DESIGN

Food irradiators must be designed and built to provide an absorbed dose in the product within the minimum and maximum limits in accordance with process specifications and government regulatory requirements. The actual minimum dose,

 $D_{\min}$ , and maximum dose,  $D_{\max}$ , as measured in the product, must be within these limits. The ratio  $D_{\max}/D_{\min}$ , termed the dose uniformity ratio, U, is a useful concept for irradiator designers and food technologists. For research applications, this ratio should be as close to 1 (unity) as possible, i.e. the dose should be very uniform in the small research sample. This is necessary in order that the experimental results can clearly demonstrate the dose–effect relationship, while for industrial applications where large process loads are irradiated, wider dose variation is unavoidable. For economic and practical reasons, for example to optimize product quality and flexibility and to maximize throughput of an irradiation facility, various techniques are used to minimize this ratio. Several concepts of irradiator design for accomplishing this in practical situations are illustrated in this section, for both radionuclide and machine sources. The methods used to achieve this objective include multipass or multi-sided irradiation, source overlap, source activity augmentation, product overlap with passes on two or more levels, use of strategically placed absorbing or scattering materials, or beam scan adjustments.

The basic components of an irradiation facility include a radiation source (radionuclide  $\gamma$  source or machine source) and the associated systems, a product transportation system (in most cases), biological shield for protection of the personnel against radiation, control system, dosimetry laboratory and product handling system (receiving, and pre- and post-irradiation storage areas). The following factors largely govern the selection of irradiator design:

*Radiation source*: Before selecting the type of radiation source it is essential to have some knowledge of the expected product throughput, the size of the process load that would be irradiated, any constraints on the time duration of irradiation and dose rate (if it affects the process).

Mode of transportation of food products: It is essential to consider whether the product is to be handled in bulk or in packaged form during collection, transportation and distribution. This determines the mechanical design of the irradiator and infacility transport systems, as well as the source-to-product geometry.

Range of required doses: In its various applications, food irradiation requires a wide range of doses, potentially from 10 Gy to 100 kGy (Section 1.4). Limitations on the maximum mechanical speed of the conveyor system may necessitate the use of only part of the radionuclide source or the use of lower electron beam currents for low dose applications. Limitations on the minimum mechanical speed of the conveyor system may require fractionated irradiation where the full dose is applied in several instalments.

<sup>&</sup>lt;sup>7</sup> Process load is defined as the volume of material with a specified loading configuration irradiated as a single entity.

Throughput: This refers to the amount of product treated with a given dose within a defined period of time, and is generally measured in units of kGy·kg/s. It directly relates to the beam power (measured in kilowatts) or the source activity (measured in becquerels). Maximum throughput is limited by the mechanical speed of the conveyor system and the radioactivity installed or the accelerator beam power.

*Economics*: This generally requires capital costs to be balanced against operational costs; for example, a complex transport system with high source utilization efficiency versus a simpler transport system with reduced source utilization efficiency; highly automated versus labour intensive. Manufacturers of irradiation facilities offer a variety of designs adapted to such requirements.

*Reliability*: Food is a perishable item, and reliability in system operation is important for the successful completion of a process/treatment.

*Process duration*: For refrigerated or frozen products, the time for which the food has to be inside the irradiation cell and, hence, outside a refrigerated area may be an important consideration.

*Safety*: Systems to protect the operating personnel from radiation hazards must be in place. These include appropriate shielding, radiation chamber entry control and radiation monitoring systems. The design of the irradiator should also minimize the risk from other industrial hazards (i.e. occupational safety).

Compliance with food regulations: The design should be such that it promotes compliance with good manufacturing practices for foods, including 'good irradiation practices', and with government food processing regulations.

Compromises must often be made in selecting the irradiator design. As an example, certain agricultural products which are susceptible to injury or damage during transport require a conveyor system that is relatively gentle. The movement of the product from loading before irradiation to unloading after irradiation and subsequently during storage and distribution should give minimal squeezing, abrasion and collision among the products. Except perhaps in the case of grain irradiated in bulk, it is preferable to use during irradiation the same containers that have been traditionally adopted for shipping or marketing the specific food. Exceptions might, however, be necessary where dose uniformity or radiation efficiency are significantly adversely affected by radiation attenuation or scattering in the packaging.

There may be an economic advantage in designing a single facility for the irradiation of different agricultural products to match different harvest periods, or for products other than food (e.g., sterilization of health care products and treatment of industrial material). It may also be advantageous to incorporate various modes of transport [56], and the practical situation may call for versatility in the conveyor design to accept products and containers of varying configurations. Furthermore, the useful dose range in food applications is from about 10 Gy to 100 kGy (a range

of a factor of 10 000); if this dose range is to be achieved solely by changing the transport speed past the source, it requires a vast variability in the conveyor speed settings. On the other hand, in some in-house facilities, only a single product is processed and the irradiator is then dedicated only for this process, thus simplifying its design [57–59].

### 3.3. SELECTION OF RADIATION SOURCE

Three principal types of radiation source can be used in food irradiation according to the Codex Alimentarius General Standard [2]:

- (a) Gamma radiation from radionuclides such as <sup>60</sup>Co or <sup>137</sup>Cs <sup>8</sup>;
- (b) Machine sources of electron beams with energies up to 10 MeV;
- (c) Machine sources of bremsstrahlung (X rays) with electron energies up to 5 MeV.

Because of their greater penetrating capability,  $\gamma$  rays and X rays may be used for processing of relatively thick or dense products (Fig. 11). For situations where only a shallow penetration is needed and where rapid conveyor speeds can be used, high power electron beams may provide a higher throughput at lower cost per unit of product when large amounts of product are involved.

## 3.3.1. Gamma rays

The  $\gamma$  rays used in food processing are obtained from large  $^{60}$ Co radionuclide sources. This type of radiation is essentially monoenergetic ( $^{60}$ Co emits simultaneously two photons per disintegration with energies of 1.17 and 1.33 MeV). Using analytical techniques such as the point kernel or Monte Carlo methods, it is possible to compute the dose distribution in irradiated food products even when very complicated source geometries such as extended plaque sources are used [60–62]. The resulting depth–dose distribution in the food products usually resembles an approximately exponential curve. Irradiation from two sides (two sided irradiation), obtained either by turning the process load or by irradiation from two sides of a plaque source, is often used to increase the dose uniformity in the process load (Fig. 12).

<sup>&</sup>lt;sup>8</sup> At present, no significant quantities of <sup>137</sup>Cs sufficient for industrial radiation processing are available; consequently, no commercial facility using this radioisotope is in operation.

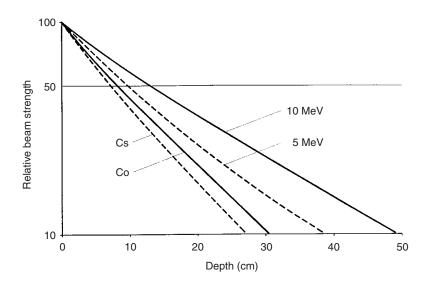


FIG. 11. Beam attenuation curves for radionuclide and X ray sources (in water): the two upper curves are for X rays from 10 and 5 MeV electrons, while the two lower curves are for  $\gamma$  rays from  $^{60}$ Co and  $^{137}$ Cs. The slight curvature in all four curves is due to the change of the photon spectrum with depth.

## 3.3.2. Electrons

Electrons emitted by accelerators have fairly narrow spectral energy limits (usually less than ±10% of the nominal energy). The energy of the electrons reaching the product is further controlled by the bending magnets of the beam handling system, if applicable. The range of an electron in a medium is finite (unlike that for photons) and is closely related to its energy (Fig. 13). An irradiation from one side of a 4 cm thick water equivalent product (most food) can be performed using 10 MeV electrons with a dose uniformity ratio approaching 1.3. For two sided irradiations, products as thick as 9 cm of water equivalent material can be treated with 10 MeV electrons (see Section 4.3.3.2 for more details). If the process load to be treated is much thicker than the range of the irradiating electrons, only a surface treatment is possible. For example, 3 MeV electrons can only be used for surface and shallow treatments [63], since they penetrate to a depth of only about 1 cm in water.

An upper energy limit of 10 MeV for electron beam applications for food has been set to avoid, with a very high level of confidence, any induction of radioactivity in irradiated food through photonuclear reactions [2, 6, 64–66].

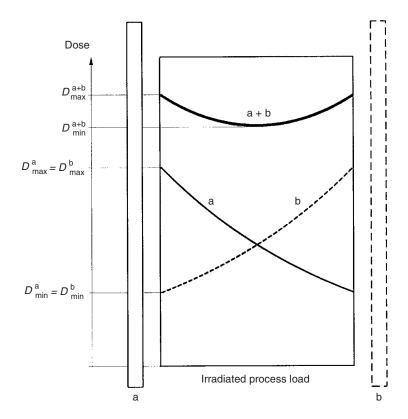


FIG. 12. Depth—dose curves in a process load irradiated from two sides with a radionuclide plaque source. During the first pass the source is on the side 'a' relative to the process load, while during the second pass the source is on the side 'b' relative to the process load. The curves a and b represent the dose contributions for the first and second passes separately, and the curve a + b represents the accumulated dose from both the passes.

## 3.3.3. Bremsstrahlung (X rays)

Bremsstrahlung irradiator design principles are essentially the same as those for electron irradiators [67, 68]. An extended source of X rays is achieved by distributing the primary electron beam over a target (X ray converter) of sufficient size. In contrast to the radionuclide sources, which emit nearly monoenergetic photons, bremsstrahlung (X ray) sources emit photons with a broad energy spectrum (Fig. 14). Since the attenuation of photons is dependent on their energy, the analytical technique used to calculate the dose distribution must take into account the entire energy spectrum. Using analytical techniques such as the point kernel or Monte Carlo

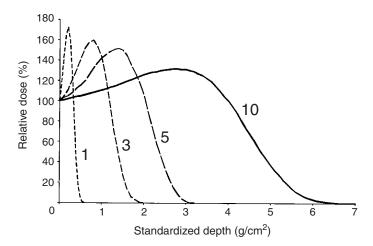


FIG. 13. Typical depth—dose curves for monoenergetic electrons of various energies in the range applicable to food processing. The electron beam energies from left to right are 1, 3, 5, and 10 MeV.

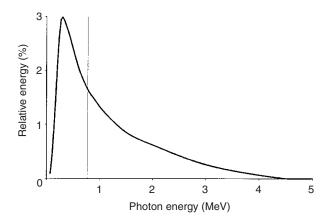


FIG. 14. Typical shape of the photon spectrum of bremsstrahlung radiation (X rays) from 5 MeV electrons: the effective energy is 0.76 MeV (vertical line).

methods, it is possible to compute the dose distribution in food irradiated with bremsstrahlung [60]. The resulting depth-dose curve is pseudo-exponential, a superposition of a series of exponential attenuation curves corresponding to the energies contained in the bremsstrahlung spectrum (Fig. 11).

An upper energy limit of 5 MeV has been set for bremsstrahlung (X ray) applications to food to avoid, with a very high level of confidence, any induction of radioactivity in the irradiated food through photonuclear reactions [2, 5]. In future an upper energy limit of 7.5 MeV might become acceptable, as the hazard of induced radioactivity is insignificant, at least up to this energy [69].

### 3.3.4. Dose distribution in the product

Although irradiators are often designed to yield a low dose uniformity ratio, for example  $U \le 1.5$ , many food product applications can tolerate a higher uniformity ratio of 2 or even 3. The Codex Alimentarius General Standard [2] even provides for U > 3 for some low dose applications. It is not always a requirement of the food irradiation technology that the lowest attainable uniformity ratio be used; however, national standards usually set closer limits and, hence low uniformity ratios.

The dose uniformity in a process load depends on the transverse and lateral dose distributions in the product. The depth—dose distribution refers to the variation of dose along the centre line of the process load, i.e. perpendicular to the plane of the source in the case of radionuclide plaque sources or along the direction of the beam axis for accelerator irradiation. The lateral dose distribution is the variation of dose within the process load in a plane that is parallel to the plane of the radionuclide plaque source or in a plane perpendicular to the direction of the beam for accelerator irradiation.

The depth-dose uniformity is determined by the product density, product thickness and the radiation energy and type. It can be improved by various actions, including: irradiating the process load from two or more sides, using a multipass irradiator system and reducing the thickness of the process load.

The lateral dose distribution depends mainly on the source-to-product geometry and can be improved in several ways:

- (a) By allowing the process load to move past the source at a uniform speed or in a shuffle-dwell motion. This technique is used in both radionuclide and accelerator irradiation and contributes to improving the uniformity of dose along any line in the process load that is parallel to the direction of its motion.
- (b) By introducing additional source elements in the vicinity of the low dose regions (referred to as source activity augmentation). In electron accelerators a similar effect is achieved by using scatter plates in the vicinity of low dose regions or by electronically modifying the scan speed (allowing the beam to

TABLE V. PRESENT STATUS OF IRRADIATORS WORLDWIDE [70]

Energy range (MeV)	Number of facilities	Average power (kW)	Total power (MW)
Electron beam: 0.1–0.3	250	100 <sup>a</sup>	25
Electron beam: 0.3-5	600	25 <sup>b</sup>	15
Electron beam: 5-15	30	$10^{b}$	0.3
Gamma ray beam: 1.25	180	15 <sup>c</sup>	2.7

<sup>&</sup>lt;sup>a</sup> Up to 300 kW.

scan more slowly in the regions of expected low dose). In X ray machines (with extended converters) such an effect can be obtained by varying the different partial beam currents (increasing the flux in the low dose regions).

- (c) By allowing the source to extend beyond the process load in the vicinity of the low dose regions (referred to as source overlap). This technique may be used with source activity augmentation to provide optimum dose uniformity in many radionuclide irradiators.
- (d) By allowing the process load to extend beyond the source and by moving the process load at different levels (e.g., process loads passing a vertical plaque source at two or more levels). This provides an effect equivalent to a large source overlap in the vertical direction.

There are many irradiators in use today for various applications; these are listed in Table V [70]. Many of the radionuclide source irradiators utilize the two sided (or multi-sided) irradiation principle for minimizing the depth—dose variation, and at least one of the techniques discussed above for minimizing the lateral dose variations. While two sided irradiation can be used in an accelerator irradiator, a satisfactory depth—dose distribution can be achieved with one sided irradiation by restricting the thickness of the process load. Irradiation of relatively thin process loads may be accomplished by using low energy electrons. The basic principles and some examples of the irradiator types are described below.

### 3.4. SPECIFIC IRRADIATOR DESIGNS

Irradiator systems can be placed in categories based on any of their general features, for example:

<sup>&</sup>lt;sup>b</sup> Up to 150 kW per installation.

<sup>&</sup>lt;sup>c</sup> Equivalent to 1 MCi.

- (a) Source type, such as radionuclide or accelerator;
- (b) Purpose of treatment, such as sprout inhibition, insect disinfestation, shelf life extension, elimination of pathogenic micro-organisms and parasites, or sterilization;
- (c) Type of product, such as dedicated to one category of foods or multiproduct including health care products;
- (d) Conveyor system design, such as stationary, one pass continuous, multipass continuous or multipass shuffle-dwell.

Categorization by the conveyor system design is taken as the basis for illustration of the irradiators in this section. Several general types of irradiator design are described here, including the methods used for reducing the dose uniformity ratio. In addition, examples of irradiators presently in operation for food processing, such as pallet irradiators and grain irradiators, are briefly described in Annex I.

#### 3.4.1. Radionuclide source irradiators

The irradiator designs described below and illustrated in Figs 15–19 have vertical plaque sources. The same concepts apply in principle if the source plaque is

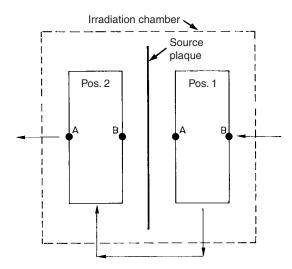


FIG. 15. Sequence of irradiation in a single plaque, two position, stationary radionuclide source irradiator. A and B are fixed points on the side surfaces of the process loads, which are first irradiated in position 1 and then in position 2.

installed horizontally and the product is moving on a horizontal conveyor system, i.e. with a  $90^{\circ}$  rotation of the geometry.

### 3.4.1.1. Stationary irradiators

The simplest type of large volume irradiator is the stationary irradiator. These irradiators are called 'stationary' because the process load is not moving during the irradiation process. A single plaque, two position, stationary irradiator is illustrated in Fig. 15. In this system there are two process loads in the irradiation chamber at any time, one on each side of the source plaque. Two sided irradiation is accomplished by moving a process load into position 1, with surface A facing the source, and allowing it to remain there, with the source raised (irradiation position), for a predetermined period of time. After the source is lowered to the safe position, the process load is then transferred to position 2, with surface B now facing the source, where it remains, with the source raised, for the same predetermined period of time. Quasi-continuous operation is achieved by filling position 1 with the next process load after the previous one has been moved to position 2. In the simplest stationary irradiators, the process load movement is performed manually, using, for example, fork lift trucks and trolleys to assist in the movement of heavy process loads, and the timing is controlled by an operator, who initiates the lowering of the source after the predetermined irradiation time.

Figure 16 shows variations in the above stationary radionuclide source irradiator in order to allow for better energy absorption efficiency. In this single plaque multiposition stationary irradiator there may be numerous process loads in the irradiation chamber at the same time.

Lateral dose variations are minimized in stationary irradiators by providing source overlap and/or source activity augmentation.

### 3.4.1.2. One direction multipass irradiators

In one direction multipass irradiators, the process load is moved by some type of conveyor system past the source plaque in a direction that is parallel to the plane of the source. For the purpose of the description given here, it is assumed that a (two dimensional) source plaque of large dimensions is used which is positioned vertically. In the two pass system, two sided irradiation is obtained by making one pass on each side of the source (Fig. 17). A process load is introduced into the irradiation chamber at position 1, at one end of the chamber, and is moved horizontally past the source to the other end of the chamber. The process load is transferred to the other side of the source plaque from position 10 to position 11 and traverses the irradiation chamber in the opposite direction from position 11 to position 20, completing its two sided irradiation. In moving past the source, the process loads are abutted (touching) and

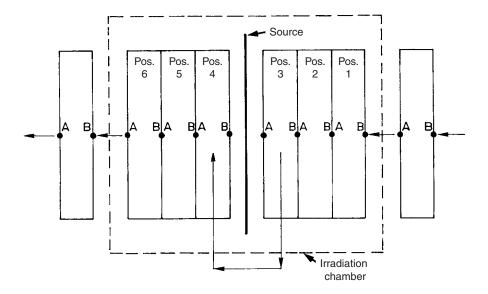


FIG. 16. Sequence of irradiation in a single plaque, multiposition, stationary radionuclide source irradiator. A and B are fixed points on the side surfaces of the process loads, which are sequentially irradiated in positions 1, 2 and 3 on one side of the plaque source, and then in positions 4, 5 and 6 on the other side. (Note: point A on the process load in position 1 is coincident with point B on the process load in position 2, etc.)

are either moved continuously at a uniform speed, or moved from one dwell position to another in a shuffle–dwell fashion (in effect, a series of stops and starts). In the shuffle–dwell mode of operation, each process load remains at each irradiation position for the same predetermined length of time. In this type of irradiator, moving the process load horizontally eliminates or minimizes dose non-uniformities in this horizontal direction. Dose variations in the vertical direction are minimized, as in a stationary irradiation, by source activity augmentation and/or overlap.

Figure 18 illustrates a single plaque, one direction, four pass irradiator. The additional two passes increase the utilization (absorption efficiency) of the radiation energy as well as reduce the depth–dose non-uniformities, and may also reduce the lateral dose variations.

### 3.4.1.3. Two direction multipass irradiators (product overlap irradiators)

In principle, two direction, multipass irradiators (commonly known as product overlap irradiators) are similar to the one direction systems discussed above, except that the process load is moved vertically as well as horizontally as it undergoes its two

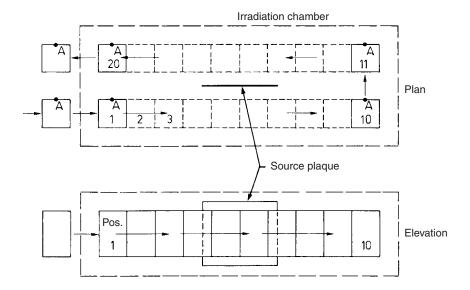


FIG. 17. Sequence of irradiation in a single plaque, two pass, one direction, shuffle–dwell radionuclide source irradiator. A is a fixed point on the side surface of the process load, which passes first on one side of the plaque source from position 1 to position 10 and then on the other side from position 11 to finally position 20.

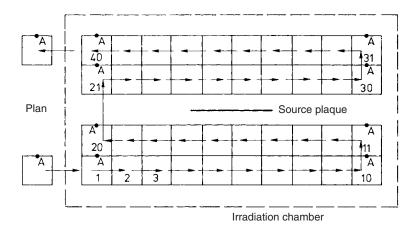


FIG. 18. Sequence of irradiation in a single plaque, multipass, one direction, shuffle–dwell radionuclide source irradiator. A is a fixed point on the side surface of the process load, which passes through the irradiation chamber on both sides of the plaque source from position 1 to position 40, with two passes on each side of the source.

sided irradiation. Figure 19 shows a typical single plaque, two direction, multipass, shuffle–dwell irradiator. Compared with the one direction irradiators with only horizontal movement of the process loads (Fig. 18), the additional second direction of movement is vertical. This type of irradiator is especially useful for irradiation of large quantities of homogeneous products at high doses (e.g., sterilization of health

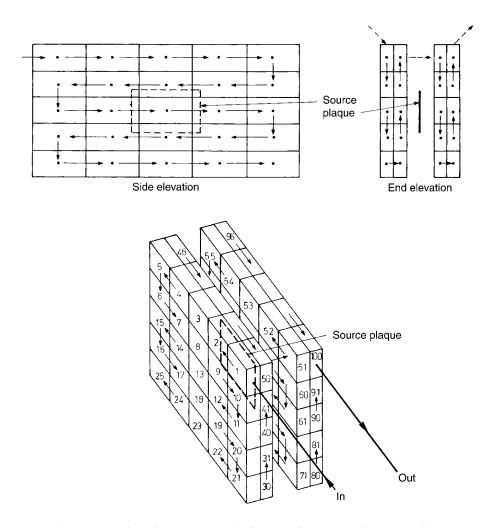


FIG. 19. Sequence of irradiation in a single plaque, multipass, two direction, multiposition, shuffle–dwell radionuclide source irradiator. A typical pathway of process loads is illustrated; during its passage through the irradiation chamber every process load occupies each of the 100 dwell positions for the same time interval.

care products) because of its better energy utilization (absorption efficiency). In this illustration, there are 25 dwell positions for each of the four passes; larger number of passes could also be provided. The arrows in the figure indicate a sequence of process load movements arbitrarily selected for illustrative purposes. In practice, the sequencing of the process loads differs from one irradiator to another and depends on the design of the conveyor system. The important point is that each process load sequentially occupies each of the 100 irradiation positions (in this example) in one cycle through the irradiator, and the process load is not turned on any of its axes during its passage, hence, undergoing two sided irradiation.

In this type of irradiator, the lateral dose distribution in a homogeneous process load is essentially uniform in both the vertical and horizontal directions, as the total dose distribution is the accumulation (average) of all the dose distributions at each irradiation position. This irradiation geometry provides the equivalence of a large product overlap in both the vertical and horizontal directions.

### 3.4.1.4. Other irradiator concepts

Many further variations in the irradiator design are possible, for example,

- (a) A cylindrical source geometry,
- (b) Different methods for transportation of the process loads and presentation of them to the radiation source,
- (c) Two or more source racks,
- (d) Four sided irradiation of process loads,
- (e) Rotation of the process load in front of the source,
- (f) Placement of attenuators to reduce the maximum dose.

These variations are introduced by irradiator designers for many reasons. These include:

- (1) To achieve the required dose uniformity when irradiating products on standard transport pallets,
- (2) To improve the economics of the process by minimizing labour requirements,
- (3) To improve the dose uniformity by providing a better distribution of structural material,
- (4) To maintain refrigerated or frozen products at the required temperatures.

Of these variations, only the irradiator using a cylindrical source geometry and four sided irradiation is described here. This design can be used in a continuous or a shuffle–dwell mode and can be used for the irradiation of products on pallets. As an example, a carousel type system is shown in Fig. 20. It is a one direction source

overlap system and the process loads undergo four sided irradiation. The source itself is usually a cylindrical array of vertical <sup>60</sup>Co rods, arranged along the periphery of the source support. It should be noted that the process loads always maintain their orientation and, hence, are irradiated equally from all four sides during each complete lap. Many industrial irradiators use this design concept because of the processing flexibility: the timing is set such that one lap around the source gives only a partial dose, requiring several laps to accumulate the required dose. This allows different doses to be given to different types of product simultaneously. For example, the facility may be set for a dose of 2 kGy per lap. A specific process load may be withdrawn after five laps if its target dose is 10 kGy, whereas other process loads complete ten laps each for their target dose of 20 kGy. This is possible in this particular design since the product in one process load does not significantly affect the dose in other process loads. Modern computer controlled irradiation facilities allow control to be kept over rather complex loading and circulating regimes.

Four sided irradiation can also be performed in a stationary type irradiator by continuously rotating the process loads (on turntables) or by rotating the process loads through  $90^{\circ}$  after every quarter of the total irradiation time. Thus, each of the four sides of the process load faces the source for the same time duration. The effect

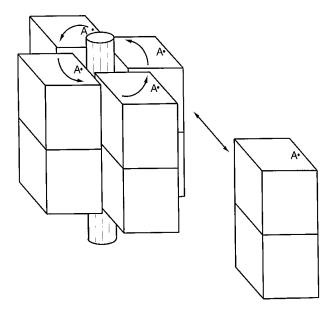


FIG. 20. Sequence of irradiation of a rectangular process load in a one direction carousel-type radionuclide source irradiator. The fixed position A on the side surface of the process load indicates that its movement around the source results in four sided irradiation.

of the four sided irradiation is to minimize depth—dose variations. This results in the minimum dose lying along a line parallel to the axis of rotation near the centre of the process load. Variations in the dose distribution along such lines are minimized, as in other irradiators, by using source overlap and/or source activity augmentation.

#### 3.4.2. Accelerator irradiators

The terminology used to describe the various types of accelerator irradiator (sometimes also called machine source irradiators) is analogous to that used for radionuclide source irradiators. The primary radiation in machine irradiators is by electrons accelerated to high energies. Other accelerated particles are not used in food irradiation. In such irradiators, the electron beam emerging from the accelerator can be used for treatment of food. Alternately, this electron beam can be converted into X rays (also called bremsstrahlung) through the use of a conversion target. The electrons are incident on high atomic number target material (the most commonly used converters are tungsten and tantalum), and the difference in the kinetic energies of the incident electrons and the decelerated electrons emerges in the form of photon energy.

Two basic types of accelerator (based on the physical mode of particle acceleration) are in common use,

- (a) The steady current type (e.g. Dynamitron and van de Graaff), where the accelerating high voltage is actually present in the facility;
- (b) The pulsed beam type (e.g. LINAC and Rhodotron), where microwave energy in a resonant cavity is converted into acceleration and relativistic mass augmentation of the electrons.

For both cases, continuously moving conveyors are utilized to achieve a more uniform lateral dose distribution in the direction of the product motion. The dose distribution in the lateral direction perpendicular to the direction of motion of the product is usually made more uniform by scanning the beam electromagnetically. With beam scanning, the scan frequency and the conveyor speed must be coordinated so as to ensure that the entire process load is evenly irradiated. For the same reason, in a pulsed beam accelerator, the pulse repetition rate as well as the scanning frequency and the conveyor speed must be co-ordinated. These considerations also apply if the electron beam is converted into bremsstrahlung (X rays). Some irradiators are designed to give a very broad beam of electrons striking an extended converter. This converter may be as large as an isotope plaque source so as to achieve dose uniformity by source overlap. The electronic control of the accelerator together with on-line measurements of the electron flux render information which can be used for feedback and real time control of the dose distribution [71–73].

## 3.4.2.1. Stationary type accelerator irradiators

The only well defined example of a stationary type accelerator irradiator was the pot anode X ray tube system used for the demonstration of fish irradiation which is no longer in use [74]. In this system, the product was placed within the irradiation chamber (inside the pot) and was irradiated by bremsstrahlung from all sides simultaneously. Upon completion of the irradiation the X ray generator was turned off, the irradiated product was removed and a new batch inserted.

# 3.4.2.2. One direction one pass irradiators

In this concept the process load is moved through the electron beam in a direction of motion that is perpendicular to the direction of the beam. The effect of the motion in this type of irradiator is the same as that for radionuclide source irradiators, i.e. flattening of the lateral dose distribution in the process load in the direction of the motion. With 10 MeV electrons, for example, only one sided irradiation is necessary, provided that the thickness of the process load is restricted to ensure adequate depth—dose uniformity. For high dose applications, it is sometimes necessary to subject the product to more than one irradiation cycle. Dose distributions in the lateral direction perpendicular to the direction of product motion are flattened by employing beam overlap and/or by using side scatter plates (Fig. 21). A two sided irradiation may be performed in order to increase the usable thickness of the treated process load (see Section 4.3.3.2 for more discussion). However, maximum permissible thickness is again limited by the physical range of the electrons to ensure required dose uniformity.

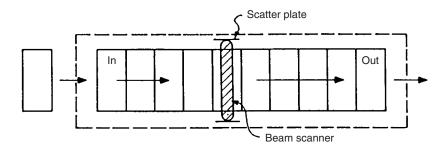


FIG. 21. Sequence of irradiation in a one direction, one pass electron irradiator with beam overlap and scatter plates as illustrated. A process load passes through the irradiation zone from the position 'in' to the position 'out'.

This kind of design is most common with industrial irradiation facilities, where the transport system may vary from conveyor belts, to carriers on several types of rails and even to bulk flow of particulate materials. It provides great flexibility as, in principle, each process load could be irradiated according to its specific treatment procedure by adjusting the beam parameters and transport characteristics by use of computer controls.

## 3.4.2.3. Two direction two pass irradiators

In this type of irradiator, the process load may also be shifted along the scan direction after one pass through the radiation zone, and then moved in the opposite direction for the second pass through the radiation zone. This is achieved, as illustrated in Fig. 22 by passing the process load through the beam from 'in' to the end of the first conveyor, from where it is shifted onto the second conveyor. It finally returns through the beam to 'out', completing a second pass; the irradiation is one sided, however. This procedure increases the lateral dose uniformity in the process load.

The two direction two pass irradiator giving two sided irradiation uses an identical product flow scheme as in the irradiator described above, except that the process load is turned through  $180^{\circ}$  on its second pass, resulting in an improved depth-dose distribution. The two types of two direction two pass irradiator are feasible concepts with both machine sources, electrons and X rays. This design

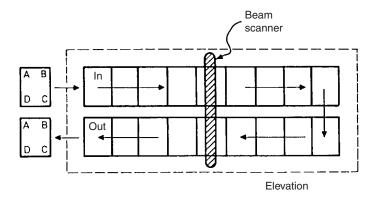


FIG. 22. Irradiation sequence in a two pass electron irradiator. After irradiation on one conveyor, the process loads are moved (but not turned) to the second conveyor and then passed along a parallel path through the other half of the scanned beam. A process load passes through the irradiation zone from position 'in' to position 'out'.

principle when adapted to the special requirements of radiation processing of wires, cables, tubes and hoses is expanded to multipass irradiators, where the product is threaded in a rather large number of loops through the radiation zone.

# 3.4.2.4. Beam scanning and dose uniformity

As mentioned earlier, the electron beam is scanned to improve the dose uniformity across the width of the conveyor. Normally the beam is scanned in one of two ways,

- (a) It goes to and fro across the conveyor, resulting in an overlap of the trace at each end of the scan (as shown in Fig. 23(a)),
- (b) It goes in one direction across the conveyor followed by a very rapid fly-back, resulting in an even trace of the beam without any overlap zone (as shown in Fig. 23(b)) [72].

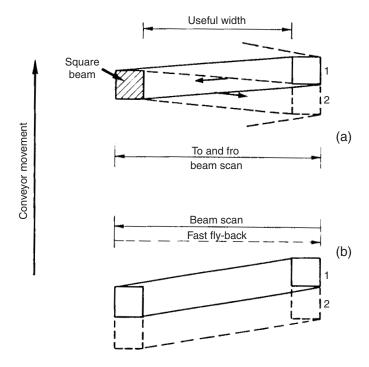


FIG. 23. Lateral beam path patterns on the top surface of a process load for a scanned beam electron irradiator, with simultaneous conveyor movement and beam scanning: (a) scanning to and fro; (b) scanning in one direction with an extremely fast return. (Note: The rectangles represent the trace of the beam spot on the product, see text.)

For convenience, a square cross-section of the beam trace on the surface of the process load is assumed in these figures. In real cases the beam spot profile must be measured carefully; usually it has an elliptical shape with axis lengths between 2 and 10 cm. The dose distribution within the beam cross-section is generally bell shaped and the contour line at 50% of the maximum dose is used to define the beam cross-section.

Continuous beams: In most continuous beam accelerators, the scan frequency is high enough (100–300 Hz) to ensure that, for any given conveyor speed, multiple overlap of successive scan traces is achieved resulting in lateral dose uniformity. Many facilities of this type — especially for high dose applications — utilize to and fro scanning; the wedge in the beam trace on both ends of the scan does not give rise to significant overdoses. The conveyor speed, of course, must not be set so high as to result in a gap between the successive traces.

Pulsed beams: In pulsed beam accelerators, the pulse repetition rate is usually limited to  $\leq 300$  pulses per second. This places limits on the scan frequency that can be used, which, in turn, imposes limits on conveyor speeds. Dose uniformity can be obtained only by the proper harmonization of scan frequency, pulse repetition rate and the conveyor speed to achieve optimum contiguity of successive pulses irradiating the product.

### 4. PROCESS VALIDATION

#### 4.1. GENERAL

The operator of an irradiation facility must show that the entire radiation process is continuously under control, so that the necessary documentation can be provided to demonstrate that all the parts of the product in every process load have received the specified treatment. A complete validation programme must be followed in order to provide the required documentation for approval of an irradiated product. Such a validation programme consists of the following elements:

- Product qualification (setting the maximum and minimum dose limits);
- Facility qualification (characterization of the equipment such as the irradiator, conveyor system and dosimetry system);
- Process qualification (dose mapping and selection of process parameters);
- Documentation and certification.

This validation programme is based on that for sterilization of health care products prepared by ISO [10]. Each of these steps and the final release of the irradiated product have a great reliance on the appropriate dosimetry [75].

Process validation is one of the prerequisites for starting HACCP procedures for describing the nature of the product and the process and for analysing the associated hazards (Section 5.4).

# 4.2. PRODUCT QUALIFICATION

In general, the dose values used by the food technologist to define a process and which eventually become the regulatory dose limits recommended by the health authorities, are derived from experience gained in food irradiation research. The lower dose limit is set so as to achieve the desired effect in the food product. The upper dose limit is selected so as to avoid any detrimental effects on the food product as well as any unwanted effects on the food packaging material if applicable. On the basis of such data, the process parameters are set to achieve the process at commercial facilities.

Thus, it is very important that dosimetry be properly carried out even during the research phase and be accurately documented (ASTM Standard E1900 [45]). The dose given to the small research sample should be quite uniform (i.e. the uniformity ratio is close to unity) in order to correlate the observed effects as closely as possible to dose and to clearly demonstrate how the beneficial effect of the process depends on dose.

# 4.3. FACILITY QUALIFICATION

#### **4.3.1.** General

The commissioning and the subsequent facility qualification are the responsibility of the facility owner. Facility qualification includes the concepts of testing, calibrating and characterizing the equipment at the facility. This includes the source and its ancillary control system, the conveyor system, any weighing equipment and the dosimetry system(s) in use at the facility. This should be carried out after the commissioning of the facility, and repeated annually, and whenever changes are introduced that may influence dose or dose distribution in irradiated products.

The purpose of dosimetry in qualifying an irradiation facility is to establish baseline data for evaluating facility effectiveness, predictability and reproducibility for the range of conditions of operation. In other words, the aim is to understand the behaviour of the facility thoroughly. For example, dosimetry should be used (ASTM Standards E1204, E1431 and E1649 [51, 52, 54]):

(a) To characterize the facility, including establishing relationships between absorbed dose and operating parameters of the facility;

- (b) To measure absorbed dose distributions in reference materials with the reference geometry;
- (c) To characterize absorbed dose variations when processing parameters fluctuate statistically and through normal operations.

The measurement of dose by the dosimetry system(s) should be traceable to national or international standards, and any uncertainty in the measurements and the associated readout equipment should be known. The dosimetry system should be carefully selected for this purpose (Section 2.5) and calibrated (Section 2.4.2).

### **4.3.2.** Characterization of the facility

Before the irradiation facility can be used for commercial purposes, it should be thoroughly characterized [76, 77]. Since the dose absorbed by the product is affected by various parameters, relations between the dose and these parameters should be determined over the full operational range of the parameters. These parameters include source strength and source arrangement, conveyor speed or dwell time, multipass mode, irradiation geometry and bulk density of the process load. For a machine source, there are also other parameters that are important, such as beam current, beam energy, beam spot, scan width and scan frequency.

#### 4.3.2.1. Radionuclide source irradiators

The process equipment, including the radiation source, conveyor mechanisms, safety devices and ancillary systems, should be tested to verify satisfactory operation within the design specifications. All the equipment should also be calibrated at regular intervals, including irradiator cycle timers or conveyor speed and weighing equipment.

The dose delivered to the product in an irradiator depends strongly on either the selected dwell time or conveyor speed, and it is most frequently used to control the dose to the product. Dose also depends on the bulk density of the process load. Delivery of the same dose to a product takes longer as the bulk density increases. These relationships should be established during facility qualification; this understanding is of practical help during process qualification and the operation of the facility. For this purpose, process loads with either real products or dummy products may be used. The bulk density of the dummy products should be chosen to be the same as, or as close as possible to, the mean bulk density of the products that are expected to be irradiated at the facility. The dosimeters are placed, by preference, in locations where the minimum dose is expected. The data should then be analysed using regression analysis to obtain the relationships between the variables. Some examples are given in Figs 24 and 25. It can be seen from Fig. 24 that dose depends linearly on dwell time; however, the intercept on the y axis suggests that in some

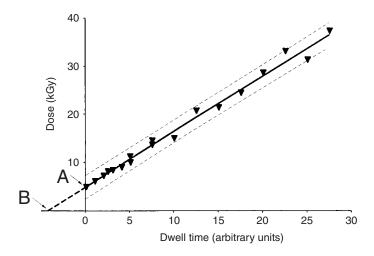


FIG. 24. Minimum dose as a function of dwell time; each data point is the mean of three dosimeter films per dwell time setting. Linear regression of the data results in  $A = 4.86 \pm 0.40$  (kGy),  $m = 1.15 \pm 0.03$  (kGy/unit time); where A is the sum of the transit dose and the shuffle dose, and m is the slope of the regression line. The dashed lines are 95% confidence limits.

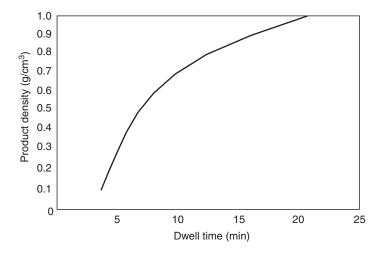


FIG. 25. Dwell time as a function of bulk density of the process load for a  $^{60}$ Co irradiator (0.5 MCi). In this example, the product receives a minimum dose of 1 kGy.

irradiators there is a finite value for transit doses. In such cases, dose is not directly proportional to the dwell time. Figure 25 shows a typical relationship between the product bulk density and the dwell time to give the same dose [78].

### 4.3.2.2. Accelerator irradiators

Characterization of an accelerator irradiator would also include measuring the mean energy of the electron beam, beam spot profile and scan width [54]; information about the last two parameters helps to ensure that the dose is uniformly delivered on the surface of a process load. The penetration of the electrons depends on the beam energy. It is measured by determining the depth—dose distribution along the beam axis in a reference material, usually water or polystyrene. Figure 26 gives a typical depth—dose distribution which is generally measured by exposing several thin film dosimeters at different depths in the reference material. The thickness designated  $r_{50}$  (half-value depth in water) can be used for estimation of the mean electron beam energy based on the following relationship (ASTM Standard E1649 [54] and Ref. [79]):

$$E_{\text{mean}} = 2.33r_{50} \tag{3}$$

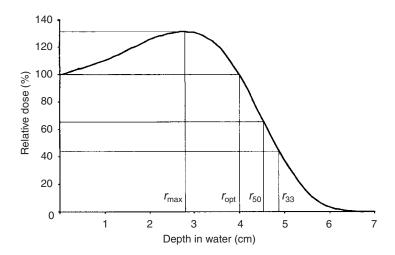


FIG. 26. Depth—dose curve for 10 MeV electrons in water, where the entrance (surface) dose is 100%. The various ranges are identified as:  $r_{max}$  is the depth at which the maximum dose occurs,  $r_{opt}$  is the depth at which the dose equals the entrance dose,  $r_{50}$  is the depth at which the dose equals half of the maximum dose and  $r_{33}$  is the depth at which the dose equals a third of the maximum dose.

For the example of Fig. 26,  $E_{\rm mean}$  is calculated to be 10.6 MeV for  $r_{50}$  = 4.53 cm (of water).

For the measurement of the beam spot, strips or sheets of film dosimeters are useful. For certain types of facility, this information is necessary to ensure that the subsequent pulses overlap as the beam is scanned. The scan width may be conveniently measured by placing several small dosimeters or strips of dosimeter film along the scan direction. This information is necessary to ensure that the radiation zone covers the lateral size of the process load expected to be irradiated. Figure 27 shows a typical dose distribution along the scan width (direction perpendicular to the conveyor motion) [54].

Similar to the characterization with radionuclide source irradiators (Section 4.3.2.1), the relationships of conveyor speed with dose and bulk density should also be established. For electron facilities, quite often the pulse frequency (pulse repetition rate, number of pulses per second) is increased to deliver higher doses instead of changing the conveyor speed. This gives a larger flexibility to such irradiators.

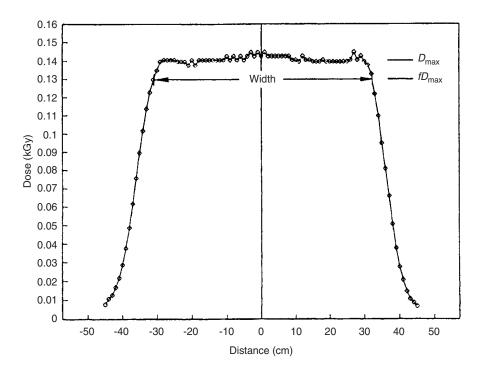


FIG. 27. A typical dose distribution along the scan direction for an electron irradiator. The scan width may be defined as the width at some defined fractional level (90% in this example) of the average maximum dose [54].

## 4.3.2.3. Dosimetry systems

The necessary dosimetry systems should be carefully selected on the basis of the criteria listed in Section 2.3. A facility should have at least one routine dosimetry system; however, it is advisable to have two in the case of unexpected problems. In addition, some facilities have a reference dosimetry system; however, it is not absolutely necessary. The selected dosimetry system(s) should be well characterized as discussed in Section 2.4.2. Additionally, the accuracy of the dose measurements depends on the correct operation of the analytical equipment used to measure the dosimeter response. It should be regularly calibrated and its performance checked periodically to ensure that it is within specifications.

## 4.3.3. Reference dose mapping

It is very important to locate the regions of maximum and minimum dose in a process load so as to determine the capability of an irradiator. This is achieved by establishing the three dimensional dose distribution (dose mapping) in a reference process load. This also characterizes the irradiator with respect to the magnitude, distribution and reproducibility of dose delivery. For this purpose the process load should be filled to its design limits with material (real or dummy) of homogeneous density within the limits of the bulk density range for which the irradiator is to be used. If there is more than one product path through the irradiator, dose mapping should be carried out for each path. This helps define the operating capability of the facility.

Such dose mapping generally requires placing about 50–100 dosimeters in the process load. Dosimeters should be selected on the basis of the irradiation geometry; the size of the dosimeters should be such that they can spatially resolve the dose variation in the process load. For example, thin film dosimeters are essential for electron facilities. Since the aim is to identify the location of extreme doses, more dosimeters should be placed in those regions where such dose values are expected from general knowledge or previous experience with similar facilities or from theoretical calculations [60-62]. Some examples of typical dose distributions are given later in this section. Dose mapping should be performed for several reference materials with bulk densities covering the range expected during commercial operation. An increase in the product density generally results in a decrease in the minimum dose. The maximum dose may not change appreciably (for a given dwell time) or it may decrease, but to a lesser degree than the minimum dose; thus, the dose uniformity ratio increases. Changes in the source loading, source geometry or product transport system can affect the dose distribution. In such cases, this exercise should be repeated.

The procedures for dose mapping described in this section may not be feasible for some types of bulk flow irradiators. In this case, extreme doses should be

estimated by using an appropriate number of dosimeters mixed randomly with and carried by the product through the irradiation zone. Enough dosimeters should be used to obtain statistically significant results. It is, of course, possible to calculate the dose distribution in an *ideal* product [80]. In general, agreement between calculated and measured dose distributions is reasonable.

Examples are given below that illustrate general dose distribution patterns in large scale irradiators.

#### 4.3.3.1. Radionuclide source irradiators

Any type of  $\gamma$  ray plaque source irradiator (such as those shown in Figs 15 and 16) designed to give a two sided irradiation to a rectangular process load has  $D_{\rm max}$  somewhere in the outside (surface) planes, which are parallel to the source plaque, and  $D_{\rm min}$  somewhere in the midplane, which is also parallel to the source plaque. This is illustrated in Fig. 28. For this geometry, the positions of  $D_{\rm max}$  are found along

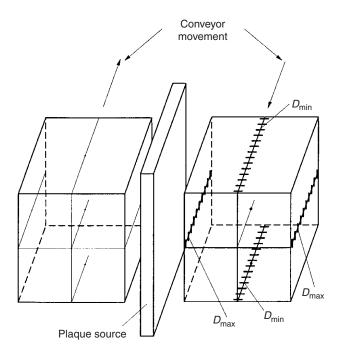


FIG. 28. Regions of  $D_{min}$  and  $D_{max}$  (indicated by hatching) for a rectangular process load after two passes, one on each side of a stationary  $\gamma$  ray plaque source.

<sup>&</sup>lt;sup>9</sup> There are several modern codes for dose calculations, such as FUGI, FUDGE-4A, ETRAN, EGS4, SANDYL, ITS ('Tiger series'), CEPX, CEPXS and MCNP.

a centre line of the outside planes of the process load parallel to the direction of motion, and the positions of  $D_{\min}$  are found on the edges of the midplane. This is also valid for an irradiator with a cylindrical source arrangement (Fig. 20). This is true for product overlap irradiators. However, for source overlap irradiators, the position of  $D_{\min}$  moves from the edges of the midplane into the inside of the process load but still on this plane. This is common with certain multipurpose irradiators where the position of the minimum dose is not accessible for dosimetry during routine processing. With stationary irradiation (which is often operated under source overlap conditions) or one direction, two sided irradiation (which is effectively source overlap, see also Fig. 18), the position of  $D_{\max}$  is at the centre of the outside planes (the middle of the  $D_{\max}$  lines in Fig. 28) and that of  $D_{\min}$  is on the four corner edges of the midplane (parallel to the motion and perpendicular to the source plaque).

#### 4.3.3.2. Accelerator irradiators

When processing with electrons, it is important to realize that there is always a dose buildup region within the product [54, 79]. The maximum of the dose will always occur under the surface, i.e. inside the product. For mean energies of about 10 MeV, the characteristics of the depth-dose distribution in water are presented in Fig. 26. Normalizing the surface dose to 100%, the maximum dose  $D_{\rm max}$  of 130% occurs at a depth  $r_{\text{max}} = 2.8$  cm, and the entrance dose equals the exit dose at  $r_{\text{opt}} =$ 4.0 cm. For a process load of thickness between 2.8 and 4.0 cm, the dose uniformity ratio is constant with a value of about 1.3. If the process can allow a uniformity ratio of 2, the maximum useful thickness of the process load is  $r_{50} = 4.5$  cm where the exit dose equals half of the maximum dose. If a uniformity ratio of 3 is acceptable, the maximum allowable thickness can be as much as  $r_{33} = 4.8$  cm (where the exit dose equals a third of the maximum dose). The dependence of dose uniformity ratio with the thickness of the process load for the situation of one sided irradiation is given in Fig. 29 (solid curve). A steep increase in the uniformity ratio is observed as soon as the thickness exceeds the optimal range  $r_{\rm opt}$ . It approaches infinity when the maximum range of the electrons (about 6.5 cm for 10 MeV) is exceeded; any product behind that range remains untreated. Two sided irradiation can overcome this restriction and extend the processable thickness, as shown in Fig. 30. The resulting overlapping dose distributions for several thicknesses of the process load are depicted in Fig. 31. At a thickness of 6 cm (water equivalent), a dose peak is observed in the centre of the product with a uniformity ratio of about 2.5. Increasing the thickness to 9 cm results in an extremely smooth depth-dose distribution, and at a thickness of 9.5 cm the minimum possible uniformity ratio of 1.3 is achieved, which was reached for one sided electron processing at  $r_{\rm opt}$  = 4.0 cm. Increasing the thickness further causes severe underexposure in the centre. The behaviour of the dose uniformity ratio for two sided irradiation with 10 MeV electrons is also shown in Fig. 29 (dashed curve).

In a French electron beam facility for the processing of mechanically deboned poultry meat, the thickness of the deep frozen slabs of the minced meat is kept at 7 cm, which is close to 6.5 cm where a local maximum of the uniformity ratio of 2.5 occurs (Fig. 29). The advantage of this choice is that any slight variation of slab thickness or density will not increase the uniformity ratio above this value; the process is stable with regard to such variations.

In one sided irradiation,  $D_{\rm max}$  is likely to lie along a line parallel to the direction of motion of the product, through its centre or closer to the surface (Fig. 32).  $D_{\rm max}$  may extend over a midplane through this zone quite close to the edges of the slab.  $D_{\rm min}$  is found along lines parallel to the direction of the motion of the product, running through the side edges at the bottom of the process load; there are two effects that cause this. Firstly, the scanned beam strikes the surface of the process load at a small angle at the side edges, and secondly the scattered radiation from the process load will not be compensated for fully by scattered radiation from the surrounding air. If the consecutive process loads are not in contact, similar effects will also be present at the leading and trailing surface planes perpendicular to the direction of motion.

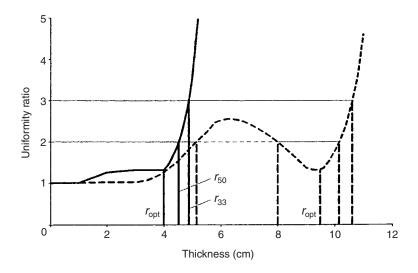


FIG. 29. Dependence of dose uniformity ratio on the thickness of water absorber for 10 MeV electrons; the solid curve refers to one sided irradiation and the dashed curve refers to two sided irradiation. For one sided irradiation  $r_{opt}$ ,  $r_{50}$  and  $r_{33}$  are also identified (solid vertical lines) as defined in Fig. 26. The vertical dashed line marked  $r_{opt}$  identifies the thickness for the smallest achievable dose uniformity ratio for two sided irradiation. (Note: The central dose can reach about 2.5 times the entrance dose for two sided irradiation for an absorber thickness of about 6.5 cm of water.)

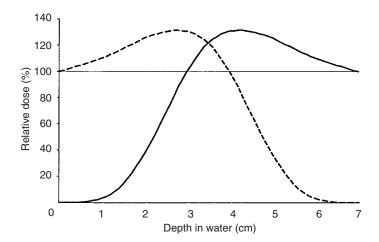


FIG. 30. Depth–dose distributions for 10 MeV electrons for two sided irradiation. Each curve refers to the dose distribution for one sided irradiation as shown in Fig. 26; dashed curve, irradiation from left hand side; solid curve, irradiation from right hand side.

For two sided (two pass, with turning of the process load) irradiation with high energy electrons (Fig. 33), the position of  $D_{\rm max}$  will be somewhere midway between the two outside planes, along two lines parallel to the motion of the product and about halfway between the top and bottom surfaces.  $D_{\rm min}$  will be found along lines parallel to the direction of motion of the product either through the side edges at the top and bottom of the process load or in the midplane at the side edges. In the case that the process load thickness is larger than the optimum thickness for two sided irradiation,  $D_{\rm min}$  is found on the midplane through the process load perpendicular to the direction of the electron beam where generally  $D_{\rm max}$  is expected to occur when the process load thickness is less than the optimum thickness.

## 4.3.4. System variability

The extent of the variability in the measured dose and the dose distribution for a reference geometry and for a set of operating parameters should be established. This includes variability due to fluctuations in the values of the operating parameters and the intrinsic variability in the dosimetry system [81]. The magnitude of these variations may be estimated, for example, by passing dosimeters in the reference geometry through the irradiation zone on the product conveyor at time intervals appropriate to the frequency of the parameter fluctuations. The reference geometry for the irradiated material is selected so that the placement of the dosimeters on and within the material will not affect the reproducibility of the measurements.

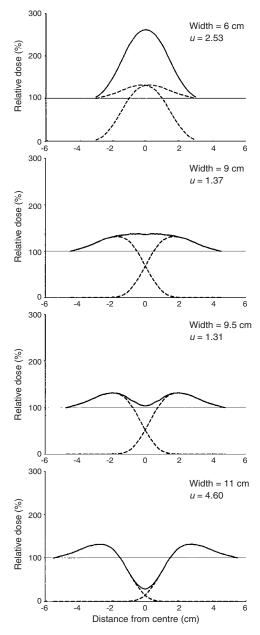


FIG 31. Depth—dose distributions for 10 MeV electrons in varying thicknesses (widths) of water absorber; the dashed curves represent the distributions for one sided irradiation from each side, and the solid curves represent the sum of the two distributions. The dose uniformity ratio, u, varies with the absorber thickness, going through a minimum value of 1.31 at about 9.5 cm of water. (Note: The fine horizontal lines represent the surface dose for one sided irradiation normalized to 100%.)

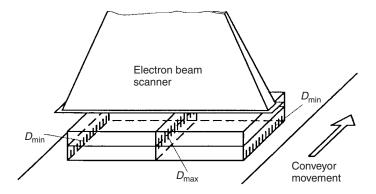


FIG. 32. Regions of  $D_{min}$  and  $D_{max}$  (indicated by hatching) for a rectangular process load for single pass electron beam irradiation.

### 4.4. PROCESS QUALIFICATION

## 4.4.1. Objectives

The most common problem in establishing a new treatment is the process of upscaling. When the effectiveness of radiation processing of some product has been proven in a well controlled laboratory situation, the results now need to be transferred to the real world of agriculture and the food industry and adapted to commercial handling. Factors that influence the process include: the nature of the product including its seasonality, the quality, the physiological status, the storage method, packaging and transport, intended use and the purpose of the treatment. For example, sprout inhibition is studied with a few potatoes in small boxes at a research laboratory; however, agricultural handling in many instances requires bulk transport after harvest and storage in large heaps or transfer to crates of 1 m<sup>3</sup> or more in volume which are finally stacked in a warehouse. The dose distribution throughout the product and the achievable maximum and minimum doses strongly depend on these geometric constraints. Appropriate dosimetry is indispensable to meet the specification for an effective process when initiating/establishing a treatment.

Thus, the purpose of dosimetry in process qualification is to ensure that the absorbed dose requirements for a particular product and process can be satisfied, for example, the dose received by every part of the product must be between the two specified dose limits,  $D_{\rm max}({\rm limit})$  and  $D_{\rm min}({\rm limit})$ . Thus, the objectives of the process qualification are:

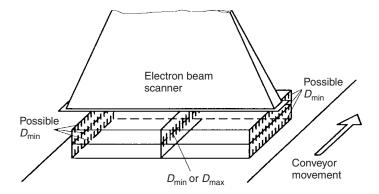


FIG. 33. Regions of  $D_{min}$  and  $D_{max}$  (indicated by hatching) for a rectangular process load for two pass, two sided electron beam irradiation. (Note: Depending on the process load thickness with respect to the electron energy/range, either  $D_{min}$  or  $D_{max}$  may occur in the centre.)

- (a) To establish process parameters (such as dwell time, electron beam parameters, conveyor speed, irradiation geometry and product loading configuration) for the given process that satisfies the dose specifications;
- (b) To identify reference location(s) on (or near) the process load where a routine dosimeter can be placed for process control.

These objectives are accomplished by establishing the dose distribution throughout the process load with a specific product and product loading configuration using the dosimetry procedures described in this section. This exercise (called dose mapping) determines the magnitude and locations (regions) of the maximum and minimum doses in the process load, and helps establish all the process parameters necessary to achieve the absorbed dose within the two specified limits [82].

# 4.4.2. Product dose mapping

The various methods of investigating dose distributions inside the product include those utilizing experimental data from the placement of thin films [32, 37, 83] or thin film strips [33, 34, 36] throughout the product, theoretical calculations [32, 35, 38] and graphical methods based upon statistical analysis of experimental data [84, 85]. The experimental dose mapping procedure is similar to that discussed in Section 4.3.3 except that during facility qualification the process load was of *inhomogeneous* reference material. In contrast, the process load here is the actual

process load of a customer with a real product, which is quite often *inhomogeneous* [86]. The dose distribution should be measured thoroughly in at least one process load — generally requiring 50–100 dosimeters [87]. The placement of dosimeters should be more concentrated where extreme doses are expected on the basis of the facility dose mapping exercise. In a process load that contains voids or non-uniform products, dosimeters should be placed at locations where discontinuities in composition or density may affect the regions of maximum or minimum dose [88]. Knowledge of the dose distribution throughout the process load is not as important as the determination of the two extreme values of the dose and their locations in the process load. Only in very rare applications does the mean value of the dose have any significance, for example, where the desired effect is a total chemical turnover. For the purpose of dose mapping, it is assumed that subsequent process loads in a production run receive essentially the same dose and dose distribution except for the fluctuations in bulk density and process parameters discussed in Section 4.4.3.

During design of the irradiation facilities, the expected dose distributions are often calculated using computer programs [89, 90]. It has been demonstrated that such calculations agree reasonably well with experimental data. The computed data available from the designer of the irradiation facility can often be used for process loads that are fairly homogeneous, in preference to carrying out the laborious dose mappings described above. Consequently, the locations of the extremes of the dose values need only to be checked, taking measurements with a few dosimeters, strategically placed.

## 4.4.2.1. Special process loads

Shallow irradiations: In shallow irradiation treatments using electrons of a few megaelectronvolts or less (soft electrons), the dose distributions are different from those discussed earlier. The dose distribution may change markedly in the first few millimetres of the product; in such cases the surface dose and the peak dose become important parameters (see Refs [83, 91, 92] and ASTM Standard E1818 [93]). The peak dose is about 2 and 3 times the surface dose for 1 and 0.5 MeV electrons, respectively (ASTM Standard E1649 [54]). It is extremely important that the position in the product for the dose quoted should be clearly specified for all surface irradiations using low energy electrons.

In certain soft electron applications, the particulate goods pass through the irradiation zone in a randomized movement (spouted bed or vibrating trough). This causes the individual particles to rotate and to expose equally all sides of the surface to the electron beam. Dosimetry in such situations is virtually impossible and is generally replaced by control of the effect. For example, grain irradiation aims at elimination of the microflora adhering to kernel surfaces; sufficient reduction of these microflora can be proven by various microbiological plating techniques. On the other hand, the treatment also aims at shielding the inner parts of the kernel from radiation;

this can be proven by a germination test of the seeds or by a chemical analysis of the flour from such milled grains. The process is also suitable for some spices, such as whole pepper [63, 94–96].

Bulk flow and liquid flow irradiators: There are situations where the expected positions for the minimum and maximum doses are not accessible for dosimetry measurement; for example, the turbulent flow of particulates or liquid materials through the irradiation zone. In this situation, suitable dosimeters may be added to the stream of the goods in a large enough number to allow extrapolation for estimating the extreme dose values [97–106]. However, at present no commercial application of radiation processing of particulates or liquid bulk food material is known except for the treatment of some Chinese spirits to remove off-flavour. Some examples of such irradiators pertain to sewage sludge irradiation [107–110]; the similarity in dosimetry techniques, however, lends itself to food applications.

Chilled or frozen foods: For chilled or frozen foods, absorbed dose mapping for process qualification may be performed with the product at room temperature. This requires that there be no change in any parameter (other than temperature) that may affect the absorbed dose during processing of the chilled or frozen food. Alternatively, absorbed dose mapping at the temperature of the chilled or frozen food may be performed using a dosimetry system that can be calibrated at that temperature. The temperature of the food (and thus of the dosimeters) must be maintained relatively constant during irradiation (e.g., by using insulated totes).

Transit dose: In some cases, especially with relatively short residence (or dwell) times (e.g., low dose applications in facilities designed for higher doses), the transit dose (i.e. the dose received by the product in its movement into and out of the irradiation field) plus the shuffle dose (i.e. the dose received by the product during its movement from one dwell position to the next) may be a significant fraction of the total dose. In such cases, the dose distribution in the product may be significantly different from that estimated for a stationary irradiation geometry, as discussed above. This effect should be investigated and accounted for. In most practical cases, however, the process parameters are such that the transit plus shuffle dose does not significantly affect the dose distribution, even though its contribution to the total dose may be significant.

## 4.4.3. Verification process

The distribution of absorbed dose in irradiated food products depends on many factors, such as plant design, type and kind of product and type and energy of radiation. These factors will not normally vary during a given irradiation treatment. However, owing to the statistical nature of the irradiation process, there are fluctuations in the values of some process parameters affecting the dose distribution [92, 111, 112]. In practice, variability is unavoidable in any radiation process as a result of several effects, including:

- (a) Variations in bulk density between process loads,
- (b) Variability in the product configuration between process loads,
- (c) Placement of dosimeters may not be made at similar locations for different process loads,
- (d) Statistical fluctuations of the operating parameters during irradiation,
- (e) Uncertainty in the dosimetry system itself.

The extent of the variability can be measured by random selection of n (n may be between 3 and 10) nominally identical process loads from the total batch of the product. Several dosimeters should be placed in each process load in the regions that are expected to receive the minimum and maximum doses, as identified during the dose mapping exercise of Section 4.4.2. The objective is to determine the magnitude of the maximum and minimum dose values for each of the n process loads. These will not have exactly the same values due to the factors discussed above. These n values are expected to follow a normal (Gaussian) distribution characterized by two parameters, the mean and the standard deviation. The two standard deviations for the maximum and minimum doses may be referred to as  $s_{\text{max}}$  and  $s_{\text{min}}$ , respectively; these may not have the same value. This exercise is sometimes referred to as the verification process.

To ensure that the delivered dose remains within the two specified regulatory dose limits,  $D_{\rm max}({\rm limit})$  and  $D_{\rm min}({\rm limit})$  in the presence of this variability, the operator must set the process parameters (including dwell time or conveyor speed) so as to deliver a dose between two more restrictive limits. Thus, the lower dose limit is increased while the upper dose limit is decreased, as shown in Fig. 34. These new dose limits are sometimes referred to as target doses and are defined as

$$(Target dose)_{min} = D_{min}(limit) + k_{min} s_{min}$$
(4)

$$(Target dose)_{max} = D_{max}(limit) - k_{max} s_{max}$$
 (5)

where  $k_{\min}$  and  $k_{\max}$  are the one sided tolerance factors [82]. Their values are selected on the basis of the economics of the process (Section 4.4.4).

## 4.4.4. Process parameters

If the process is set for the specified regulatory dose limit, namely  $D_{\min}(\text{limit})$ , it is likely that half of the process loads will receive doses less than this lower dose limit; at least, that is what the routine dosimeters would indicate. However, by setting the process at a higher value, namely (Target dose) $_{\min}$ , this fraction can be reduced. The extent of the reduction depends on the value selected for  $k_{\min}$ ; the higher the

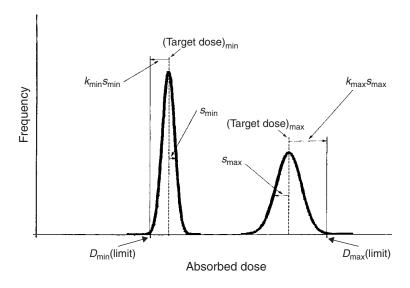


FIG. 34. Determination of the two target dose values based on the frequency distributions of the measured values of  $D_{min}$  and  $D_{max}$  in several process loads, with  $s_{min}$  and  $s_{max}$  being the sample standard deviations.  $k_{min}$  and  $k_{max}$  are the appropriate one sided tolerance factors and their values are selected on the basis of the requirements of the process.

value of  $k_{\min}$ , the lower is the probability that a process load will receive a dose less than  $D_{\min}(\text{limit})$ . However, choosing a very high value for  $k_{\min}$  is uneconomical, so a compromise is made in the selection of  $k_{\min}$ . In essence, the choice is made depending on the required degree of assurance (confidence probability) that, at most, only a certain small fraction of the product is treated at a dose which is less than the lowest acceptable dose,  $D_{\min}(\text{limit})$ . On the basis of this decision,  $k_{\min}$  is selected from the appropriate tables (e.g., Table VI [113]); the value also depends on the number, n, of the process loads used for the verification process. The determination of  $k_{\max}$  is similar.

For example, consider the case where ten process loads (n=10) are used to determine the standard deviations  $s_{\min}$  and  $s_{\max}$  during the verification process. If we require a confidence level of 95% (P=0.95) that only 10% of the product receives a dose less than the acceptable limit  $(\beta=0.1)$ , Table VI yields a value of  $k_{\min}=2.4.$ <sup>10</sup>

 $<sup>^{10}</sup>$  The example quoted here is chosen arbitrarily and the values chosen for *P*,  $\beta$  and *n* should influence neither the operator's choice nor the statistical levels of confidence that might be eventually demanded by the regulatory authorities.

TABLE VI. TOLERANCE FACTOR, k, FOR ONE SIDED TOLERANCE LIMITS

	$P^* = 0.95$					$P^* = 0.975$				
n	β	0.05	0.1	0.15	0.2	β	0.05	0.1	0.15	0.2
8		3.4	2.8	2.2	1.9		3.9	3.2	2.5	2.2
10		3.0	2.4	2.0	1.7		3.4	2.8	2.2	1.9
15		2.6	2.1	1.8	1.5		2.9	2.3	1.9	1.7
20		2.4	1.9	1.6	1.4		2.6	2.1	1.7	1.5
30		2.2	1.8	1.5	1.3		2.4	1.9	1.5	1.4

<sup>\*</sup> P is the level of confidence that at most a fraction  $\beta$  of the measurements will fall below the lower set limit and above the set limit for a test consisting of n random measurements [113].

For combinations not given in Table VI, appropriate tolerance factors may be found in standard statistics handbooks. 11

Considering the revised dose limits, the actual (measured) value of the dose uniformity ratio in a process load should be equal to or less than

$$U(\text{limit}) = (\text{Target dose})_{\text{max}} / (\text{Target dose})_{\text{min}}$$

The irradiation time (or dwell time or conveyor speed) should now be set so that the minimum dose in the process load is equal to  $(Target\ dose)_{min}$ . If, for this setting, the measured dose uniformity ratio is larger than U(limit) some other process parameters need to be adjusted. For some radionuclide facilities, an acceptable value may be achieved by extending the source beyond the boundaries of the process load or the process load may be moved past the source at several different levels. Other methods of improving the absorbed dose uniformity may include arranging the source elements so that those with greater activity are near the perimeter of the source array, using attenuators or compensating dummies, irradiating from four sides, rotating the process load during irradiation and increasing the source-to-product distance. For electron facilities, changing the beam characteristics, for example, by optimizing the electron energy, can reduce the dose uniformity ratio. Other means to reduce the dose uniformity ratio may be employed, such as use of attenuators, scatterers and reflectors. Depending on the bulk density, thickness and heterogeneity of a process load, some processes may require a two sided irradiation to achieve an acceptable

<sup>&</sup>lt;sup>11</sup> It is important to use statistical tables for *one sided* (frequency) distributions only.

dose uniformity ratio. It should be noted that for two sided irradiation, the regions of maximum and minimum dose may be quite different from those for one sided irradiation (Section 4.3.3.2). In addition, caution is necessary for a two (or multiple) sided irradiation for electron facilities because slight variations in thickness or density of the process load or in electron energy can lead to extreme over- or underexposure in the middle of the process load. In the case of bulk flow irradiators (either radionuclide or electron), absorbed dose uniformity can be improved by arranging baffles to control product flow through the irradiation zone. If the dose uniformity ratio is still not acceptable, redesign of the process load may be needed to achieve an acceptable ratio.

If in this procedure any significant changes are made to any of the process parameters that could affect the magnitudes or locations of the absorbed dose extremes, the absorbed dose mapping procedure should be repeated to the extent necessary to establish the effects. If it is found to be significant, process qualification should be repeated starting from the procedure described in Section 4.4.2.

### 4.4.5. Reference dosimetry location

For several irradiation geometries, the position of the minimum dose is inside the process load and not on the surface; hence, the placement of dosimeters for process control during routine irradiation (Section 5.3.2) might be impossible without analysing the process load. For such cases, a convenient reference location should be selected on the surface of or near the process load for process control. The essential requirement during process qualification is that the relationships between the absorbed dose at this alternative reference position and the absorbed dose extremes be established, be shown to be reproducible and documented.

### 4.5. DOCUMENTATION AND CERTIFICATION

The values of all the process parameters that are determined during process qualification that satisfy the required dose specification with a high degree of confidence should be documented for future use in subsequent routine processing of this product.

For a radionuclide facility, such process parameters include the source configuration, irradiation time (or dwell time or conveyor speed) and the path of the process loads around the source. For an electron facility, these include beam characteristics, scanning parameters and conveyor speed. For both types of facility, the process load characteristics are important parameters that should be described in detail, for example the product type, the size and bulk density of the process load and the packing arrangement of the product inside.

## 5. FACILITY OPERATION AND PROCESS CONTROL

#### 5.1. GENERAL

The International Conference on the Acceptance, Control of and Trade in Irradiated Food, held in Geneva in December 1988 recommended that governments should ensure that regulatory procedures for control purposes be adequately applied as a pre-requisite to any processing of food by radiation and its subsequent sale [114]. Certain key principles, namely registration/licensing, regulation and inspection of food irradiation facilities, documentation and labelling of irradiated food, training of control officials, operation of irradiators by trained/certified plant operators, observance of GMPs (see also the Bibliography for the ICGFI Codes of Good Irradiation Practice) and such other guidelines and standards as developed for the purpose must be implemented as part of the overall process control. The Codex Alimentarius General Standard for irradiated foods and the associated code of practice for operation of irradiation facilities including reliable dosimetry should be applied, thus providing an independent means of verification [2]. It is necessary to ensure that irradiated food which enters international trade conforms to uniform and mutually acceptable standards.

As a part of process control and quality assurance in the production of irradiated products for trade, particularly international trade, the ICGFI has set up an International Inventory of Authorized Food Irradiation Facilities on the basis of certain information agreed upon. The inventory has been maintained since 1993, with the latest update in September 1998 [115]. A facility must fulfil certain criteria before it can be included in the inventory. The inventory was created when the Codex Alimentarius General Standard required that facilities need to be licensed and registered for processing of food by ionizing radiation; however, there was no such registry available then. Any irradiated food commodity in the international trade can thus be identified as having been processed in an authorized irradiation facility functioning with standard operating conditions and under regulatory control confirming compliance with GMPs and associated GIPs with provisions for stringent dosimetric requirements and overall process control, inspection and enforcement. The decision of the ICGFI to establish the international inventory, guidelines for food irradiation regulations and a collection of codes on good irradiation practice for individual treatment and handling of food items was aimed at ensuring effective control and reliable treatment for the protection of public health. Such mandatory controls and standard operating procedures add to public confidence in the health control and inspection authorities of countries importing irradiated food. Analytical methods for post factum identification of irradiated food [116], which to some extent can also render dose estimates, are in principle unsuitable for such purposes [117, 118].

In the food industry, the control system that most countries require to ensure safety of processed food items is currently HACCP. This approach is becoming more and more recognized internationally and is already used as a standard for GMPs. A generic standard for irradiation of meat and poultry has been developed.<sup>12</sup>

### 5.2. FACILITY OPERATION

An irradiation facility should be operated and controlled by competent personnel who have successfully completed a recognized course of study such as ICGFI's Food Irradiation Process Control School (FIPCOS). All information related to the product and treatment must be properly documented and available when requested by relevant parties, in particular by regulatory authorities. Hygienic practices that are needed in GMP for other food processes are equally applicable and necessary for the process of irradiation and must be in place for product treatment (see the Bibliography for ASTM Standards and ICGFI publications).

For routine product processing, all the process parameters must be set as determined during process qualification (Section 4) before initiating the process. This includes checking that all the process loads are similarly configured as per specification. The operating parameters must be controlled, monitored and documented to ensure that the product in each process load is processed within specifications. If the operating parameters deviate outside the prescribed processing limits, appropriate action should be taken. This may include stopping the process and investigating the cause of the malfunction. More details on process control are discussed in Section 5.3.

The product transport system can follow several modes of operation as defined by the design of the facility, discussed in Section 3. Consideration of the transport system shows that the movement of product through the facility must be particularly well monitored. With a continuously moving constant speed conveyor, the speed can be calibrated with a tachometer; while for an intermittently moving conveyor the duration of a cycle is governed by presetting the master timer, which controls the successive dwell and shuffle periods. In both cases, adjustment factors may be incorporated to compensate for radioactive decay or for replenishment of the source (monthly adjustment is usually sufficient for radioactivity decay compensation). In addition, to ensure reproducible irradiation conditions, the correct positioning of the

<sup>&</sup>lt;sup>12</sup> This and other HACCP models are available at the International HACCP Alliance web site. It can be accessed at: <a href="http://www.haccpalliance.org/alliance/haccpmodels.html">http://www.haccpalliance.org/alliance/haccpmodels.html</a>. More information is available from K.B. Harris, Executive Director, International HACCP Alliance, College Station, Texas, USA.

process loads relative to the source plaque or source rod system must be electrically or mechanically assured. Furthermore, no conveyor movement should be possible until the source is fully in the irradiation position, and the source must be automatically returned to its shielded storage vault when the conveyor system fails. The interlock for the latter is a positive means of avoiding overdose to the product in the irradiation chamber in the case of mechanical or electrical failure.

For accelerator irradiators, the speed of the conveyor is frequently coupled in some fashion to the beam current, scan frequency and pulse repetition rate so that the dose is properly delivered to the product and the dose uniformity over the product is maintained. Modern accelerators are completely computer controlled, which leads naturally to recording and documenting several parameters [119]. The air gap between the beam outlet window and the process load surface and the scanning width of the beam are preset so that the optimum source-to-product geometry can be maintained [91, 92]. This distance may be variable if process loads of varying thickness are irradiated (particularly in irradiators with vertical beams) and, therefore, must be monitored.

Generally, a homogeneous radiation treatment is only achieved when the irradiation chamber is completely filled with the product to be treated. Thus, during the beginning and end of an irradiation run (when the irradiation chamber is not completely filled), especially the first and last process loads accumulate higher doses during their passage through the irradiator as they are not shielded from the source by other process loads during a significant portion of their pass. This can be mitigated by the use of process loads with 'dummy' products at both the ends of a production run if the dose distribution is found to be unacceptable. Such 'dummy' process loads may consist of the actual product, which is later discarded, or of some compensating materials. In batch operation and incremental dose facilities similar problems may be experienced if products with very different bulk densities are irradiated at the same time. If the dose distributions in some process loads are not acceptable, either dummy products may be used or the products must be sorted and only those products with similar bulk densities irradiated together.

Similar problems may not be encountered in radiation processing by electron beams as only a single process load is in the radiation zone at a time, and with electronic controls it would even be possible to give consecutive process loads completely different radiation treatments (however, this is not done in general). If the consecutive process loads are not in close contact with each other when passing

<sup>&</sup>lt;sup>13</sup> Compensating material (also called simulated product) is a mass of material with radiation attenuation and scattering properties similar to those of the product to be irradiated. When used in dose mapping studies, it is sometimes referred to as phantom material.

through the radiation zone, dose is reduced at the front and rear surfaces because of the loss of radiation energy to the surrounding air by scattering. The same applies to the side surfaces of the process loads.

In radiation processing of bulk products (e.g., grain or onions) special bulk transport system controls (spouted bed, pneumatic transport, vibrating trough conveyor and gravity flow) are indispensable in avoiding bulk density fluctuations in the irradiation zone leading to unacceptably wide dose distributions.

In certain instances there may be requirements for special handling procedures to ensure satisfactory results. An example could be a requirement for a refrigerated fruit to be brought to ambient temperature before irradiation so as to minimize radiation damage resulting from the presence of condensed moisture on its surface. Another example is the requirement in the USA that the temperature of fresh poultry be maintained between -2 and +4°C before, during and after irradiation. The requirement for such specific procedures should also be included in the total process control system.

The ASTM has also published various standard procedures and guidelines for the irradiation of such items as dried spices and herbs, fresh and frozen red meat and poultry, finfish and shellfish, and fresh fruits (see Bibliography). These standards may be used in conjunction with the ASTM Standard Guide for the selection of time temperature indicators ASTM F1416 [120] and the standard guide for packaging materials for foods to be irradiated ASTM F1640 [121]. In addition, the ICGFI has published various titles on good irradiation practices to provide information on product requirements before, during and after irradiation. The IAEA has also published technical documents regarding irradiation of various agricultural products. These are listed in the Bibliography.

Facility parameters that are not directly involved in the radiation treatment, for example safety interlocks and source hoist mechanisms, are not discussed in this book. However, it is important that these are under control for legally correct and technically smooth operation of the facility. Food irradiation facilities incorporate intense radiation sources and, hence, adequate safety precautions are indispensable for ensuring the safety of operation personnel. This subject also falls outside the scope of this book; however, there are several references dealing with safety procedures in irradiation facilities [122–124] and radiation protection [125].

### 5.3. PROCESS CONTROL

To ensure that the process is being correctly administered, i.e. that the whole product is receiving a dose within the specified range, certain process control procedures should be in place. These include documented product handling procedures before, during and after the irradiation, consistent orientation of the

process loads during irradiation, monitoring of the critical process parameters, routine product dosimetry and the documentation of the critical activities.

In considering the entire processing operation of a food irradiation plant, recognition must also be given to those aspects that are not part of the irradiation process per se. For example, receiving, storage and shipping of food must be in accordance with good practices for foods; therefore these plant operation aspects must be included in the process control measures to ensure a totally satisfactory operation.

It is clearly not possible to apply product testing to an entire batch of irradiated food product as a means of process control. In addition, product testing is never legally required. End-point product testing as part of quality control is impractical and ineffective; testing large quantities of product is, in any case, economically prohibitive. Additionally the results of many such tests would not be available until long after the product has been released, or the product needs to be stored while awaiting release for a period longer than its storage life. However, it is useful to carry out product testing as a part of process qualification to ensure that the product is responding to the treatment as expected from the food irradiation research.

The principal elements in process control are

- Monitoring of process parameters
- Routine product dosimetry
- Product control
- Documenting process interruptions (if any).

It must be stressed that process control indispensably requires recording and documentation of the facility operation in general and of dosimetry in particular. The records generated serve the following purposes:

- (a) To provide documentary evidence showing that the product received correct treatment.
- (b) To fulfil obligations to and requirements of the authorities,
- (c) To settle disputes (if any).

Such documentation is also required according to the principles of HACCP (Section 5.4).

### **5.3.1.** Process parameters

All key process parameters that affect the dose in the product must be controlled and monitored. Such parameters consist of operating parameters, process load characteristics and irradiation conditions.

For a radionuclide facility, these include source strength and its configuration, conveyor speed or dwell time and product movement mechanism. Modern information technology has contributed significantly towards reliable control and recording of relevant parameters [126, 127]. For an electron facility, these include electron beam energy, beam current, scan width and frequency, conveyor speed, irradiation geometry, multiple exposure and number of passes. In a well designed irradiation facility, these parameters can be monitored from a control console and recorded automatically and continuously.

No control of operating variables can be effective unless the characteristics of the process load are also monitored and controlled. There is invariably product heterogeneity, such as seasonal crop variations, anomalies in bulk density and uneven local density variations (as in meat and fish). These effects and random packaging of agricultural products inevitably lead to varying process load characteristics, resulting in a varying dose uniformity ratio. In addition, a control chart registering the weight of the process loads helps to maintain an effective plant operation inventory. The control of the irradiation process is simplified since products having similar bulk densities and about the same dose requirements are usually grouped together for treatment.

Quality assurance procedures based on monitoring of the process parameters have advantages over routine product dosimetry (Section 5.3.2) with regard to early alarm when the process is about to drift outside the set limits. Information on process parameters is available during the process, whereas dosimetry results become available only with a certain delay after the process is completed.

# **5.3.2.** Routine product dosimetry

One of the fundamental aspects of process control is dosimetry. As we have seen, it is used in process validation (Section 4), i.e. for product qualification, facility qualification and process qualification. Dosimetry forms the key element for the success of the treatment and for the safety of the process, for the wholesomeness of products and for confidence in effective treatments. Thus, reliable routine dosimeters — traceable to national or international standards — are an important, indeed necessary, adjunct in process control of food irradiation [128]. Now, with the increase in trade in irradiated products, traceable dosimetry has become crucial. In order that the facility operator can certify the dose applied to the food, routine dosimetry of each and every production run is essential, as required in ASTM Standards E1204 [51] and E1431 [52]. This provides a system that relevant authorities worldwide can rely on to ensure that imported products have been treated according to legal requirements. Dosimetry data may also be required in the event of mechanical failures and operational anomalies.

The choice of the routine dosimetry systems must take into account the characteristics of the radiation source and the product [51, 52]. In addition, they

should be selected on the basis of convenience of use and cost. Examples of routine dosimeters are the plastic dosimeters that have a reproducible response to radiation, such as several kinds of Perspex (PMMA) or radiochromic plastic films (Table VII). Detailed discussion of these and other systems, and their respective advantages and disadvantages are included in Section 6. It is important that routine dosimeters be calibrated under irradiation conditions as similar as possible to those prevailing during the food irradiation process [5, 92, 112, 129–132]. Alternatively, their performance should be verified under the facility irradiation conditions. In addition, these dosimetry systems should be well characterized and traceable to a recognized standards laboratory (Section 2.4.2). Besides, it is essential that their performance be audited at regular intervals and that their performance be certified, for example through the International Dose Assurance Service (IDAS) of the IAEA [133–137] (see also Section 6.2.3).

For a radionuclide facility, when operating in a continuous mode (e.g., a shuffle–dwell system where a single process load cannot be removed independently from the facility), it is recommended that there be always at least one process load

TABLE VII. EXAMPLES OF ROUTINE DOSIMETRY SYSTEMS

Dosimeter	Measurement instrument	Usable dose range (Gy)
Alanine	EPR spectrometer	1-10 <sup>5</sup>
Dyed PMMA	Visible spectrophotometer	$10^2 - 10^5$
Clear PMMA	UV spectrophotometer	$10^3 - 10^5$
Cellulose acetate	Spectrophotometer	$10^4 - 4 \times 10^5$
Lithium borate, lithium fluoride	Thermoluminescence reader	$10^{-4} - 10^3$
Lithium fluoride (optical grade)	UV/Visible spectrophotometer	$10^2 - 10^6$
Radiochromic dye films, solutions, optical waveguide	Visible spectrophotometer	$1-10^5$
Ceric-cerous sulphate solution	Potentiometer or UV spectrophotometer	$10^3 - 10^5$
Ferrous cupric sulphate solution	UV spectrophotometer	$10^3 - 5 \times 10^3$
ECB solution	Spectrophotometer, colour titration, high frequency conductivity	10–2×10 <sup>6</sup>
Amino acids	Lyoluminescence reader	$10^{-5} - 10^4$
Polymeric plastic (M centre)	Fluorescence reader	$50-5\times10^5$

containing a dosimeter set <sup>14</sup> inside the irradiation chamber. In addition, a dosimeter set should be placed on the first and the last process load of a production run. More frequent placement of dosimeter sets during a production run could result in less product rejection should some operational uncertainty or failure arise. When operating in a batch mode, including incremental dose systems where a single process load can be removed independently from the facility, it is recommended that one dosimeter set be placed on each process load. This is to minimize the loss of product in the event of a serious failure during the process. For an electron facility, there should always be one dosimeter set at the start of a production run. For long runs, dosimeter sets should also be placed near the middle of the run and at the end of the run, and at other intervals as appropriate [75, 138].

These dosimeter sets should be placed either within or on the process load at the location of minimum dose or at the reference locations determined during process qualification. After the process, the dosimeters are read and the corresponding dose values determined and compared with the set values determined during process qualification.

Certain environmental effects, such as temperature and humidity, can affect the performance of dosimeters during irradiation as well as during readout, see Refs [5, 139–142] and several ASTM standards in the Bibliography. It is therefore important to store and handle the dosimeters before, during and after irradiation in a controlled environment as specified in the respective standards or in the manufacturer's instructions. In the event of a known effect (e.g., the temperature dependence of radiochromic films), the dosimeter response should be corrected accordingly, especially if the calibration irradiation was performed at a calibration facility under different irradiation conditions. For a dosimeter that is significantly temperature dependent, it should not be used under conditions of high temperature gradient or when there is the possibility of a rapid temperature variation with time, such as in the proximity of frozen or chilled food, as this would affect the dosimeter response in an uncontrolled way. At the Biogram facility of the Atomic Energy Corporation of South Africa, for example, where shelf stable meat products were irradiated at -40°C on a regular basis, a reference position for the routine dosimeters was chosen which was outside the polystyrene irradiation process load, thus thermally insulating the dosimeter from the product.

#### 5.3.3. Product control

Plant design and administrative procedures must ensure that it is impossible to mix irradiated and unirradiated food products. In a well designed irradiation facility,

<sup>&</sup>lt;sup>14</sup> In general, a set consisting of at least three dosimeters is used, and the average taken as the dose at that location.

the areas for storing unirradiated products should be physically isolated from the areas where treated products are stored or handled in order to separate the treated and the untreated products. This also simplifies the product inventory control procedures.

Incoming product should be registered and given a code number, which is then used to identify every process load at each step in its path through the irradiation facility. After irradiation, the movement of the product should continue to be recorded until it is released for despatch.

In some applications, radiation sensitive (sometimes referred to as go/no-go) indicators may be used to show that process loads have been exposed to a radiation source (see ASTM E1539 [143]). The most common ones change colour at a certain dose level. They may be attached onto each process load to assist in inventory control, which is a regular practice in the radiation sterilization of health care products. Thus, they should be used only to provide a qualitative indication of radiation exposure and may be used to distinguish irradiated process loads from unirradiated process loads. This practice does not, however, *replace* the routine product dosimetry discussed in Section 5.3.2. In addition, the colour change is not always stable after irradiation and may, in fact, be affected by light or heat. It must be emphasized that while these indicators can conveniently be used to assist in product inventory control, they must never be used to *replace* other inventory control procedures.

# **5.3.4.** Process interruption

If there is a process failure, for example due to power loss, its implication on the product must be evaluated before restarting the process. Generally in food irradiation, the radiation induced effect is additive as in the case of elimination/reduction of microorganisms and insect pests, and the process can be restarted from where it was interrupted. However, in some other processes, such as a delay of ripening/maturation, the effect of prolonged process interruption should be critically evaluated before restarting the process. If the product is irradiated at low temperatures or in the frozen state, care should be taken to maintain these conditions throughout the interruption. In addition, special attention should be paid to some process loads that may be at a critical point in their passage through the irradiation zone; this is more likely for an electron facility. In that case, it may be advisable to discard a few process loads that were around the radiation zone when the process was interrupted.

### 5.4. FOOD IRRADIATION AND HACCP

Originally developed to invigorate hygienic concepts in food production, HACCP easily adapts to radiation processing. The following seven principles of HACCP have been designed to locate and define potential hazards and specify measures for their control in radiation processing of food products [144, 145].

*Principle 1*, Conduct a hazard analysis — the analysis of possible hazard results in identifying 'the failure to achieve the desired effects of radiation processing' as the key point.

*Principle 2*, Identify the critical control points — it can be determined that the particular critical control point (CCP) in radiation processing is the dose that has been imparted. In addition, several other CCPs apply during the process but are not specific to radiation processing alone.

*Principle 3*, Establish critical limits — the critical limits, minimum and maximum acceptable doses are derived.

Principle 4, Establish monitoring requirements — a monitoring system is established to control this critical point which results in documentation of all process parameters (Section 5.3.1) as well as the dosimetry results (Section 5.3.2). In this regard a record of the calibration of the master timer of a shuffle–dwell system or tachometer readings of a continuously moving constant speed conveyor system should provide an early indication of a pending problem. Recent trends have been for quality assurance methodology, i.e. preventing problems from occurring rather than for quality control, which implies testing of the product to ensure that a problem has not already occurred. However, dosimetry should not be used solely as a system for product release. Its particular value lies in confirmation that the process parameters (e.g., irradiation time and product geometry) that were determined through process validation are correct. Routine product dosimetry is the final step in the overall control system to ensure that the correct dose has been applied.

*Principle 5*, Establish corrective actions — the corrective action is established that needs to be taken when monitoring indicates a particular CCP is moving out of control. This could typically mean recalibration of the master timer of a shuffle–dwell system.

*Principle 6*, Establish record keeping procedures — documentation pertaining to the procedures and records appropriate to these principles and their application are established. Because traceability is so critical in food irradiation control, documentation forms a key part of any dosimetry system.

*Principle 7*, Establish verification procedures — procedures are established to verify that the HACCP system is working correctly and that the established procedures are adhered to during routine and prolonged operation.

### 5.5. PRODUCT RELEASE AND CERTIFICATION

Proper facility operation and adherence to process control entail records and documentation. Such records are necessary for the purpose of auditing by a customer or of inspection by an authority. Typically, these records should include [51, 52]:

- (a) Information about the calibration and maintenance of the equipment and instrumentation used to control or measure the dose delivered to the product;
- (b) All dosimetry data for facility qualification, product absorbed dose mapping and routine product processing;
- (c) Values of all the process parameters affecting the absorbed dose in the product;
- (d) Product description and loading pattern in the process load;
- (e) Date the product was processed, the name of the operator and any special conditions of the irradiator that could affect the dose to the product (such as process interruption);
- (f) Copies of the shipping documents and the certificate of irradiation.

All this information should be filed together and be easily accessible for inspection.

Prior to release of a product, the dosimetry data and the recorded values of the process parameters should be examined to verify compliance with specifications. For each production run, the dose delivered to the product should be certified.

### 6. DOSIMETRY SYSTEMS

### 6.1. GENERAL

There are several dosimetry systems available for different applications. ASTM has developed standard procedures for many of these systems and for their use in radiation processing facilities [146] (also see the Bibliography). Several of these have now been recognized by the ISO and thus become mandatory. The processor should select a dosimetry system that is appropriate for the relevant application and which is the simplest and the most economical [147, 148]. ASTM Standard E1261 which describes the selection and calibration of dosimetry systems should also be consulted [40]. A few of the frequently used dosimetry systems are described in this section.

As discussed in Section 2.4.1, dosimetry systems may be divided into the following four basic classes in accordance with their relative quality and areas of application [40]:

- Primary standard dosimetry systems
- Reference standard dosimetry systems
- Transfer standard dosimetry systems
- Routine dosimetry systems.

Industrial radiation processing like sterilization of health care products and irradiation of foodstuffs are both highly regulated, in particular with regard to dose. Accurate dosimetry is indispensable, which must be traceable to national or international standards. It is essential that widely accepted dosimetry systems as well as acknowledged procedures for calibration and traceability be in place at the irradiation facility. Several of the services discussed in Section 6.2 help establish — through an unbroken chain of mutual intercomparisons — such traceability. Formal accreditation of standards laboratories and of quality control systems at an irradiation facility result in mutual equivalence and recognition of high dose measurements [10]. This helps to remove trade barriers in the international trade in radiation processed products.

Various dosimetry systems are briefly described in this section. In the characterization of an irradiation facility, for process control and for other routine measurements, the use of one or more of these dosimetry systems is recommended. Some dosimeters are toxic or contain poisonous chemicals, and therefore due care and appropriate precautions should be taken to avoid inhalation or ingestion of, or contact with, such chemicals. Further details about two particular dosimetry systems (Fricke and PMMA) that are very frequently used for food irradiation are given in Appendix I, where their standard procedures are covered in order to present the underlying physicochemical principles in an exemplary manner. More details about many other systems are available from monographs [5, 149] and various ASTM standards (see Bibliography). Instructions for quality checking/recalibration of spectrophotometers are given in Appendix II, as such instruments are most frequently used in dosimetry applications. This material is included to give a better understanding of the underlying principles; it is also found elsewhere [5, 150, 151].

## 6.2. CALIBRATION AND AUDIT SERVICES

Some national laboratories have facilities to provide an absorbed dose calibration service; this may include reference irradiations of routine or reference dosimeters at precise dose values. The services of two major laboratories are discussed below. There are many other calibration laboratories and the two mentioned here serve only as examples of such services. Several private and secondary standard laboratories have become accredited according to ISO requirements. By such means, traceability of dosimetry to national and international standards can be established (Fig. 8). In addition, readout systems can be compared by exchanging identical dosimeters and reading them on two instruments, one at the irradiation facility and the other at the standards laboratory. Such studies are indispensable for reliable dosimetry and process control.

## 6.2.1. The National Institute of Standards and Technology (USA)

The National Institute of Standards and Technology  $^{15}$  (NIST) [152] provides a calibration service for  $\gamma$  radiation from  $^{60}$ Co and  $^{137}$ Cs sources as well as for low and high energy electrons, in the absorbed dose range 40– $10^5$  Gy. Irradiation of thin film dosimeters are also provided using monoenergetic continuous electron beams from 50 keV to 1.5 MeV, and with pulsed electron beams from 1 to 30 MeV.

## **6.2.2.** The National Physical Laboratory (UK)

The National Physical Laboratory  $^{16}$  (NPL) [153] has three standard  $^{60}$ Co  $\gamma$  ray calibration sources which are used to give the absorbed doses in water at dose rates ranging from 2 to 250 Gy/min. It also provides calibrations of high dose dosimeters in 10 MeV electron beams from a linear accelerator. Mailed transfer dosimetry services are provided using alanine and dichromate dosimeters. Alanine EPR dosimetry operates over the dose range from 0.02 to 70 kGy, and dichromate dosimetry over the dose range from 2 to 55 kGy.

#### **6.2.3.** The International Dose Assurance Service of the IAEA

Although a few national standards laboratories provide calibration services to radiation processing facilities in their own countries, the increasing international trade in irradiated products has emphasized the need for reliable dosimetry control [136]. Recognizing this, the IAEA<sup>17</sup> offers a high dose audit service with the following aims [133–137, 154, 155]:

- (a) To promote dosimetric accuracy in products processed in the irradiation facilities of IAEA Member States,
- (b) To provide regulatory health authorities concerned with the trade in irradiated products with the confidence that such products have been irradiated to the specified absorbed dose.

<sup>&</sup>lt;sup>15</sup> Ionizing Radiation Division, National Institute of Standards and Technology, Gaithersburg, MD, 20899, USA.

<sup>&</sup>lt;sup>16</sup> National Physical Laboratory, Queens Road, Teddington, Middlesex, TW11 0LW, UK.

<sup>&</sup>lt;sup>17</sup> Dosimetry and Medical Radiation Physics Section, International Atomic Energy Agency, Wagramer Strasse 5, A-1400 Vienna, Austria.

The IAEA began this formal high dose assurance service in 1985 for radiation processing plants worldwide. It provides an independent check on the entire dose measurement system of the participating facility; for example, the dosimeters, measuring equipment, procedure for the use of the system, any computer software being used and the skill of the technical staff. The objective of IDAS is fulfilled by provision of transfer standard dosimeters to participating facilities. These are then irradiated by the facility operator along with its dosimeters under similar conditions and then returned to the IAEA. The dosimeter response is then analysed, the relative deviation of the participant's system calculated and the results conveyed to the participant. The transfer dosimetry system of the IAEA consists of alanine-ESR [156]; this system was chosen because of its consistent response, stability and wide useful dose range (100 Gy–100 kGy).

### 6.3. PRIMARY STANDARD DOSIMETRY SYSTEMS

Primary standard dosimeters are established and maintained by national standards laboratories for calibration of radiation fields. The two most commonly used primary standard dosimeters are ionization chambers and calorimeters.

#### **6.3.1.** Ionization chambers

It is possible to use the quantity 'exposure' to determine absorbed dose in certain materials. This is done with ionization chambers if appropriate corrections are applied. However, this method is usually not very practicable in food processing, because ionization chambers are subject to saturation effects at the high dose rates generally present at such irradiation facilities. Therefore, ionization chambers will not be considered in this book. Most texts on dosimetry have extensive information about the use of such instruments [5, 157, 158].

## **6.3.2.** Calorimetry

The instrument that gives a reading of absorbed dose directly is the calorimeter. The principles of calorimetry in applied radiation technology are relatively simple and well covered in the literature [5, 21, 34, 40, 159–162]. It measures the total energy dissipated or the rate of energy dissipation in a material in terms of the thermal properties of the absorbing body through the observation of the temperature increase (or the rate of increase [163]) of this body. For example, water temperature increases by about 2.4°C for doses of 10 kGy under ideal adiabatic conditions. Thus calorimeters are absolute dosimeters that can be used for calibrating other dosimeters. They are especially useful for measuring the high doses encountered in food

processing, in particular, with electron beams. Calorimetry has been used successfully in industrial electron beam processing as a calibration method as well as for documentation of key electron beam processing parameters related to administered doses [162, 164].

The practice of using calorimetry in a routine application, however, is usually not simple, and has not been used widely especially for  $\gamma$  irradiation. Nevertheless, there is some merit in using calorimetry with electron beams, because it is the most direct method of measuring the absorbed dose or the absorbed dose rate in different materials (e.g., water, graphite, polystyrene). In addition, with some practical designs, it is becoming a convenient routine method both for day-to-day monitoring of the high energy scanned electron beam characteristics used for processing and for calibrating routine dosimeters in electron beams of interest [162, 164, 165].

The most useful materials for an absorbing body in a calorimeter for food irradiation applications are water, graphite, aluminium and water or tissue equivalent plastics, for example polystyrene. The thermal changes are determined by the presence of small calibrated thermocouples, or thermistors, embedded in the absorbing body. If the heat capacity of the absorbing body and its functional dependence on temperature are known, calorimeters do not need to be calibrated. Alternatively, they are calibrated by measuring the temperature rise due to a known amount of energy that is supplied directly to the absorbing body in the form of ohmic heat resulting from the passage of an electric current through a resistor embedded in or surrounding the absorbing body. Ideally, it is preferable if the calorimeter is under adiabatic conditions; however, there is always some heat loss. A fairly simple correction for this heat loss is possible since most of these heat losses prove to be linear with time over the limited time interval of interest [5, 159, 160]. Other corrections may be necessary, such as that for interference caused by the presence of extraneous materials (glass containers, metal wires, etc.), but these are relatively minor corrections and are usually found to be negligible with high energy electron beams.

#### 6.4. REFERENCE STANDARD DOSIMETRY SYSTEMS

Reference standard dosimeters are used to calibrate radiation fields and routine dosimeters. Reference standard dosimeters may also be used as routine dosimeters. Reference dosimetry systems that are at present acceptable for high dose applications are the ferrous sulphate (Fricke), dichromate, ceric-cerous, ECB and the alanine-EPR. Sometimes calorimeters are also used as reference dosimeters. For food irradiation, Fricke and dichromate dosimeters are most frequently used as reference measurement systems for calibrating radiation fields and routine dosimetry systems. Table VIII lists some of the properties of these systems, some of which are commercially available. These systems will now be briefly described.

TABLE VIII. EXAMPLES OF REFERENCE STANDARD DOSIMETRY SYSTEMS

Dosimeter	Measurement instrument	Usable dose range (Gy)
Calorimeter	Thermometer	10 <sup>2</sup> -10 <sup>5</sup>
Alanine	EPR spectrometer	$1-10^5$
Ceric-cerous sulphate solution	UV spectrophotometer or electrochemical potentiometer	$5 \times 10^2 - 5 \times 10^4$
ECB solution	Spectrophotometer, colour titration, high frequency conductivity	10–2×10 <sup>6</sup>
Ferrous sulphate solution	UV spectrophotometer	$20-4\times10^2$
Dichromate solution	UV/visible spectrophotometer	$2 \times 10^3 - 5 \times 10^4$

# **6.4.1.** Ferrous sulphate (Fricke) dosimetry

The Fricke dosimetry system provides a reliable means of measurement of absorbed doses in water, based on a process of oxidation of ferrous ions to ferric ions in acidic aqueous solution by ionizing radiation [166]. The change in absorbance of the solution is measured by use of a spectrophotometer at about 303 nm. It is generally accepted as a reference dosimetry system for in-house calibration of routine dosimetry systems, see Refs [166, 167] and ASTM Standard E1026 [168]. The calibration of Fricke dosimeters should be traceable to and consistent with the national standards laboratory through the use of transfer standard dosimeters [5, 169]. In situations not requiring traceability to national standards, this system can be used for absolute determination of absorbed doses, as the radiation chemical yield of ferric ions from ferrous ions by oxidation is well known (Appendix I).

The Fricke dosimetric solution response is highly sensitive to impurities, particularly organic impurities, and to traces of metal ions. It is also sensitive to higher dose rates in accelerator facilities and appropriate corrections for its response must be applied. The temperature during irradiation should be within the range 10–60°C. Its useful response is limited to doses from 20 to 400 Gy. Appendix I gives details of the precise formulation and handling of the Fricke dosimetry system for food irradiation applications and how the optical and chemical properties are related to absorbed dose. The main areas in which caution needs to be exercised in the use of this reference dosimetry system are:

(a) Follow precisely the procedural guidelines given in Appendix I,

(b) Make fairly frequent checks of the equipment and solution to detect any detrimental changes in the system with long term use.

## 6.4.2. Alanine-EPR dosimetry

This dosimetry system provides a reliable means of measuring the absorbed dose based on the generation of specific stable radicals in crystalline alanine by ionizing radiation. The energy from the radiation is stored in these free radicals, and their concentration can be measured by an EPR spectrometer. This measurement procedure is non-destructive, thus the alanine dosimeters can be read out repeatedly. This dosimeter is used in the form of tablets, small rods or ropes of 3–5 mm diameter and various lengths, consisting primarily of  $\alpha$ -alanine and a small amount of paraffin or other binder material. The applicable dose range is 1–10<sup>5</sup> Gy. Recently, dedicated spectrometers which require less training for operation have become available. IDAS of the IAEA uses alanine-EPR as a transfer dosimetry system [156]. This system is commercially available.

For more information on this dosimetry system, see Refs [170–174] and ASTM Standard E1607 [175].

## **6.4.3.** Ceric-cerous sulphate dosimetry

This dosimetry system has long been recognized as a high dose measurement system. Radiation leads to the reduction of ceric ions to cerous ions. Doses in the range  $0.5-50 \, \mathrm{kGy}$  may be determined by conventional spectrophotometric analysis in the ultraviolet region, or by measuring the difference in the electrochemical potential between the irradiated and unirradiated solutions in an electrochemical potentiometer [176, 177]. However, extreme purity of chemical constituents and absolute cleanliness of all apparatus are essential for reproducible dosimetry, see Refs [178–180] and ASTM Standard E1205 [181]. Solutions of cerous sulphate (i.e. cerous ions), when added to the ceric sulphate dosimeter solution, have been reported to remove the effect of trace impurities [182].

It is also suitable as transfer and routine dosimeter and has reasonable food equivalence as long as it is not used in radiation fields which have a large component of low energy photons. This system is commercially available.

For more information on this dosimetry system, see ASTM Standard E1205 [181].

## **6.4.4.** Dichromate dosimetry

This dosimetry system provides a reliable means of measuring doses in water, on the basis of a process of reduction of dichromate ions to chromic ions in acidic aqueous solution by ionizing radiation. It contains potassium dichromate plus silver dichromate in aqueous perchloric acid. The applicable dose range is 2–50 kGy. It can be used for the lower dose range from 2 to 10 kGy when only silver dichromate is used. The response of this dosimeter can be enhanced further by adding acetic acid and increasing the perchloric acid concentration.

For more information on this dosimetry system, see ASTM Standard E1401 [183].

## **6.4.5.** Ethanol-chlorobenzene dosimetry

The ECB dosimetry system provides a reliable means of measuring dose based on the process of radiolytic formation of hydrochloric acid (HCl) in aqueous ethanolic solutions of chlorobenzene by ionizing radiation [184, 185]. This dosimeter comprises an aerated solution of chlorobenzene and water in ethanol to which a small quantity of acetate is added. Measurement of HCl can be achieved in one of two ways: (a) titration to determine the chloride ion concentration; (b) a high frequency measurement of change in the dielectric constant, which can be undertaken with the solution still in its sealed ampoule. The useful dose range is 10 Gy to 2 MGy [186]. This system is commercially available.

For more information on this dosimetry system, see ASTM Standard E1538 [187].

#### 6.5. TRANSFER STANDARD DOSIMETRY SYSTEMS

Transfer standard dosimeters are specially selected for transferring dose information from an accredited calibration laboratory or a national standards laboratory to an irradiation facility in order to establish traceability for that facility. These dosimeters should be sufficiently precise and stable that they can be transported for irradiation at a processing facility for dose evaluation, or for calibration of routine dosimeters. Examples of transfer dosimeters are alanine, dichromate solution, ceric-cerous sulphate solution and ethanol-chlorobenzene solution. Generally, all reference dosimeters should be good transfer dosimeters (calorimeters are an exception). Besides possessing all the desirable characteristics of reference dosimeters, transfer dosimeters should satisfy the following additional criteria (ASTM Standard E1261 [40]):

- (a) Long pre-irradiation shelf life;
- (b) Post-irradiation response stability;
- (c) Portability, i.e. the ability to withstand transport between facilities, and insensitivity to varying environmental conditions during transport.

#### 6.6. ROUTINE DOSIMETRY SYSTEMS

The main functions of routine dosimeters are different from those of reference dosimeters. Their primary function is for process control during product irradiation. They are also employed, preferably as thin dosimeters [5, 24], to measure absorbed dose distributions in process loads [36, 188, 189] and to monitor day-to-day variations in radiation fields. Although they may not be as accurate or reproducible in their radiation response as reference dosimeters, they are generally much more practicable and versatile for everyday use. Routine systems must be relatively inexpensive and easy to handle and must have a response that can be easily measured since they are generally used in much greater number and frequency than are reference dosimeters. They must be frequently calibrated against reference or transfer dosimeters, as they may not be sufficiently stable and independent from environmental or radiation field conditions and as they may show significant variations from batch to batch.

Among the complications that might interfere with effective utilization of a routine dosimetry system are dependence of dosimeter response on influence quantities such as dose rate, radiation spectrum and environmental conditions (temperature, atmosphere, humidity, light, etc.), and some other factors [42]. Such other factors include: instability before and after irradiation, impurity or chemical effects, batch-to-batch variations, size (thickness) variations within a batch, nonlinearity in the response characteristics, and readout error and imprecision. It is strongly recommended that the extent of these effects should be determined as accurately as possible before using a given dosimetry system, and that corrections or compensation be employed as necessary. One method of diminishing the effect of some of the influence quantities is to perform in-house calibration of the routine dosimetry system, with the routine dosimeters being irradiated in the production facility along with transfer dosimeters from a calibration laboratory (Section 2.4.2).

Several dosimetry systems (plastic plates, pellets or films, chemical solutions, dye systems, phosphors or glasses) are used for routine food irradiation dosimetry [5, 24, 92, 190]; a variety of these is also available commercially. Guidelines for selection of appropriate systems are available in ASTM Standard E1261 [40] and are also described in Section 2.5. Examples of routine dosimeters are listed in Table VII. Some reference dosimeters are often used as routine dosimeters, such as the ceric-cerous, ECB and alanine types. Other newer types might also be included if their response is reproducible enough to give a reasonably accurate dose interpretation in an appropriate material of interest and in a prescribed dose range.

Some of the more common routine dosimetry systems are briefly described here with respect to their proper use in food irradiation absorbed dose determinations.

## **6.6.1.** Clear PMMA dosimetry

This dosimetry system is based on the measurement of the radiation induced absorbance change in clear PMMA, having commercial names such as Perspex, Lucite and Plexiglas. Irradiation of clear PMMA induces a broad absorption band in the 250–400 nm region with maximum sensitivity between 304 and 320 nm. The recommended measurement wavelength is 314 nm [191] where effects of temperature and humidity are less pronounced. Although a special Perspex for dosimetry is available commercially, any locally made PMMA sheets having a consistent mixture, guaranteed continuing supply and suitable optical properties may be used as a dosimeter. However, every batch should be calibrated and should be traceable to and consistent with the appropriate national standards laboratory through the use of transfer standard dosimeters. Unless its properties, including its environmental effects, are fully characterized, such locally produced PMMA materials have only a limited use. A point of caution: most commercially available clear PMMA sheets contain a UV absorber, which precludes their use in dosimetry.

In addition, for more general information on this dosimetry system, see Refs [192–194] and ASTM Standard E1276 [195]. This system is commercially available.

# **6.6.2. Dyed PMMA dosimetry**

Several types of specially prepared PMMA containing certain dyes which become darker upon irradiation are available for dosimetry [196–199]. These include red Perspex 4034, amber Perspex 3042 and Gammachrome YR, and are supplied in sealed pouches. Depending on the type (colour) of the dye, the change in absorbance is measured at specific wavelengths in a spectrophotometer. General references which may be consulted are Refs [140–142, 190, 192, 200].

A detailed procedure for the use of this dosimetry system is presented in Appendix I, and more information on these dosimetry systems is given in ASTM Standard E1276 [195]. These systems are commercially available.

### 6.6.3. Radiochromic dye systems

One type of this dosimetry system is based on colourless triphenylmethane dye cyanide or dye methoxide solutions. Permanent colouration is developed when these solutions are exposed to ionizing radiation. The wavelength of the peak in the absorption band produced by radiation is dependent upon the specific dye and solvent used [190, 201–203]. The absorbance change at the analysis wavelength is related linearly (or in some cases nearly linearly) to the radiation dose, although at high doses saturation occurs in some systems [204]. The dye systems can be used in liquid or solid solutions, and the concentration of the solution or the wavelength used for the

measurement of absorbance can be chosen to cover the appropriate dose range [92, 190]. The dye systems can be made into coated or self-supporting thin films and can thus be used for electron dosimetry for accurate depth—dose distribution measurements [92, 189, 205]. These systems are commercially available.

Some of the advantageous characteristics of these dosimeters are

- —Long shelf life
- Similar response for electrons and photons
- Low atomic number and thus food product equivalence
- Small temperature dependence in the case of solid systems
- Stable response
- Linear dose response for some systems
- Low sensitivity to impurities
- Simple readout procedures.

These dosimeters, however, should be protected from UV or fluorescent light and some against humidity changes [206]; some are very sensitive to elevated temperatures [207]. The optical surfaces of solid film dosimeters should not be touched or scratched. The systems fall into two main categories: liquid dye cyanide solutions and plastic film or paper impregnated with radiochromic dye. Other, non-cyanide, dye systems incorporated into plastic films are also commercially available. These have the same advantages as the cyanide dye systems described above.

Additional information regarding the use of these dosimetry systems can be found in ASTM Standards E1275 [208], E1540 [209] and E1650 [210]. Some general information can also be found in Refs [92, 201, 204–207, 211–219].

### 6.7. OTHER SYSTEMS

Several other dosimetry systems are available; some are well developed but less suited to food processing applications, while a few others would be especially suited but are not yet well developed or commonly used in food processing applications. Some of the systems described below are also commercially available.

A new emerging system is the SUNNA fluorescence dosimeter [220, 221]. It is commercially available in large quantities and is suitable for routine applications in the dose range suitable for food irradiation. A dedicated reader has also become available. Other emerging systems include fluorescent salts on several supporting materials and semiconductors that allow on-line measurements; systems have also been proposed which can be used at very low and extremely high temperatures [222]. However, these systems have not yet been developed to the extent that allows their reliable utilization in routine applications. There are several other systems (e.g.,

lyoluminescence of crystalline food compounds such as sugar or salt) which have already been used to some extent but which could not reach the common recognition of their metrological quality required and which are, therefore, not covered by this book.

There have been several attempts to make optical readings of film dosimeters (in transmission) independent of the thickness of the film [223, 224]. This is achieved by measuring absorbance at two sufficiently different wavelengths.

## **6.7.1.** Polyvinyl chloride dosimetry

Dosimetry using polyvinyl chloride (PVC) is based on the change in absorbance at 395 nm induced by radiation. The film changes from nearly clear to green through yellow brown to dark brown depending on the dose. The useful dose range is  $5 \times 10^2$  to  $5 \times 10^4$  Gy. After irradiation, the PVC dosimeter should be developed by heating either at 50°C for 1 h or at 80°C for 5–10 min to give full colour development. The material comes in large sheets or rolls and may be cut in strips. It should not contain plasticizers or stabilizers which affect its dose response function.

PVC is not recommended for accurate dosimetry but is often used in electron irradiation facilities to determine the beam cross-section and position, scan widths and uniformity, dose uniformity in both lateral directions, and to monitor approximately that the required dose level has been reached.

## **6.7.2.** Thermoluminescence dosimetry

Certain inorganic crystals store energy from radiation in the form of trapped electrons. Upon irradiation of these crystals, electrons are knocked out of their usual lattice positions and positively charged holes remain. The electrons remain trapped elsewhere in the lattice. Upon heating, the electrons are released from the trapping sites and recombine with the holes releasing energy as light in the process. Hence, the effect is named thermoluminescence (TL). The amount of light emitted is proportional to the radiation dose. Several commercial TL readout units are available having a reproducible heating and readout cycle. The major disadvantage of most TL systems for food irradiation dosimetry is that the useful range is generally limited to doses below about 1 kGy. Dust on food contains some crystalline material which may be used for TL dosimetry [101]; this type of dosimetry is being developed for radiation hygienization of sewage sludge [225]. This effect is already being used for analytical detection of irradiated food and subsequent dose estimation.

## 6.7.3. Label dosimetry

Radiation sensitive indicators are in widespread use in the radiation processing industry (ASTM Standard E1539 [143] and Ref. [226]). Attached to the goods to be

irradiated, their particular advantage is a significant visual change when exposed to radiation. This property facilitates inventory control in an irradiation facility. However, such devices cannot serve as dosimeters: the visual change is not related in a quantitative manner to the dose absorbed.

By contrast, there are labels (that are put on the process loads) that may exhibit a measurable response to radiation which can be related to absorbed dose in a quantitative manner. Such dosimetry systems may then be called label dosimeters [227–231]. Such systems are presently not available commercially. However, a few systems that are commercially available may be considered dosimeters for a *single* dose value, indicating that a specified threshold dose was exceeded. They establish this dose value rather accurately as compared with radiation sensitive indicators. Such dosimeters are in use for low dose applications, such as insect disinfestation.

### Appendix I

### DOSIMETRY PROCEDURES: FRICKE AND PMMA

#### I.1. INTRODUCTION

Standard procedures are presented in this Appendix for two important dosimetry systems. The choice of the Fricke and of the PMMA systems here is arbitrary. As examples, their description should contribute to a better understanding of the underlying physicochemical principles and recommended practices which have resulted from experience in practical utilization of these systems. All equations are developed exclusively in SI units; for conversion to historical units the earlier manual may be consulted [4].

Several components of the dosimetry systems are toxic or poisonous chemicals and therefore due care and appropriate precautions should be taken to avoid inhalation or ingestion and contact with such chemicals. Each of the steps mentioned in any of the procedures is of the utmost importance, no matter how trivial it may appear to be, and should be strictly adhered to for optimum and meaningful use of the system.

For more details the reader is referred to pertinent monographs [5, 149], and ASTM Standards E1026 [168] and E1276 [195]. In addition, detailed procedures for quality checking or recalibration of spectrophotometers can be found in Appendix II, as such instruments are most frequently used in dosimetry applications, including for Fricke and PMMA systems.

### I.2. FERROUS SULPHATE (FRICKE) DOSIMETRY

The Fricke dosimetry system provides a reliable means of measurement of absorbed dose and is a generally accepted reference dosimetry system for in-house calibration of routine dosimetry systems at processing facilities [168]. However, the calibration of the Fricke system should be traceable to and consistent with the national standards laboratory through the use of transfer standard dosimeters. In situations not requiring traceability to national standards, this system can be used for absolute determination of absorbed doses, as the radiation chemical yield of ferric ions from ferrous ions by oxidation is well known. The change in the absorbance of the solution is measured by the use of a spectrophotometer at about 303 nm.

The Fricke dosimetric solution response is highly sensitive to impurities, particularly organic impurities and traces of metal ions; it is also sensitive to higher dose rates in accelerator facilities. Its useful response is limited to doses from 20 to

400 Gy. The standard procedure for the Fricke dosimetry system is briefly described below.

General references that may be consulted for Fricke dosimetry are Refs [166, 167, 169, 178] and ASTM Standard E1026 [168].

### **I.2.1.** Procedure for the use of the system

The ferrous sulphate dosimeter<sup>18</sup>, usually called the Fricke dosimeter, is based on the chemical process of oxidation of ferrous ions (Fe<sup>2+</sup>) in aqueous sulphuric acid solution to ferric ions (Fe<sup>3+</sup>) by ionizing radiation. The method can be used for accurate absorbed dose determination by measuring the change in the absorbance of an irradiated dosimetric solution in a temperature controlled spectrophotometer at 303 nm wavelength (the peak of the absorption spectrum). Since absorbance readings at a given wavelength may vary from one spectrophotometer to another, the molar linear absorption coefficient should be determined for each spectrophotometer at the same wavelength and slit width that are used for the Fricke dosimetry measurements. Since the change in absorbance is linearly proportional to the absorbed dose in the useful range, the dose can be determined by multiplying the change in absorbance by a suitable conversion factor.

The accuracy of the measurement of the dose is not affected for dose rates below  $10^6$  Gy/s [178]. To minimize the effect of dose rate at levels somewhat above this value the dosimetric solution described below should be modified by excluding NaCl and increasing the ferrous ammonium sulphate concentration to 0.01 M (Section I.2.2) [232]. The reliability of the method is not significantly influenced by the temperature of the system between 1 and  $60^{\circ}$ C during irradiation. <sup>19</sup> The response of the system is nearly independent of the spectral energy of the radiation in the range from 0.5 to 16 MeV [166].

A spectrophotometer with matched quartz cells (cuvettes) of 1 cm optical path length, equipped with a temperature controlled cell compartment, should be used for optical absorbance measurements. Chemically resistant borosilicate glass or its equivalent should be used to hold the solution during irradiation (e.g., Kimball 'quick break' blue line ampoules of Pyrex). Polyethylene containers may also be used if they are well cleaned. Dosimetry containers and other glassware may be cleaned by filling them with ferrous ammonium sulphate solution and then irradiating them with at least 500 Gy. When a container is needed, the irradiated solution should be poured out,

<sup>&</sup>lt;sup>18</sup> In fact, in this procedure, the iron salt used for making the Fricke dosimeter is ferrous ammonium sulphate because, in general, it results in smaller errors than does ferrous sulphate.

<sup>&</sup>lt;sup>19</sup> The ferric ion yield depends slightly on the irradiation temperature. For more details, see Section I.2.5.

the container rinsed at least three times with unirradiated solution and then refilled with the dosimetric solution for irradiation. When not in use, polyethylene containers should be stored containing dosimetry solution. Reagents should be of analytical grade. The use of triply distilled water from all-glass or silica stills is recommended. Deionized water or distilled water stored in plastic containers should not be used for Fricke systems.

## I.2.2. Preparation of the dosimetry solution

Following Ref. [168] dissolve 0.392 g of ferrous ammonium sulphate (Fe(NH<sub>4</sub>)<sub>2</sub>(SO<sub>4</sub>)<sub>2</sub> · 6H<sub>2</sub>O) and 0.058 g of sodium chloride (NaCl) in 12.5 mL of 0.4 mol/L sulphuric acid (H<sub>2</sub>SO<sub>4</sub>).<sup>20</sup> Dilute to 1 L in a volumetric flask<sup>21</sup> with 0.4 mol/L H<sub>2</sub>SO<sub>4</sub> at 25°C (the solution concentrations are 0.001 M ferrous ammonium sulphate, 0.001 M sodium chloride and 0.4 mol/L sulphuric acid). This solution is not very stable, but it can be stored in a clean, dark brown, stoppered bottle at 15–20°C for up to eight weeks. A marked increase in the absorbance of the non-irradiated solution at a wavelength of 303 nm indicates that the solution is no longer reliable.

## I.2.3. Determination of the molar linear absorption coefficient, $\varepsilon$

The molar linear absorption coefficient (also referred to as the molar extinction coefficient) should be measured for each spectrophotometer at 25°C as this value varies from one instrument to another. In doing so, the performance of the spectrophotometer is independently verified. For determining the molar extinction coefficient, the absorbance of solutions of different known concentrations of ferric ions is measured. A curve (it is generally linear) of absorbance versus ferric ion concentration is obtained from which the molar extinction coefficient is calculated.

Prepare a concentrated ferric ion solution as follows:

- (a) Weigh 100 mg of spectrographically pure (purity of at least 99.99%) iron wire to the nearest 0.1 mg.
- (b) Place this in a long necked 1000 mL calibrated volumetric flask.
- (c) Add 60 mL of distilled water and 22.5 mL of concentrated sulphuric acid (density 1.84 g/cm<sup>3</sup>).

<sup>&</sup>lt;sup>20</sup> The 0.4 mol/L sulphuric acid is made by dissolving 22.5 mL of concentrated sulphuric acid (density 1.84 g/cm<sup>3</sup>) in distilled water to make 1 L of solution in a volumetric flask (care should be taken: add the acid little by little to the water; *never* add water to acid).

<sup>&</sup>lt;sup>21</sup> The calibration temperature of volumetric flasks is normally 25°C; check this on the flask being used.

- (d) Heat the unstoppered flask gently under a fume hood until the wire is completely dissolved. With a beaker inverted over the mouth of the flask, allow the solution to cool. During heating, cooling and refluxing, the neck of the flask should be maintained at an angle of 45° to the horizontal to prevent loss of solution.
- (e) Add 3–5 mL of 35% hydrogen peroxide solution to the flask and boil under reflux condensation for 0.5–1 h, or until the bubbles of excess peroxide are driven off. Allow the flask to cool and place it in a thermostat at 25°C.
- (f) Dilute the solution with distilled water to give 1000 mL of solution. This reference solution has an absorbance of about 4 at a wavelength of 303 nm.

The molarity (mol/L) of the reference solution may be calculated as follows:

$$c_{\text{Fe}^{3+},\text{ master}} = \left(\frac{m_{\text{Fe}^{3+}}}{V}\right)k \tag{6}$$

where

 $m_{\rm Fe^{3+}}$  is the mass (kg) of the iron dissolved in step (a),

V is the volume (L) of the final solution used in step (f),

k is the conversion factor; for Fe<sup>3+</sup> ions, k = 17.91 mol/kg (=1 mol/0.056 kg) (note: 0.056 kg = 56 g  $\approx$  gram atomic weight of iron).

Transfer 1, 5, 10, 15, 20 and 25 mL of the reference solution into six 100 mL volumetric flasks and dilute each portion to the 100 mL mark by adding 0.4 mol/L sulphuric acid. Measure the optical absorbance of these diluted solutions using 1 cm path length quartz cells (cuvettes) in a temperature controlled (25°C) spectrophotometer at the absorption peak (303 nm wavelength). Use a 1 cm path length quartz cell containing distilled water as a reference blank. Plot the absorbance of the diluted solution (on the ordinate, i.e. the y axis) against the molarity of the ferric ion solution (on the abscissa, i.e. the x axis). The molarity,  $c_{\text{Fe}^{3+},\text{dil}}$ , of the several diluted solutions can be calculated from:

$$c_{\text{Fe}^{3+},\text{dil}} = \frac{c_{\text{Fe}^{3+},\text{master}}}{s} = \left(\frac{m_{\text{Fe}^{3+}}}{V}\right) \frac{k}{s}$$
 (7)

where the meanings of  $m_{\rm Fe^{3+}}$ , V and k are as in Eq. (6) and s is the dilution factor, which is the final volume (100 mL) divided by the initial volumes (1, 5, 10, 15, 20 or 25 mL) taken from the reference solution.

The slope at any point on the curve is the molar extinction coefficient,  $\varepsilon$ , as determined by the particular spectrophotometer at the particular absorbance level for the optical path length used. In a good optical system the 'curve' should be a straight

line, and the molar extinction coefficient should be  $\varepsilon = 219 \text{ m}^2/\text{mol}$  over the entire absorbance range at 25°C [168]. The value of the molar extinction coefficient varies with temperature and has a positive temperature coefficient. If the measurements are undertaken at a temperature  $t(^{\circ}\text{C})$ , the value of the molar linear absorption coefficient at 25°C can be calculated using the formula:

$$\varepsilon_{\text{Fe}^{3+},25^{\circ}\text{C}} = \frac{\varepsilon_{\text{Fe}^{3+},t^{\circ}\text{C}}}{1 + \xi(t - 25)} \text{ for } 15^{\circ}\text{C} < t < 35^{\circ}\text{C}$$
 (8)

where  $\varepsilon_{Fe^{3+},25^{\circ}C}$  and  $\varepsilon_{Fe^{3+},\,\it{t^{\circ}C}}$  are the values of the molar linear absorption coefficient at 25 and  $\it{t(^{\circ}C)}$ , respectively, and  $\xi$  is the temperature coefficient of  $\varepsilon_{Fe^{3+},25^{\circ}C}$ , which is 0.7% per Kelvin (i.e.  $\xi$  = 0.007 K<sup>-1</sup>).

# I.2.4. Dosimetry procedure

Fill clean dosimetry containers  $^{22}$  (cleaned as described in Section I.2.1) with a freshly prepared dosimetry solution and place the containers in the radiation field for a carefully measured period of time. If used in a  $\gamma$  ray field, care should be taken that conditions of electron equilibrium (Section 2.3.3) prevail and that the position of irradiation is accurately reproducible. After irradiation, read the absorbance,  $A_i$ , of the irradiated solution in the spectrophotometer at the absorbance peak (about 303 nm). Either use a non-irradiated solution as a reference blank in the spectrophotometer or measure the absorbance of the non-irradiated solution,  $A_0$ , using an air path as the 100% transmission reference. The equation for calculating the absorbed dose in the Fricke dosimetric solution is

$$D = \frac{(\Delta A)N_{A}}{\rho G \varepsilon d} \tag{9}$$

where

D is the absorbed dose (Gy),

 $\Delta A$  is the change in absorbance at 303 nm and 25°C (dimensionless),  $\Delta A = A_i - A_0$ , where  $A_i$  and  $A_0$  are the absorbances of the irradiated and non-irradiated solutions, respectively,

 $N_{\rm A}$  is Avogadro's number (6.022 × 10<sup>23</sup> mol<sup>-1</sup>),

 $\rho$  is the density of the dosimetric solution (1.024 × 10<sup>3</sup> kg/m<sup>3</sup>),

 $<sup>^{22}\,</sup>$  Typically, a container is an ampoule of volume 2–5 mL with an outer diameter of 12–15 mm.

- G is the radiation chemical yield of Fe<sup>3+</sup> ions (9.74 × 10<sup>17</sup> molecules/J) (This G value is valid for electrons or photons in the energy range 0.5–16 MeV at absorbed dose rates of less than  $2 \times 10^7$  Gy/s.),
- ε is the molar linear absorption coefficient (at 303 nm and 25°C) as measured for the particular spectrophotometer (with a nominal value of 219 m²/mol), and
- d is the optical path length in quartz cells, usually d = 0.01 m.

For irradiation and absorption measurement temperature of 25°C, with a 1 cm path length cuvette, and using the values of  $\varepsilon$  and G given above, Eq. (9) reduces to

$$D_{\text{Fricke}} (\text{Gy}) = 278 \,\Delta A. \tag{10}$$

# I.2.5. Temperature correction

If the optical absorbance measurements are not carried out at 25°C, a correction must be applied to the measured values. As discussed in Section I.2.3, the temperature coefficient of the molar extinction coefficient at 25°C has the value

$$\xi = 0.007 \text{ K}^{-1} \text{ valid for } 15^{\circ}\text{C} < t < 35^{\circ}\text{C}$$

where  $t(^{\circ}C)$  is the actual temperature of the Fricke solution during the optical observance measurement.

A further correction is needed for the effect on the G value of the irradiation temperature of the dosimetric solution. The temperature coefficient of the G value at  $25^{\circ}$ C has the value

$$\xi' = 0.0015 \text{ K}^{-1} \text{ valid for } 10^{\circ}\text{C} < t' < 60^{\circ}\text{C}$$

where t' (°C) is the solution temperature during irradiation. Consequently, Eq. (10) is modified to read

$$D_{\text{Fricke}}(\text{Gy}) = \frac{278\Delta A}{[1+0.007(t-25)][1+0.0015(t'-25)]}$$
(11)

#### I.2.6. Precision

A precision of about 1% can be expected with this system if appropriate precautions, especially regarding cleanliness and purity, are followed in the handling of the solutions, their ingredients and the containers.

#### I.3. AMBER PMMA DOSIMETRY

Under the influence of ionizing radiation, chemical reactions take place in PMMA creating and/or enhancing absorption bands in the visible region of the spectrum. The absorbance is determined at two different wavelengths, 603 and/or 651 nm, depending on the prevailing irradiation conditions [140]. Every batch of dosimeters should be calibrated and should be traceable to and consistent with the appropriate national standards laboratory through the use of transfer standard dosimeters.

Unprotected PMMA dosimeters are sensitive to changes in humidity, and the dosimeters are therefore individually sealed in water impermeable pouches at the manufacturing stage. For example, amber 3042 dosimeters are supplied by the manufacturer in such pouches. They must be kept in these sealed pouches during irradiation and removed only for the spectrophotometer analysis.

In the general literature Refs [140, 233, 234] may be consulted. For more specific information on this dosimetry system, see ASTM Standard E1276 [195].

#### I.3.1. Dosimeter care

In order to obtain reproducible results with this dosimetry system, the procedure given below for the handling of the dosimeter should be followed:

- (a) The dosimeter should be kept in the sealed pouch during irradiation and until the time of analysis.
- (b) After irradiation, each pouch should be inspected for any imperfections.
- (c) The dosimeter should be handled by its edges only.
- (d) The dosimeter should be inspected for any imperfections, such as scratches.
- (e) If necessary, the dosimeter should be cleaned before analysis; an accepted method is wiping with paper tissue moistened with ethyl alcohol.

# **I.3.2.** Dosimetry procedure

For  $\gamma$  irradiation, the dosimeter should be surrounded with some plastic material to achieve electron equilibrium conditions (Section 2.3.3). After irradiation and before measuring the absorbance, the spectrophotometer should be set at the selected wavelength (603 or 651 nm) with a slit width of 0.4 mm. The null point setting (dark current) is adjusted and the reference (air path) is set to 100% transmission; these two steps should be repeated until no further change occurs. The dosimeter is then placed vertically in the holder, and the absorbance value A determined.

If and when no further absorbance measurement is to be made on the dosimeter, it is removed from the holder and its thickness, d, is determined in the region

traversed by the analysing light beam using a calibrated thickness gauge (e.g. a micrometer). The radiation induced specific absorbance, k, is calculated as k = A/d, where d is the thickness of an individual dosimeter. The dose can then be calculated from the specific absorption value using the calibration relationship for the dosimetry system.

The information from the supplier should be consulted regarding the applicability of this system for ranges of various parameters, such as dose, dose rate and temperature.

## I.3.3. Precision

PMMA dosimeters are easy-to-handle, approximately food equivalent, dosimeters covering the range of doses typically used in food irradiation. With proper use, measurements can be made with a precision of better than 1.5%.

## Appendix II

### CALIBRATION OF SPECTROPHOTOMETERS

## II.1. INTRODUCTION

Most of the dosimeters mentioned in this book have radiation induced effects that are measured with a spectrophotometer. Consequently the following information is given to enable the user to check that the spectrophotometer is in good working condition.<sup>23</sup> Modern spectrophotometers have some self-calibration features; however, these need to be cross-checked by physical methods. Such services may be available from national metrological laboratories if the spectrophotometer has been designed for such calibration services. Such certifications include wavelength as well as absorbance scale.

In the general literature Refs [235–238] may be consulted, as well as ASTM Standards E925 [239] and E958 [240].

#### II.2. WAVELENGTH CHECK

A small low pressure mercury vapour lamp<sup>24</sup> may be used to check the wavelength scale in the UV and visible region of the spectrum. The mercury lines are very well defined and a lamp, such as the Penray Type II SC-1<sup>25</sup> can be inserted without removing the normal lamp, and the check made as described below. Two alternatives are available in the visible part of the spectrum:

- (a) Lamps already provided with a spectrophotometer (e.g., mercury, hydrogen or deuterium lamps) may be used.
- (b) Special optical filters (e.g., holmium oxide or didymium glass filters or Fabry–Perot interference filters) may be used.<sup>26</sup>

<sup>&</sup>lt;sup>23</sup> Spectrophotometers are components of dosimetry systems and thus their performance affects the quality of dose measurements.

<sup>&</sup>lt;sup>24</sup> For better efficiency, spectrophotometer lamps are usually high pressure mercury, hydrogen or deuterium lamps. Except for the red end of their emission, spectrum lines are purposefully broadened and smeared over the gaps between lines; consequently, such lamps are not as useful for wavelength calibration as a low pressure mercury lamp.

<sup>&</sup>lt;sup>25</sup> Such lamps are available as accessories from several suppliers of spectrophotometers.

<sup>&</sup>lt;sup>26</sup> Such filters are available as accessories from several suppliers of spectrophotometers.

The mercury lines are at the following wavelengths: 253.7, 296.5, 302.2, 312.6, 313.2, 365.0, 365.5, 404.7, 453.3, 546.1 and 577.0 nm. The two lines at 312.6 and 313.2 nm may not always be resolved but can be used to indicate how well the spectrophotometer can resolve wavelength differences. As many dosimeters are read in the UV range, calibration of the wavelength scale in this range is generally recommended. Hydrogen or deuterium lamps have two useful lines, one at 486.0 nm and the other at 656.1 nm.

## II.2.1. Procedure for calibrating wavelength control

- (a) Adjust the wavelength to 253.7 nm (the first mercury line).
- (b) Adjust the 0% (zero per cent) and 100% transmission controls with an empty compartment.
- (c) Switch off the normal light sources and remove the cover from the lamp housing.
- (d) Clean the bulb of the low pressure mercury vapour lamp with a tissue dampened with alcohol. Do not touch the bulb.
- (e) Set the low pressure mercury lamp in the light path between the lamp mirror and the entrance slit of the monochromator.
- (f) Close the slit and switch on the mercury vapour lamp. Allow a warm-up period of at least 5 min.
- (g) Open the slit slowly until a small reading of transmission/absorbance (optical density) is obtained.
- (h) Adjust the position of the lamp slowly until there is maximum deflection of the transmission/absorbance indicator. Then lock the lamp in that position.
- (i) If necessary, keep the maximum absorbance reading on-scale by decreasing the slit width, using the slit-width control.
- (j) Adjust the wavelength control until the maximum deflection of the transmission/absorbance indicator is obtained, keeping the maximum deflection on-scale using the slit-width control.
- (k) Note the wavelength scale reading at maximum deflection. This reading should not differ from the nominal value for this line by more than the value quoted in the specification manual of the spectrophotometer as a normal tolerance.
- (l) Adjust the wavelength control to the setting for the next mercury line and repeat steps (j), (k) and (l) for all other mercury lines.
- (m) Repeat the whole procedure using the hydrogen or deuterium lamp for the lines at 486.0 and 656.1 nm.

Note: If the wavelength control does not agree with the nominal mercury line values, a re-calibration of the wavelength control can be made for this region with a graph of wavelength control versus nominal values. Alternatively, the spectrophotometer supplier can be asked to readjust the wavelength control.

## II.2.2. Wavelength check using glass filters

- (a) Select wavelength at the first identified peak in the filter absorption spectrum.
- (b) Set 0% transmission with slit closed.
- (c) Open slit to obtain a small absorbance reading and adjust 100% transmission with an air path.
- (d) Insert glass filter.
- (e) Set the wavelength to the position of the nominal absorption peak and take readings at wavelengths slightly above and below this setting. Determine the wavelength setting on the instrument that gives the maximum or minimum absorbance reading. Then compare this with the calibration spectrum.
- (f) Repeat step (e) for other identified maxima and minima in the calibrated spectrum of the respective filter.

## II.3. ABSORBANCE (OPTICAL DENSITY) SCALE CHECK

A solution of potassium dichromate can serve as a standard to check the absorbance scale at various wavelengths [236, 237]. Certified reference materials (liquid or solid) for this purpose are also available from NIST and NPL.

The solution of potassium dichromate in dilute sulphuric acid exhibits two maxima and two minima in the absorption spectrum. A standard solution of appropriate concentration gives an indication of the absorbance scale calibration for a given instrument. A series of further dilutions from a master solution can, correspondingly, be used to give a range of absorbance levels. It should be noted that spectrophotometers with a fixed slit width (or when operated with too wide a slit width) will give a slightly lower reading in the maxima and a slightly higher reading in the minima. The calibration procedure is set out below.

### **II.3.1.** Preparation of the solution

- (a) Dilute 10 mL of clear concentrated sulphuric acid (H<sub>2</sub>SO<sub>4</sub>, analytical grade) in 1000 mL of distilled water. (Care should be taken: add the acid little by little to the water: *never* add water to acid.)
- (b) Pour about 500 mL of distilled water into a clean calibrated volumetric 1000 mL flask.
- (c) Mix 35 mL of the  $\rm H_2SO_4$  solution obtained in step (a) with the 500 mL of water in the flask.
- (d) Dissolve 55 mg of potassium dichromate (analytical grade) in the solution in the flask. Any amount of potassium dichromate between 50 and 60 mg may be used but the weight must be known accurately to 0.1 mg.

(e) Fill the flask to just under the calibration mark with distilled water. Allow the solution to stabilize at a particular temperature, or place in a thermostat set to the measurement temperature. Then fill the flask to the calibration mark with distilled water. After thorough mixing, the solution is ready to be used.

#### II.3.2. Calculation of absorbance

The absorbance, A, of the solution (with the aqueous sulphuric acid solution as a blank) can be calculated using the formula

$$A = acd (12)$$

where

- a is the extinction coefficient ( $L \cdot g^{-1} \cdot cm^{-1}$ ), see Table IX,
- c is the concentration of potassium dichromate in the sulphuric acid solution (g/L),
- d is the path length of the cuvette used (cm).

The potassium dichromate solution has two maxima and two minima, as given in Table IX. The appropriate extinction coefficients and the resulting absorbances for a dilution of 55 mg potassium dichromate in 1000 mL of solution are also given.

#### II.3.3. Measurements

- (a) Adjust the wavelength control to 235 nm.
- (b) Adjust 0% transmission with source slit in closed position.
- (c) Adjust 100% transmission scale reading with a small slit opening and with distilled water in a cuvette in the light beam.
- (d) Repeat steps (b) and (c) until no further change is observed.
- (e) Move the potassium dichromate solution into the light beam and note the indicated absorbance value.

TABLE IX. POTASSIUM DICHROMATE ABSORBANCE SCALE STANDARD

Wavelength (nm)	Maximum or minimum	Extinction coefficient $(L \cdot g^{-1} \cdot cm^{-1})$	Resulting absorbance for 0.055 g/L K <sub>2</sub> Cr <sub>2</sub> O <sub>7</sub> solution
235	Minimum	12.5	0.687
257	Maximum	14.5	0.797
313	Minimum	4.9	0.270
350	Maximum	10.7	0.588

- (f) Adjust the wavelength control to 4 nm above the calibration value mentioned in step (a).
- (g) Repeat steps (b)–(e).
- (h) Adjust wavelength control to 4 nm below the calibration value mentioned in step (a).
- (i) Repeat steps (b)–(e).
- (j) Of the three absorbance values noted, the first one should be the lowest. If this is so, proceed further. If not, then repeat the wavelength calibration procedure using the mercury lamp.
- (k) Adjust the wavelength reading in step (a) to 313 nm and repeat steps (a)–(j).
- (l) Adjust the wavelength reading in step (a) to 257 nm and repeat steps (a)–(i).
- (m) The first of the three absorbance readings in this wavelength region should be a maximum. If it is not, check the wavelength calibration.
- (n) Adjust the wavelength reading in step (a) to 350 nm and repeat steps (a)–(i) and (m).
- (o) Check that the value of the absorbance measured at each of the four nominal wavelengths agrees to within 2% with the calculated value determined from your own, known, solution strength. If the agreement is within 2%, the wavelength and absorbance scales of the spectrophotometer are in good working order over this wavelength range.
- (p) If some of the points are within 2% of the calculated values and some are not, or if all four values are 'off' by more than 2%, the curve of measured absorbance versus calculated absorbance will indicate whether the absorbance scale is nonlinear or is linear but shifted. If the scale is non-linear, repeat the whole measurement procedure, checking carefully the 0 and 100% transmission settings. If the results are the same and the scale remains non-linear, the curve of measured values versus calculated values can be used to correct experimentally determined absorbance values. If the curve is linear but shifted, then check the wavelength calibration curve and check that the real minima at wavelengths of 235 and 313 nm and the real maxima at 257 and 350 nm occur at these wavelength settings. If so, repeat the procedure and use the curve obtained to correct the experimental absorbance readings. If a discrepancy is found, the supplier of the instrument should be asked to service it to bring it up to specification. After the instrument has been serviced, the complete calibration procedure should be repeated.

## II.3.4. Absorbance check using filters

Glass filters may also be used for checking the absorbance scale. The same glass filters as used for checking the wavelength scale may also be calibrated for absorbance. See footnote 26 (p. 105) for availability.

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#### Annex I

#### PRACTICAL IRRADIATOR DESIGNS

In this Annex several practical irradiators are described including some of their salient design features. A few examples are given of the facilities that are currently in operation. Even though there is no discussion of the dosimetry requirements here, this Annex is included to give examples of the variety of irradiators and how this could influence the demands placed on dosimetry.

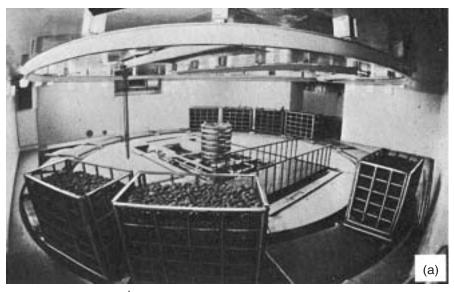
## I-1. RADIONUCLIDE IRRADIATORS

## I-1.1. Pallet irradiators

In this type of irradiator, transportation of food is usually on pallets to minimize labour requirements; there is an incentive to irradiate the products on the same pallets on which they are transported to the irradiation facility. This places special demands on the irradiator designer in devising facilities which can economically process products on such pallets and maintain an acceptable dose uniformity ratio.

The first commercial pallet irradiators were designed to process potatoes and onions in large pallet boxes. For such products, the containers are usually bulky, having a capacity of 1 t or more in a single unit. To co-ordinate with the methods of harvesting, storage and shipment of products, it is preferred to use the same containers within the irradiation facility. Furthermore, potatoes are sensitive to skin damage, wound healing is inhibited after irradiation and accelerated rotting may occur; therefore, careful handling is indispensable for potatoes before, during and after radiation processing.

In a radionuclide facility, potatoes are irradiated in wire-net containers with inside dimensions of  $100 \text{ cm} \times 160 \text{ cm} \times 130 \text{ cm}$  and with an average capacity of about 1.5 t of potatoes [I–1 to I–6]. Such a facility has been in operation in Japan since 1974. The layout of the plant is illustrated Fig. I–1. The process loads to be irradiated are introduced at point 1 and pass along the circular conveyor 3 for an irradiation time of about 1 h. After coming out at point 4, they are rotated through  $180^{\circ}$  at point 5 and passed back onto the circular conveyor for another hour of irradiation. The irradiated process loads finally emerge in the warehouse through point 6 at a rate of about  $10 \text{ h}^{-1}$ . The circular source frame A has a diameter of 100 cm and contains several  $^{60}$ Co source rods; three such frames are stacked on top of each other resulting in a total height of about 100 cm. The initial source consisted of 36 source rods, each containing about 280 TBq (7500 Ci) in activity. The average



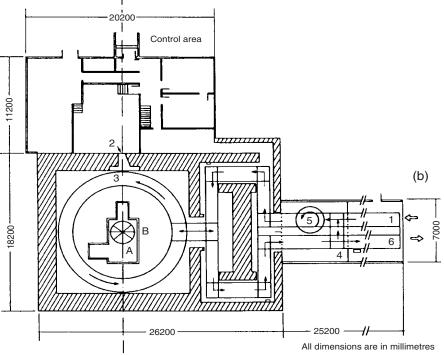


FIG. I–1. Shihoro Agricultural Co-operative Association potato pallet irradiator [I-1, I-2] — (a) view of the irradiation chamber; (b) plan view of the facility: A,  $^{60}$ Co source; B, water pool; 1, entrance line; 2, observation window; 3, irradiation conveyor; 4, transfer from exit to entrance line; 5, turntable; 6, exit line.

values of  $D_{\rm min}$  and  $D_{\rm max}$  were measured to be 60 and 150 Gy [I–1, I–2], i.e. a dose uniformity ratio of 2.5. The same facility can be adapted for the irradiation of onions, with a capacity of about 0.5 t/h per 10 TBq (2 t/h per kCi) of  $^{60}$ Co, over a dose range of 30–66 Gy.

Another early irradiator design dedicated to commercial potato processing in Canada used wooden pallet boxes having internal dimensions of  $98~\rm cm \times 114~\rm cm \times 84~\rm cm$  [I–7]. A dose uniformity ratio of about 2 was achieved for onions and of about 2.5 for potatoes, with the help of attenuators between the source frame and the containers, together with suitable adjustment of the source-to-product distances. The product was irradiated on conveyors passing above, below and on both sides of a horizontal cylindrical source frame, which was raised to the irradiation position from the shielded source pool. This facility is no longer in use and is included here for its particular design principles.

Several pallet irradiators designed for multipurpose applications are now in operation [I–8 to I–10]. Products are placed on standard pallets and transported around a central source. The source geometry used for pallet irradiators is planar, circular or hexagonal. A number of different irradiation geometries have been used. In general, products are irradiated from four sides to obtain an acceptable dose uniformity ratio. Attenuators may be used to improve the dose uniformity. The irradiator may use either the source overlap geometry or the product overlap geometry.

An example of a multipurpose pallet irradiator is shown in Fig. I–2. In this irradiator, products are irradiated at a series of positions around two annular sources. For this design, steel attenuators located at the corners of the rectangular areas around each source reduce the dose at different locations on the outer surfaces of the process loads to improve dose uniformity. Alternatively, the products are irradiated at a sequence of positions along an extended plaque source. Irradiation takes place on two levels (two pallets stacked into one carrier) to provide efficient utilization of the  $\gamma$  energy and to minimize the dose variation in the vertical direction.

A requirement which may need to be considered in the design of pallet irradiators is the necessity to process over a wide dose range, from the low doses required for sprout inhibition of onions to the higher doses required for microbial decontamination of spices. When a high activity source is installed to permit the high throughput processing, for example, of spices at 10 kGy or more, it may not be possible to run the conveyor system sufficiently rapidly for onion irradiation to give a maximum dose of 50 Gy. This requires an increase of the transportation speed of the conveyor system by a factor of about 200. To overcome this, some irradiators have two or more independent source racks so that some of the source activity can remain in the source storage pool during the low dose applications (in the example of Fig. I–2, one source cylinder may be hoisted separately).

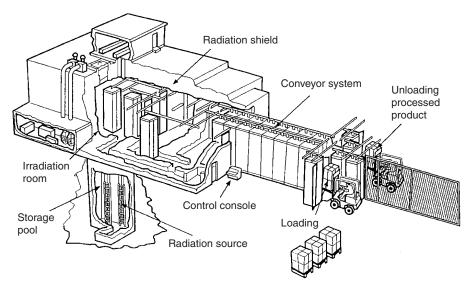


FIG. 1–2. Schematic diagram of a typical <sup>60</sup>Co commercial multipurpose pallet irradiator (also see Fig. 19, p. 46).

## I-1.2. Grain irradiators

A design for a continuous flow irradiator for the disinfestation of grains is illustrated in Fig. I–3. In this design, grain is transferred through pneumatic feed ducts and irradiated as it moves by gravity through annular zones surrounding the source [I–11]. The irradiation chamber is divided into three concentric annular zones, with different residence times in each zone. For a typical irradiation arrangement, the control valves were set to give irradiation times of 130 s for the interior zone, 195 s for the middle zone and 280 s for the exterior zone thus compensating for the lower dose rates in the outer zones. By introducing a series of baffles within the annular zones to cause more turbulent grain flow, a uniformity ratio of about 2 was achieved, as measured by thermoluminescence dosimetry using LiF pellets [I–11, I–12]. The dosimeters were put into miniature capsules, which were mixed in the grain stream and later recovered by sieving as their size was slightly larger than that of the grain kernels. This facility is also no longer in operation and is included here for its particular design principles.

Another design was adopted for a pilot grain irradiator installed in Savannah, Georgia, USA. This design was based on a continuous grid source concept [I–12]. The grain flowed downwards through a horizontal grid consisting of <sup>60</sup>Co source rods, each covered by a protective sheath. This design provided high radiation utilization efficiency and, except for a possible overdose of a small fraction of the

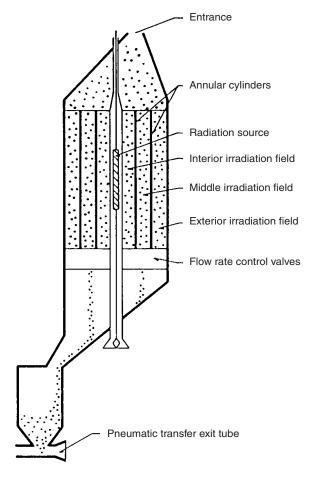


FIG. I–3. A gravity flow grain irradiator with a centrally positioned  $^{60}$ Co  $\gamma$  ray source surrounded by concentric annular cylinders for controlling grain flow during irradiation [I–11].

grains due to being held up or sticking on their passage through the grid, the overall dose uniformity was considered satisfactory.

A different grain irradiator design used tall carriers moving laterally past multiple vertical source plaques [I–13]. This design employed a number of single direction, single pass geometries in a single irradiation room to handle a large throughput. The movement of fixed carriers, rather than particles of grain, around the source racks permits the processing of a variety of grains, flours and other pelleted or powdered products, which may have different flow patterns. However, irradiating particulate products in some kind of silo of the same size as the carriers of any pallet or multipurpose irradiator (Section 3.4.1.2) would serve the same purpose.

## I-1.3. Other multipass irradiators

Packaged cartons containing, for example, dried fruits, fruits and meat products may be irradiated in a <sup>60</sup>Co irradiator similar to those designed for radiation sterilization of health care products. Among the many existing irradiator designs, this type of plant is the most widely used and the dose uniformity ratios achievable are well established. It shows good radiation utilization efficiency and fairly good dose uniformity but usually requires either manual or automated transfer of products from transport pallets to the irradiation carriers. Examples of this type of irradiator design are given in Section 3.4. Some specific multipass radionuclide irradiators used for food are described in Refs [I–14 to I–21].

Such facilities contain a number of transport and transfer systems, which include conveyors to and from the storage area, conveyors for transferring the product through the labyrinth into and out of the radiation chamber, a transfer mechanism at the load/unload stations, and the conveyor system and process load transfer devices which transport the product through the irradiation cycle.

Numerous types of conveyor system have been used. They include:

- (a) Cartons stacked in hanging containers moving along monorails or similar supporting beams,
- (b) Roller conveyors with mechanical pushing devices to transport cartons,
- (c) Cartons in trays that slide or move on wheels with the aid of mechanical or pneumatic transport devices,
- (d) Cartons moved by electrically driven carts.

The process loads are usually irradiated on both sides to flatten the dose distribution in the direction perpendicular to the source plaque (Fig. 12, p. 38). To attain improved uniformity of lateral dose distributions, complex movement schemes are often followed during a complete irradiation cycle, such as that shown in Fig. 19 (p. 46). Depending on the specified values of  $D_{\min}$  and  $D_{\max}$  for different products irradiated together, the packages may be irradiated for different dose increments and the total dose may be obtained by irradiation in two or more irradiation cycles.

## I-1.4. Other bulk flow irradiators

Other foods in addition to grain may be irradiated in bulk. For example, Ref. [I–22] describes an irradiator designed for the bulk flow irradiation of onions. Onions were poured on a large turntable and moved in bulk through the irradiation zone [I–23 to I–25]; the few <sup>60</sup>Co rods necessary for this process were borrowed from a nearby multipurpose irradiation facility during the season.

#### I–2. ACCELERATOR IRRADIATORS

# I-2.1. De-boned poultry irradiator

A linear accelerator facility dedicated to the irradiation of mechanically deboned poultry is currently operating in France [I–26 to I–28]. In this irradiator, frozen slabs of de-boned poultry are irradiated using a 10 MeV linear accelerator to eliminate salmonella and other pathogens. Irradiation is usually performed from two sides with the process load turned over at the end of the first pass. The thickness of the slabs was chosen to be 7 cm in order to achieve optimum homogeneity of dose (Fig. 29, p. 62).

# I-2.2. Meat pasteurization irradiator

Another linear accelerator facility (Fig. I–4) became operational recently with an annual capacity of about 200 000 t of ground meat; presently it is operated in

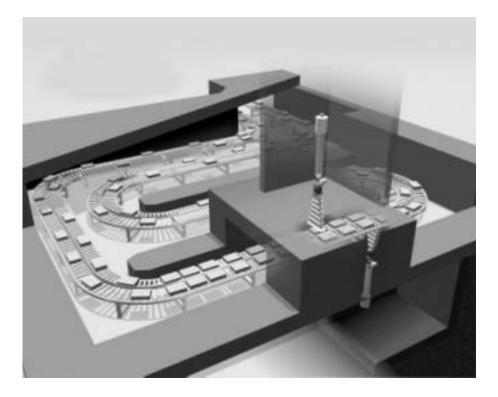


FIG. I-4. Electron beam meat pasteurization facility in Iowa, USA; in operation since 2000.

an alliance with food companies which produce about 75% of the ground meat in the USA [I–29]. The purpose is to eliminate micro-organisms like *Eschericia coli*, *Listeria* and *Campylobacter* as well as other pathogens which are a severe health hazard if hamburgers are only 'cooked rare'.

#### I-2.3. Grain irradiator

An electron beam facility (Fig. I–5) with two irradiation paths, each with one 1.24 MeV accelerator, has been in operation in Odessa, Ukraine since 1984 for the irradiation of imported grain [I–30 to I–32]. It is used when imported grain is found

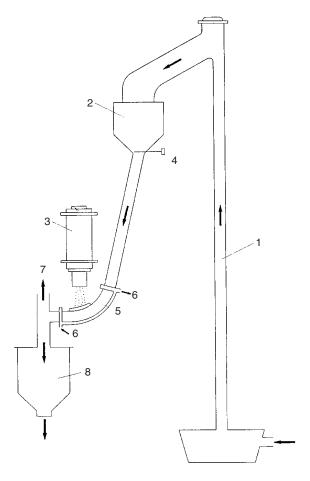


FIG. I–5. Schematic diagram of one irradiation path of the Odessa grain irradiator, with one 1.24 MeV electron accelerator: 1, grain elevator; 2, supply silo; 3, accelerator; 4, sliding valve; 5, irradiation chamber with thin window for electrons; 6, cooling water for irradiation chamber; 7, air and dust exhaust; 8, receiving silo.

to be infested by insects. The facility has the capacity to treat over 400 000 t of grain per year.

#### I-2.4. Other irradiators

Several electron beam pilot-scale irradiators have been built for the investigation of various processes [I–33, I–34]. New high power accelerators are now available which allow high product throughputs with electrons [I–35, I–36] or provide the capability for conversion of electrons to bremsstrahlung.

Another area of intense interest is the possibility of the installation of self-shielded electron beam irradiators at the ends of production lines to allow on-line irradiation of products. A possible application for self-shielded electron beam irradiators is on-line irradiation of ground beef patties prior to final packaging [I–37].

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#### Annex II

## DOSIMETRY REQUIREMENTS AND APPLICATIONS: A CHECKLIST

#### II-1. INTRODUCTION

Dose measurement is central to good quality products in food irradiation processing. It is also essential and required by regulators for a safe and acceptable process, which requires a fully characterized and well documented dosimetry system. In addition, without a recognized traceability such a system has no basis. This system is then used to characterize the facility, qualify the process and provide the necessary process control data. It is expected that the owner of an irradiation facility recognizes the strong relation between dosimetry and successful implementation of the process. To be able to accomplish this it is important that the owner/operator follows documented procedures within a quality assurance plan.

These procedures are discussed in detail in the main text; however, a brief checklist is presented here to capture the essence of the process.

#### II–2. DOSIMETRY REQUIREMENTS

## II-2.1. Selection of dosimetry systems

- (a) To be able to make a meaningful selection, it is important to understand thoroughly the operation of the irradiator and to know the dose requirements of all products that may be irradiated at the facility.
- (b) At least one routine dosimetry system should be carefully selected. If one system cannot cover the required dose range, it may be necessary to select two systems. In any case, it is recommended to have more than one system to ensure redundancy, should one system fail.
- (c) Besides dose range, it is important to consider several other properties such as ease of operation, economics and the dependence of the dosimeter response on various quantities, such as temperature and humidity (see also Section 2.5).
- (d) In some cases, a reference dosimetry system may also be needed, but not all facilities need to have such a system.

#### II-2.2. Characterization

#### II-2.2.1. Calibration

A decision needs to be made as to where to irradiate the routine dosimeters for calibration: either in the production facility or at a calibration facility (Section 2.4.2.1).

## II-2.2.2. Traceability

Depending on the choice of the calibration irradiation, a traceability chain needs to be established to a recognized national or international standards laboratory (Section 2.4.2.2).

### II–2.2.3. Influence quantities

The dosimeter performance is affected by various quantities/parameters. In general, this information is available from the supplier of the dosimeters. Otherwise, this influence should be determined for the relevant quantities, which should include temperature, humidity and dose rate (Section 2.4.2.5).

### II–2.2.4. Uncertainty

It is essential that the uncertainty in the measured dose value is thoroughly investigated and determined considering contributions from all relevant components (Section 2.6).

#### II-3. DOSIMETRY APPLICATIONS

Once a dosimetry system has been characterized, it is ready for various applications at the irradiation facility. These include: facility qualification, process qualification and process control. Each of these activities has its own objectives and thus a certain expertise is required for meaningful accomplishment of the activity.

## II-3.1. Facility qualification

The purpose of dosimetry here is

- (a) To characterize the radiation source, especially for accelerator facilities;
- (b) To carry out dose mapping in a reference geometry;

- (c) To establish relationships between dose and various relevant operating parameters;
- (d) To determine the effect on dose when these parameters are statistically fluctuating during normal operation.

These activities require on the part of the operator an ability to select the irradiation locations for the dosimeters and to interpret the results.

## II-3.2. Process qualification

The purpose of dosimetry here is

- (a) To determine the magnitude and identify the locations of maximum and minimum dose inside a selected process load;
- (b) To measure the variation in these extreme dose values due to small statistical variations in the operating parameters, process load characteristics and uncertainty in the dosimetry system, which helps to establish the two target dose limits;
- (c) To establish the relationships between the extreme dose values and the dose at the reference locations used for process control.

#### II-3.3. Process control

The purpose of dosimetry in process control is

- (a) To provide the evidence that shows that the process has been successfully implemented, i.e. the entire product received dose between the two specified limits.
- (b) To help establish monitoring of the CCPs for the HACCP process.
- (c) To provide the documented evidence necessary for release of the product for its intended use; such evidence is needed by the regulators and by the national authorities in the case of international trade.

#### II-4. TRAINING

It is expected that the dosimetry personnel at the irradiation facility have enough training and understanding of the dose measurement process so as to be able

(a) To carry out all the dosimetry activities at the irradiation facility as described in this book,

- (b) To interpret the results of the measurements,
- (c) To understand the significance of uncertainties in the measured values,
- (d) To interpret the measurements made under different irradiation conditions,
- (e) To calculate the dose to the material of interest (if needed),
- (f) To appreciate the effect of statistics in the measured values.

It is also expected that the owner of the facility appreciates the importance of dosimetry and is committed to support it financially for activities including staff training, procurement of necessary equipment, calibration, and establishing and maintaining traceability, commensurate with the commercial commitment of the facility.

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Published by and available from:

American Society for Testing and Materials (ASTM), 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, USA (website — http://www.astm.org).

- E170 Standard Terminology Relating to Radiation Measurements and Dosimetry<sup>1</sup>
- E275 Standard Practice for Describing and Measuring Performance of Ultraviolet, Visible, and Near-Infrared Spectrophotometers<sup>2</sup>
- E456 Standard Terminology for Relating to Quality and Statistics<sup>3</sup>
- E666 Standard Practice for Calculating Absorbed Dose from Gamma or X Radiation<sup>1</sup>
- E668 Standard Practice for Application of Thermoluminescence-Dosimetry (TLD) Systems for Determining Absorbed Dose in Radiation-Hardness Testing of Electronic Devices<sup>1</sup>
- E925 Standard Practice for the Periodic Calibration of Narrow Band-Pass  $Spectrophotometers^2$
- E958 Standard Practice for Measuring Practical Spectral Bandwidth of Ultraviolet–Visible Spectrophotometers<sup>2</sup>
- E1026 Standard Practice for Using the Fricke Reference Standard Dosimetry System<sup>1</sup>

<sup>\*</sup> Each Standard is updated about every five years. The latest Annual Book of ASTM Standards should always be consulted.

<sup>\*\*</sup> From October 2002, Standards E1204–E1956 will become joint ASTM and ISO Standards. The new designations will be, for example, ISO/ASTM 51204 instead of ASTM E1204, where 'E' is replaced by '5'.

<sup>&</sup>lt;sup>1</sup> Annual Book of ASTM Standards, Vol. 12.02.

<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol. 03.06.

<sup>&</sup>lt;sup>3</sup> Annual Book of ASTM Standards, Vol. 14.02.

E1204	Processing <sup>1</sup>
E1205	Standard Practice for Use of a Ceric-Cerous Sulfate Dosimetry System <sup>1</sup>
E1261	Standard Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing <sup>1</sup>
E1275	Standard Practice for Use of a Radiochromic Film Dosimetry System <sup>1</sup>
E1276	Standard Practice for Use of a Polymethylmethacrylate Dosimetry System <sup>1</sup>
E1310	Standard Practice for Use of a Radiochromic Optical Waveguide Dosimetry System <sup>1</sup>
E1400	Standard Practice for Characterization and Performance of a High-Dose Radiation Dosimetry Calibration Laboratory <sup>1</sup>
E1401	Standard Practice for Use of a Dichromate Dosimetry System <sup>1</sup>
E1431	Standard Practice for Dosimetry in Electron and Bremsstrahlung Irradiation Facilities for Food Processing <sup>1</sup>
E1538	Standard Practice for Use of the Ethanol-Chlorobenzene Dosimetry System <sup>1</sup>
E1539	Standard Guide for the Use of Radiation-Sensitive Indicators <sup>1</sup>
E1540	Standard Practice for Use of a Radiochromic Liquid Dosimetry System <sup>1</sup>
E1607	Standard Practice for Use of the Alanine-EPR Dosimetry System <sup>1</sup>
E1608	Standard Practice for Dosimetry in an X-Ray (Bremsstrahlung) Facility for Radiation Processing <sup>1</sup>
E1631	Standard Practice for Use of Calorimetric Dosimetry Systems for Electron Beam Dose Measurements and Dosimeter Calibrations <sup>1</sup>
E1649	Standard Practice for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies between 300 keV and 25 MeV <sup>1</sup>

<sup>&</sup>lt;sup>1</sup> Annual Book of ASTM Standards, Vol. 12.02.

E1650	Standard Practice for Use of Cellulose Acetate Dosimetry Systems <sup>1</sup>
E1702	Standard Practice for Dosimetry in a Gamma Irradiation Facility for Radiation Processing <sup>1</sup>
E1707	Standard Guide for Estimating Uncertainties in Dosimetry for Radiation Processing <sup>1</sup>
E1818	Standard Practice for Dosimetry in an Electron Beam Facility for Radiation Processing at Energies between 80 and 300 $\rm keV^1$
E1900	Standard Guide for Dosimetry in Radiation Research on Food and Agricultural Products <sup>1</sup>
E1939	Standard Practice for Blood Irradiation Dosimetry <sup>1</sup>
E1940	Standard Guide for Dosimetry for Irradiation of Insects for Sterile Release Programs <sup>1</sup>
E1956	Standard Practice for Use of Thermoluminescence-Dosimetry (TLD) Systems for Radiation Processing <sup>1</sup>
E2116	Standard Practice for Dosimetry for a Self-Contained Dry-Storage Gamma-Ray Irradiator <sup>1</sup>
F1355	Standard Guide for Irradiation of Fresh Fruits as a Phytosanitary Treatment <sup>1</sup>
F1356	Standard Guide for the Irradiation of Fresh and Frozen Red Meat and Poultry to Control Pathogens and Other Microorganisms <sup>1</sup>
F1416	Standard Guide for the Selection of Time–Temperature Indicators <sup>4</sup>
F1640	Standard Guide for Packaging Materials for Foods to be Irradiated <sup>1</sup>
F1736 F1885	Standard Guide for Irradiation of Finfish and Shellfish to Control Pathogens and Spoilage Microorganisms <sup>1</sup> Standard Guide for Irradiation of Dried Spices, Herbs, and Vegetable Seasonings to Control Pathogens and Other Microorganisms <sup>1</sup>

<sup>&</sup>lt;sup>1</sup> Annual Book of ASTM Standards, Vol. 12.02.

<sup>&</sup>lt;sup>4</sup> Annual Book of ASTM Standards, Vol. 15.09.

#### B. LIST OF ICGFI PUBLICATIONS

These documents are available from the ICGFI secretariat at IAEA, Wagramer Strasse 5, A-1400 Vienna, Austria (website — http://www.iaea.org/icgfi/d5/index.html).

Document No. 1: Guidelines for Preparing Regulations for the Control of Food Irradiation Facilities (1991).

Document No. 2: International Inventory of Authorized Food Irradiation Facilities (also updated on the Internet) (1999).

Document No. 3: Code of Good Irradiation Practice for Insect Disinfestation of Cereal Grains (1991).

Document No. 4: Code of Good Irradiation Practice for Prepackaged Meat and Poultry (to control pathogens and/or extend shelf-life) (1991).

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Document No. 6: Code of Good Irradiation Practice for Shelf-life Extension of Bananas, Mangoes and Papayas (1991).

Document No. 7: Code of Good Irradiation Practice for Insect Disinfestation of Fresh Fruits (as a quarantine treatment) (1991).

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#### **GLOSSARY**

**absorbance,** A**.** Also referred to as optical density, OD. This is defined as the logarithm to base 10 of the reciprocal of the transmittance,  $\tau$ , of an optically absorbing medium in a densitometric or spectrophotometric measurement:

$$A = \log_{10}(1/\tau)$$

where  $\tau = III_0$ ,  $I_0$  being the incident luminous flux density of the light and I the transmitted luminous flux density. For example, A=1.0 represents 10% incident light transmitted and A=2.0 represents 1% transmitted. When this quantity is measured at a given wavelength of light,  $\lambda$ , it is called spectral absorbance,  $A_{\lambda}$ , or spectral optical density.

**absorbed dose,** D. The mean energy,  $d\bar{\epsilon}$  imparted by ionizing radiation to matter in a volume element divided by the mass, dm, of matter in the volume element

$$D = d\bar{\epsilon}/dm$$

The SI derived unit of absorbed dose is the gray (Gy); the traditional special unit was the rad, use of which is being phased out.

**absorbed dose rate,**  $\dot{D}$ . The change in absorbed dose, dD, divided by the time interval, dt, during which absorption occurs:

$$\dot{D} = dD/dt$$

The SI unit is the gray per second (Gy/s); the traditional special unit was the rad per second (rad/s), use of which is being phased out.

- **absorber.** Any matter placed in the path of a radiation beam. Such matter causes a reduction in the radiation flux and, often, a change in radiation quality in the beam behind the absorber; the magnitude of the change varying with the type and spectrum of the radiation and the amount and atomic constituency of the material.
- **absorption coefficient.** Generally refers to the energy absorption coefficient. This could be for  $\gamma$  rays or photons, and could refer to either the linear energy absorption coefficient,  $\mu_{\rm en}/\rho$ . The energy absorption coefficient of a material, for uncharged particles, is the product of the energy transfer coefficient of the material and 1-g, where g is the fraction of the energy of liberated charged particles that is lost in radiative processes in the material. For materials with low atomic number (most foods), g is negligible. Thus, the two coefficients are almost the same.

- **accuracy of measurement.** The closeness of agreement between a measurement result and an accepted reference value. (This should not be confused with 'precision').
- activity, A. The amount of radionuclide in a particular energy state at a given time, defined as

$$A = dN/dt$$

where dN is the expectation value of the number of spontaneous nuclear transformations from the given energy state in the time interval dt. The SI derived unit of activity is the reciprocal second ( $s^{-1}$ ), termed the becquerel (Bq). The traditional special unit was the curie (Ci), use of which is being phased out.

- **annihilation radiation.** Electromagnetic radiation of 0.51 MeV energy (two photons) resulting from the interaction between a positron and an electron, where both are annihilated.
- **beam power.** The power or energy flux of a radiation beam. Usually given in watts  $(\equiv J/s)$ .
- **becquerel**, (**Bq**). The name for the SI unit of activity, equal to one transformation per second. It supersedes the traditional special unit curie (Ci),

1 Bq = 
$$2.7 \times 10^{-11}$$
 Ci (approximately, for exact value see curie)

- **binding energy.** For a particle in a system, it is the net energy required to move it from the system to infinity. For a system, it is the net energy required to decompose it into its constituent particles.
- bremsstrahlung. Broad spectrum electromagnetic radiation emitted when an energetic electron (or a charged particle) is influenced by a strong magnetic or electric field, such as that in the vicinity of an atomic nucleus (the German word literally means 'braking radiation'). In practice, it is produced when an electron beam strikes any material (converter). The bremsstrahlung spectrum depends on the electron energy, and the converter material and its thickness, and has its maximum energy corresponding to the maximum energy of the incident electrons.
- **broad beam.** A stream of incident radiation where the lateral dimensions are relatively large with respect to the size of the absorber or target (as contrasted to a narrow beam).

- **buildup.** In the passage of radiation through a medium, the increase with depth of energy deposition due to the forward moving secondary radiation. It leads to a maximum in the depth–dose curve. For example, for  $^{60}$ Co  $\gamma$  radiation, this maximum occurs at a depth of about 0.5 cm in water.
- **bulk density.** The mass per unit volume of the product 'en mass', as it would be irradiated (e.g. with potatoes, the air space between the potatoes is also considered in determining the bulk density).
- **calibration curve.** A graphical representation of the relationship between dosimeter response and absorbed dose for a given dosimetry system, established under controlled conditions in which the doses are determined by comparison with a standard reference dosimeter.
- **Codex Alimentarius.** A collection of internationally adopted food standards (codes of practice, guidelines, etc.) resulting from the activities of the Joint FAO/WHO Food Standards Programme as implemented by the Codex Alimentarius Commission, on which 161 states were represented in 1999.
- **confidence level.** Also referred to as level of confidence. See confidence probability.
- **confidence limits.** Upper and lower values in a distribution of data within which a characteristic value of interest from a succeeding measurement falls with the confidence probability. Both values of the confidence limits are calculated from the parameters of the distribution by the use of the *t* value or of a coverage factor. The value of interest can be the mean value from a series of measurements or an individual measurement value.
- **confidence probability.** The probability with which a result will fall within specified confidence limits.
- **conversion efficiency.** Also referred to as conversion ratio. In the production of bremsstrahlung (X rays) by the slowing down of electrons or other charged particles, it is the ratio of the energy flux of the resulting photons emitted in the forward direction to the energy flux of the incident electrons.
- **correction and correction factor.** A value added algebraically and a numerical multiplying factor, respectively, to compensate for the systematic error of the uncorrected measurement.

- **coverage factor.** Also referred to as the *k* value. In statistical treatments of measured data, the coverage factor is a function of the fraction of measurements considered, the confidence probability required and the number of measurement values available. It is used as a multiplier of the combined standard uncertainty in order to obtain an expanded uncertainty. Also see 'uncertainty'.
- **curie** (Ci). The special unit of activity, which is superseded by the becquerel (Bq). The curie is defined as:

1 Ci = 
$$3.7 \times 10^{10}$$
 Bq (exactly)

- **cycle time.** In shuffle–dwell irradiators, the cycle time is equal to the dwell time plus the shuffle time; where the dwell time is the time interval during which a process load is at rest at an irradiation position and the shuffle time is the time interval during which the process load is being transferred from one irradiation position to the next.
- **depth-dose distribution.** The variation of absorbed dose with depth from the incident surface of a material exposed to radiation, as determined along the central axis of the beam.
- **disinfestation.** Control of the proliferation of insect and other pests in grain, cereal products, dried fruit, spices, etc. This requires a dose of  $\approx 0.2-1$  kGy, which will usually kill pests in all life-cycle stages.

dose. See absorbed dose.

**dose distribution.** The spatial variation of absorbed dose throughout the process load, the dose having the extreme values  $D_{\max}$  and  $D_{\min}$ .

dose rate. See absorbed dose rate.

- **dose uniformity ratio,** U. Also referred to as the max/min ratio. The ratio of maximum to minimum absorbed dose in a process load, i.e.  $U = D_{\text{max}}/D_{\text{min}}$ .
- **dosimeter.** A device that, when irradiated, exhibits a quantifiable change in some property of the device that can be related to absorbed dose in a given material using appropriate analytical instrumentation and techniques.
- **dosimetry system.** A system used for determining absorbed dose, consisting of dosimeters, measurement instruments and their associated reference standards, and procedures for the system's use.

- **dwell time.** The time interval during which a process load is at rest at an irradiation position in a shuffle–dwell irradiator.
- **electron accelerator.** A device for imparting large amounts of kinetic energy to electrons.
- **electron beam.** An essentially monodirectional stream of electrons which have usually been accelerated electrically or electromagnetically to high energy.
- **electron equilibrium.** A condition that exists in an incremental volume within a material under irradiation if the kinetic energies and the number of electrons entering that volume are equal to those leaving the volume.
- **electronvolt** (**eV**). A unit of energy. One electronvolt is the kinetic energy acquired by an electron in passing through a potential difference of one volt in vacuum. It is approximately equivalent to

$$1 \text{ eV} = 1.60219 \times 10^{-19} \text{ J}$$

- **entrance dose.** Absorbed dose in a product at the entrance surface (or extrapolated to this surface), i.e. where the radiation beam enters the product.
- **error** (of measurement). The result of a measurement minus the true value of the measurand; note: since a true value cannot be determined, in practice a best estimate or conventional true value is used.
- **exit dose.** The absorbed dose in a product at the exit surface (or extrapolated to this surface), i.e. where the radiation beam leaves the product.

Gaussian distribution. See normal distribution.

- **go/no go monitor.** Also referred to as a radiation sensitive indicator. A radiation detector that undergoes an easily detectable change under irradiation at a given absorbed dose level such that it is possible to state that the detector material has been irradiated to roughly that dose or above that dose. It is frequently used for inventory control in food irradiation facilities to confirm that a process load has passed through the irradiation process. Visually observable changes, such as colour changes, are typical of those exhibited by go/no go monitors.
- **gray** (Gy). The SI derived unit of absorbed dose of ionizing radiation, being equal to one joule of energy absorbed per kilogram of matter undergoing irradiation. Its relationship to the now obsolete special unit, the rad, is 1 Gy = 100 rad.

**G value.** This is the measure of radiation chemical yield in an irradiated substance. It is defined as the quotient of n(x) by  $\varepsilon$ , where n(x) is the mean amount of a specified entity, x, produced, destroyed or changed by the energy imparted,  $\varepsilon$ , to the matter of that system; thus  $G(x) = n(x)/\varepsilon$ , and the unit is mol/J.

Examples of such chemical changes are production of, for example, particular molecules, free radicals and ions.

**Note:** However, most data are given in number of molecules produced, destroyed or changed per 100 eV. The conversion to SI units is as follows:

1 mol/J = 
$$9.65 \times 10^6$$
 molecules/100 eV  
1 molecule/100 eV =  $1.036 \times 10^{-7}$  mol/J

- **half-life** (**radioactive**),  $T_{1/2}$ . For a radionuclide, the time required for the (radio)activity to decrease, by a radioactive decay process, by half. In food irradiation, it is used to determine the reduction of the source strength (activity) of a  $\gamma$  ray source with time.
- **heat capacity.** The quantity of heat (i.e. energy) required to raise the temperature of a given mass of substance by one degree Kelvin. The SI unit is J/K. The SI unit for specific heat capacity, or heat capacity per unit mass, is  $J \cdot kg^{-1} \cdot K^{-1}$ .
- **inventory control.** An administrative check to ensure that each process load is treated once and only once, this also includes separation of treated and untreated product.
- **ionization.** A process in which a charged particle is created from a parent atom or molecule or other bound state.
- ionizing radiation. Any type of radiation consisting of charged (directly ionizing) particles or uncharged (indirectly ionizing) particles, or both, that as a result of physical interaction, creates ions by either primary or secondary processes. For example, the charged particles could be positrons or electrons, protons, or other heavy ions and the uncharged particles could be X rays, γ rays or neutrons.
- **irradiation cycle.** The entire sequence of events experienced by a process load from the instant it enters the irradiation chamber (beginning of radiation treatment) to the instant it leaves the irradiation chamber (completion of radiation treatment).
- **irradiation facility.** An engineering plant, housing the radiation source and all the ancillary equipment required for carrying out a radiation process.

**irradiation geometry.** The spatial description of the relative positions of the process load and the radiation source during the radiation treatment (comprises source-to-product distance, size, spacing, shape and position of scattering or shielding materials, etc.).

**irradiator.** That part of the irradiation facility that houses the radiation source and associated equipment, i.e. the radiation chamber inside the radiation protection shield.

**isotopes.** Nuclides having the same atomic number, Z (i.e. the same chemical element) but having different mass number, A.

k value. See coverage factor.

**labyrinth.** A passage linking two areas that is designed to follow a tortuous path such that no radiation originating in one area can reach the other area without undergoing at least one reflection or scattering off the passage wall.

**level of confidence.** See confidence probability.

mass per unit area. A parameter used for specifying the thickness of an absorber. It is obtained by multiplying the absorber thickness by the density of the absorber. There is no agreement on a name for this, it being variously called surface density, density thickness, area density, mass thickness, standardized thickness and, simply, thickness. The dimensionally descriptive title is used in this book:

mass per unit area  $(kg/m^2)$  = thickness  $(m) \times density (kg/m^3)$ .

mass stopping power. See stopping power.

measurand. The particular quantity subject to measurement.

micro-organisms. Microscopically small organisms, mostly unicellular, including groups like bacteria, yeasts, moulds and viruses. Except for viruses, the first three groups may grow on (inanimate) food material, some may cause its spoilage (undesirable sensory changes), others (the pathogens) may cause illness of the consumer (food-borne disease). They may cause spoilage and damage to health simultaneously. Some micro-organisms, of which bacteria are of special practical importance, produce enduring forms called spores which normally are more resistant to destructive agents in their environment (e.g.

radiation, heat and chemicals). Other micro-organisms cannot form spores and consist only of vegetative cells which, as a rule, are less resistant (non-spore-forming micro-organisms) to such destructive agents.

**molar linear absorption coefficient, E.** Also referred to as the molar extinction coefficient. A constant relating the spectrophotometric absorbance,  $A_{\lambda}$ , of an optically absorbing molecular species at a given wavelength,  $\lambda$ , per unit path length, d, to the molar concentration, c, of that species in solution:

 $\varepsilon = (A_{\lambda}/d)/c$ , and the SI unit is m<sup>2</sup>/mol.

**narrow beam.** In beam attenuation measurements, a radiation beam in which only the unscattered and small angle forward scattered radiation reach the detector.

**normal distribution.** Also referred to as a Gaussian distribution. It is the probability distribution of a continuous random variable. The probability density function is mathematically described by the Gaussian equation, which is completely determined by two parameters, the mean value and the standard deviation. The distribution is bell shaped with replicate values deviating randomly on either side of the mean value; the mathematical equation and detailed treatment of the subject may be found in Ref. [43].

optical density, OD. See absorbance.

plaque source. An arrangement of radionuclide sources in the planar configuration.

**precision.** The closeness of agreement between measurements obtained under prescribed conditions.

**primary (standard) dosimeter.** Dosimeter of the highest metrological quality, established and maintained as an absorbed dose standard by a national or international standards organization. The two most commonly used types of primary standard dosimeter are ionization chambers and calorimeters.

**primary radiation.** Incident radiation before interaction with any medium.

process load. Volume of material with a specified loading configuration irradiated as a single entity, such as a box, tote or carrier. For example: A product carrier of an irradiation facility may hold two pallets packed with a large number of retail packages to capacity; the contents of this carrier are called the process load. Also see production run.

- **processor.** The organization, group, company, contractor or person responsible for an irradiation facility.
- **product.** The food, in bulk or packaged, raw or processed, that is to be treated by radiation, i.e. the target of the irradiation process. The term 'product' is used in this book for the food product at any time before, during and after irradiation.
- **product equivalence.** The quality of a material such that its radiation interaction (absorption and scattering) characteristics correspond closely to those of the product of interest.
- **product overlap.** With radionuclide irradiators, the extension of the product beyond the cross-sectional dimensions of the source, as the product passes the plaque source. This technique is usually employed to improve the energy absorption efficiency in multipass, single position or multiposition, one or two direction irradiation facilities.
- **production run.** Series of process loads consisting of materials, or products having similar radiation absorption characteristics, that are irradiated sequentially to a specified range of absorbed dose. This refers to both continuous flow and shuffle–dwell processes. Also see process load.
- radappertization. Exposure of foods in sealed containers to doses of ionizing radiation sufficient to kill all organisms of food spoilage and public health significance to achieve 'commercial sterility'. In the absence of post-processing contamination, no microbial spoilage or toxins should become detectable with recognized testing methods, however long or whatever the conditions under which the food is stored. Dose ranges from ≈25 to 70 kGy.

radiation. Refers to ionizing radiation in this book. See ionizing radiation.

- **radiation process.** As applied to food irradiation, the act of irradiating a product in order to treat it in a beneficial way, for example to improve its intrinsic or commercial value and to extend its keeping qualities.
- **radiation source.** An apparatus or radioactive substance in a suitable support that constitutes the origin of the ionizing radiation (e.g. an electron accelerator or <sup>60</sup>Co source rods in a frame).

radiation spectrum. The distribution of spectral energy of a given radiation.

**radicidation.** Practical elimination of pathogenic organisms and micro-organisms other than viruses by means of irradiation. It is achieved by: (i) the destruction of organisms like tape worm and trichina in meat, for which doses range between 0.3 and 1 kGy, (ii) the reduction of the number of viable specific non-spore-forming pathogenic micro-organisms, such that none is detectable in the treated food by any standard method, for which doses range between 2 and 8 kGy.

radioactivity. See activity.

radionuclide. A radioactive nuclide.

radurization. The application to foods of doses of ionizing radiation sufficient to enhance their keeping quality (usually at refrigeration temperature) by causing a substantial decrease in the number of viable specific spoilage microorganisms. The dose ranges vary from ≈1 to 10 kGy.

**range.** The distance that an electron (in general a charged particle) penetrates a given substance in a specified direction before its kinetic energy is reduced to such a level that it can no longer cause ionization.

**reference** (**standard**) **dosimeter.** A dosimeter of high metrological quality used as a standard to provide measurement traceability to and consistency with primary standard dosimeters.

**repeatability.** The closeness of the agreement between the results of successive measurements of the same measurand carried out under the same conditions of measurement. These conditions are called repeatability conditions, which include the same measurement procedure, the same observer and the same measuring instrument used under the same conditions, at the same location and repeated over a short period of time.

**residence time.** The time the product must be in the irradiation chamber to complete its radiation treatment, usually equal to the time required for one irradiation cycle.

**response.** The radiation induced effect in the dosimeter measured by an instrument.

**routine dosimeter.** A dosimeter used for routine absorbed dose measurements and which must be calibrated against a primary, reference or transfer standard dosimeter.

- **scattering.** The change in direction of a particle or photon due to a collision or interaction with another particle, atom or system.
- **secondary radiation.** Radiation resulting from the interaction of primary radiation with any medium.
- **shuffle dose.** In a shuffle–dwell irradiator, the absorbed dose received by the product during its movement from one dwell (irradiation) position to the next.
- **shuffle–dwell irradiator.** An irradiator in which a process load moves discontinuously past the irradiation source, alternately being moved (indexed) to a new irradiation position and then remaining at rest for a specified period at that position.
- source activity augmentation. Also referred to as source activity enhancement. Introduction of additional radionuclide source material or higher activity source material in specific places in a large area plaque source to improve dose uniformity in the process load being treated. Typically such augmentation is required at the edges of a source plaque near which the dose would otherwise tend to diminish.
- **source overlap.** For a radionuclide irradiator, it is the extension of the radiation source beyond the cross-sectional dimensions of a process load as it passes the plaque source or a collimated beam. For an accelerator source, it is the sweep of the beam, especially for a scanned electron beam, beyond the edges of a process load.
- **source strength.** With reference to a  $\gamma$  ray source, it is the activity level of the radioactive material expressed in becquerels (or curies).
- **spectrophotometer.** An instrument for measuring either transmittance or absorbance (optical density) of light as a function of wavelength for a given material.
- standardized thickness. See mass per unit area.
- **stopping power.** This could refer to either the linear stopping power, S, or the mass stopping power,  $S/\rho$ . Its value depends on the material and the energy of the charged particle. The mass stopping power of a material, for charged particles (such as electrons), is the quotient of dE by  $\rho$  dl, where dE is the energy lost by a charged particle in traversing a distance dl in the material of density  $\rho$ , thus.

 $S/\rho = dE/\rho dl$  in SI units of J·m<sup>2</sup>/kg

The linear stopping power is then defined as

S = dE/dl in SI units if J/m.

surface dose. See entrance dose and exit dose.

- **throughput.** In food irradiation, this is the amount of product emerging from the irradiator per unit time (kg/h) multiplied by the absorbed dose (J/kg). This should be equal to the power of the radiation source multiplied by the energy utilization efficiency.
- **traceability.** Also referred to as measurement traceability. This is the 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 and internationally recognized standards. It is commonly achieved through a secondary standards laboratory or a primary standards laboratory that is certified for this purpose.
- **transfer** (**standard**) **dosimeter.** Dosimeter, often a reference standard dosimeter, suitable for transport between different locations used to compare absorbed dose measurements. It is generally used for establishing measurement traceability. These dosimeters should be used under conditions that are carefully controlled by the issuing laboratory.
- **transit dose.** For a stationary radiation source, this is the dose received by the product during its movement into and out of the radiation field. For a stationary product, it is the dose received by the product while the movable source moves into or out of its irradiation position.

transmittance. See absorbance.

- t value or Student's t value. The value used in statistical analyses to determine the range and reliability of measurements and results. It is a function of the confidence probability required and the number of measurement values available.
- **uncertainty** (of measurement). The parameter, associated with the result of a measurement, that characterizes the dispersion of the values reasonably attributed to the measurand. The parameter may be, for example, a standard

deviation, or the halfwidth of an interval having a stated confidence probability. There are two methods for estimating uncertainty: *Type A*, by statistical analysis of a series of observations; *Type B*, by other means. When the uncertainty is expressed as a standard deviation, it is referred to as the standard uncertainty. The total uncertainty obtained by combining all the standard uncertainties corresponding to various components of the measurement process is referred to as the combined standard uncertainty. The expanded uncertainty is obtained by multiplying the combined standard uncertainty by the coverage factor, *k*. For a fuller treatment of this subject, refer to Ref. [43].

uniformity ratio. See dose uniformity ratio.

**utilization efficiency.** The fraction of radiation energy emitted by the radiation source that is absorbed by the total product during the irradiation cycle.

water equivalence. The quality of a material such that its radiation interaction (absorption and scattering) characteristics correspond closely to those of water.

**X rays.** Penetrating electromagnetic radiation (photons) usually produced by high energy electrons impinging on a metal target. Also see bremsstrahlung.

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