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DOSIMETRY SYSTEMS FOR RADIATION PROCESSING*

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ABSTRACT

Dosimetry serves important functions in radiation processing, where large absorbed doses and dose rates from photon and electron sources have to be measured with reasonable accuracy. Proven dosimetry systems are widely used to perform radiation measurements in development of new processes, validation, qualification, and verification (quality control) of established processes and archival documentation of day-to-day and plant-to-plant processing uniformity. Proper calibration and traceability of routine dosimetry systems to standards are crucial to the success of many large-volume radiation processes. Recent innovations and advances in performance of systems that enhance radiation measurement assurance and process diagnostics include dose-mapping media (new radiochromic film and solutions), optical waveguide systems for food irradiation, solid-state devices for real-time and passive dosimetry over wide dose-rate and dose ranges, and improved analytical instruments and data acquisition.

KEY WORDS

Calibration; dose mapping; dosimetry systems; electron processing; gamma-ray processing; high-dose dosimetry; novel dosimeters; radiochromic dosimeters; solid-state dosimeters

INTRODUCTION

Industrial radiation applications of ionizing radiation sources of photons (x- and gamma rays) and electrons (accelerators) encompass a wide range of absorbed doses, D (about six orders of magnitude), dose rates, $\dot{D}$ (twelve orders of magnitude), and energies, E (over two orders of magnitude.

It is the quantity, D, that must be measured (or calculated) by dosimetry (or computational characterization of parameters) (McLaughlin *et al.*, 1989a; Seltzer and Berger, 1987; McKeown and Drewell, 1994). The aim here is to feature essential dosimetry concepts and advances which have arisen since the IMRP8 meeting in Beijing (Huang Qitao *et al.*, 1993). Table 1 gives a synopsis of systems, read-out methods, and useable dose ranges.

*The mention of commercial products throughout this paper does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the products identified are necessarily the best available for the purpose.

Table 1. Important dosimetry systems for radiation processing

Dosimeter Type	Method of Readout	Examples	Typical Absorbed Dose Range (Gy)
calorimeters	temperature measurement	graphite, water, polystyrene	$10^1 - 10^4$
organic crystals	EPR spectrometry	alanine, sucrose, cellulose	$10^0 - 10^5$
diamonds	electrical measurement EPR spectrometry	diamond crystals and films	(dose rate measurement)
semiconductors	electrical measurement	Si diodes, MOSFETs	$10^0 - 10^4$
inorganic crystals	spectrophotometry EPR spectrometry	LiF, SiO_2, Suprasil™ glass	$10^3 - 10^7$
chemical solutions	spectrophotometry spectrofluorimetry	ceric-cerous, organic acids, ethanol-chlorobenzene	$10^2 - 10^5$
radiochromic films and optical wave guides	spectrophotometry microdensitometry	dyed plastics, polydiacetylenes	$10^0 - 10^6$
fluorescent systems	spectrofluorimetry spectrophotometry	inorganic and organic fluors	$10^0 - 10^4$

IMPLEMENTATION OF DOSIMETRY

High-dose dosimetry for radiation processing entails the performance of several well-tried functions of dosimetry (McLaughlin *et al.*, 1989a; Saylor and McLaughlin, 1993):

- The dose measurements in radiation research leading to successful processing
- Proper calibration and traceability to standards using reference dosimetry.
- Process validation, including dose settings for meeting process specifications.
- Process qualification, including the establishment of locations and values of minimum and maximum doses.
- Process verification, by the coordination of routine dosimetry and the monitoring of process parameters.

When planning and conducting a given gamma-ray or electron-beam process, optimization of the dosimetry in each of these stages may require different approaches, and sometimes different dosimetry systems, depending on the type of process. For example, with photons, the main process parameters are source decay factors, source array patterns, product carrier speed and dwell times, product movements and indexing codes, and carrier load configurations. With electron beams, the critical process parameters are beam energy, beam current, scanning factors and uniformity, beam pulse characteristics, carrier speed, and carrier load configuration. In the case of the stringent requirements of medical product sterilization or food irradiation, the

case of the stringent requirements of medical product sterilization or food irradiation, the documentation of validation, accurate dose interpretation and dose mapping, and the proper choice of suitable dosimetry systems must all be keyed to these parameters.

The criteria for selecting suitable dosimetry systems are summarized in Table 2.

Table 2. Criteria for the selection of routine dosimeters

- Calibrated response over a specified dose range for a given radiation type and energy
- Suitability of a given dosimetry system and its response function over the dose range of interest
- Measurement reproducibility and stability
- Energy deposition characteristics: radiation type, spectrum, fractionation and dose rate
- Conditions before and after irradiation
- Conditions during irradiation
- Packaging, handling, geometrical conditions of irradiation
- Analytical technique and conditions and ease and speed of readout
- Dosimeter stability factors
- Cost and availability versus application

DOSIMETER CALIBRATIONS

National and secondary standards laboratories provide calibrations of dosimetry systems traceable to national standards for radiation processing facilities. At the U.S. National Institute of Standards and Technology (NIST), high-dose dosimeters can be calibrated over the dose range $10^0 - 10^7$ Gy using ^{60}Co gamma-ray sources or high-energy (1 to 28 MeV) electron beams. The experimental calibrations are also supported by theoretical calculations and reference data. The main participants in such calibrations are medical radiotherapy (clinical dosimetry) and industrial (radiation processing) users. Figure 1 gives a diagram of the facilities, typical applications, and reference dosimetry techniques as they are related to the official NIST high-dose measurement services (Humphreys *et al.*, 1988).

The gamma-ray and electron calibrations at NIST are traceable to two kinds of calorimetry as the national standards. They are, respectively, the water calorimetry measurements (Domen, 1994) in the NIST teletherapy (14.5 cm × 14.5 cm) ^{60}Co gamma-ray beam and the graphite calorimetry measurements (McLaughlin, *et al.*, 1994a) at the NIST Medical-Industrial Radiation Facility (MIRF) electron linac beams. As shown in Fig. 2 the former method utilizes alanine/EPR (electron paramagnetic resonance) transfer standard dosimetry (Desrosiers *et al.*, 1994) to establish absorbed dose rates in three NIST reference ^{60}Co sources (water pool source and two gamma cells GC 45 and GC 232) for high-dose calibration of different routine dosimeters in different geometries.

A reference calibration service by mail, featuring the alanine/EPR system, similar to that of the UK National Physical Laboratory (NPL) using packaged calibrated alanine pellets, is being introduced at NIST. This service is parallel to the one that has been used at NIST for more than the past 20 years, which employs packaged calibrated radiochromic film dosimeters (Humphreys *et al.*, 1988). The transfer from the primary calorimetry standard to the reference standard for gamma radiation is illustrated in Fig. 3 for the NIST pool source geometry. The radiation

quantities for reference calibrations are the absorbed dose, D_w, and dose rate, $\dot{D}_w$, in water and, therefore, appropriate corrections from dose rate in polystyrene, $\dot{D}_{ps}$, must be applied. The indicated calculations in Fig. 3, involving ratios of mass energy-absorption coefficients, which must be weighted over the approximate photon energy fluence spectrum for each gamma-ray source geometry (McLaughlin *et al.*, 1989a). Differences in attenuation between the more bulky polystyrene calorimeter and the relatively small alanine or film holder must, accordingly, also be taken into account.

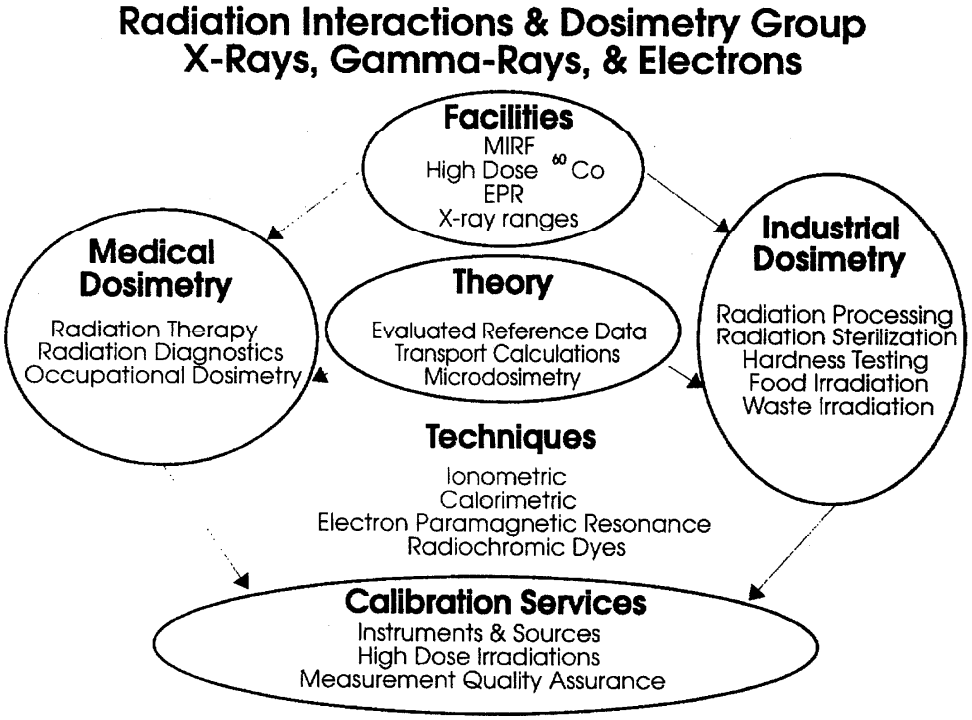

Fig. 1. Radiation facilities and support groups at NIST for medical and industrial dosimetry applications, techniques, and calibration services.

RECENT ADVANCES IN DOSIMETRY SYSTEMS

"Dosimetry systems" implies not only the radiation sensor itself but also the analytical methods that relate reproducibility of the radiation-induced signal to the absorbed dose at a location in a given material. There are important trends in enhancements of existing high-dose dosimetry systems (e.g., radiochromic films, reference and routine calorimeters, solid-state dosimeters, and chemical solutions) or novel dosimeters, under development, such as those that can be used and reused after "annealing" or by "real-time" measurement (diamond detectors, semiconductors, solid-state devices, amorphous ceramics, and fluorescent sensors) as well as remote-sensing systems (McLaughlin, 1990, 1991, 1993a). The increasing use of electron-beam and x-ray facilities demands more versatile, fast, and easily read systems, for simplicity and cost-effectiveness.

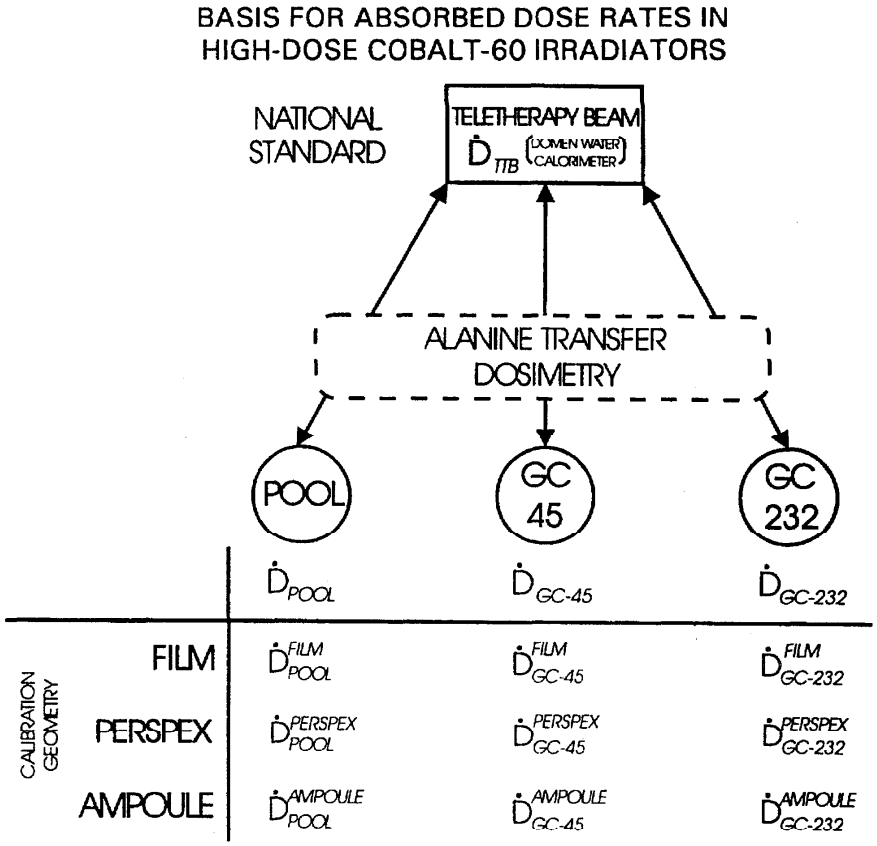

Fig. 2. Basis for traceability of absorbed dose rates at NIST in three gamma-ray sources for calibrating different dosimeter geometries with traceability through transfer alanine dosimeters to the national standards (water calorimeter).

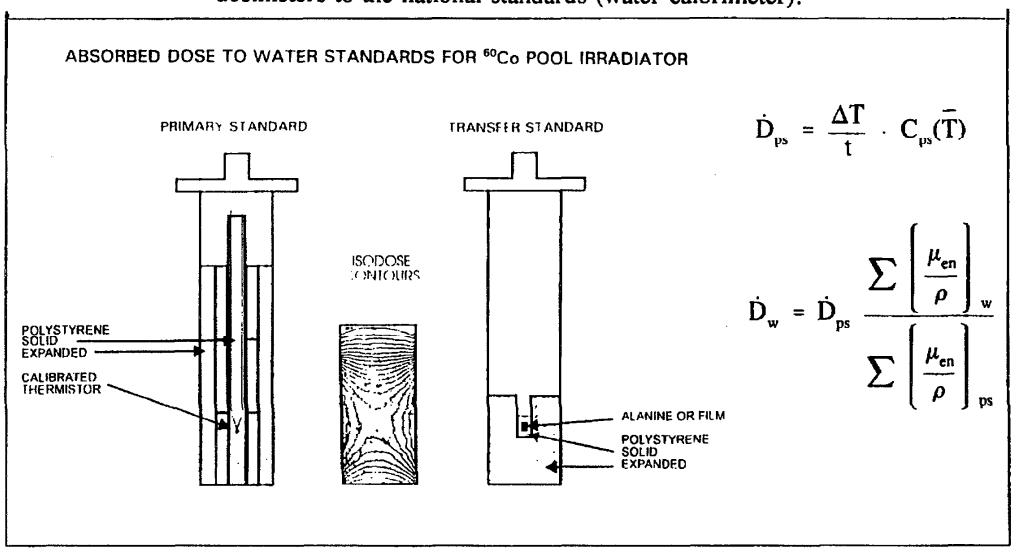

Fig. 3. Approach to transfer polystyrene calorimetry measurement to absorbed dose in water for alanine or radiochromic film irradiated with the NIST ^{60}Co gamma-ray pool source.

With the advent of less expensive and user-friendly read-out equipment, one of the most promising dosimeters, which may under careful preparation and calibration qualify as a reference dosimetry system, is *L-α-alanine* as measured by EPR spectrometry (Regulla *et al.*, 1993; McLaughlin, 1993b; McLaughlin *et al.*, 1993). It is also proving to be useful for transfer dosimetry, as shown by its application in the IAEA International Dose Assurance Service (IDAS) (Nam and Regulla, 1989). This dosimetry system is described in an accompanying paper of these proceedings (Desrosiers *et al.*, 1994).

Several aqueous or organic solutions are finding success as reference dosimeters for high-dose applications. Two of these systems, *ceric-cerous* aqueous acidic solutions (5×10^2 to 5×10^4 Gy) and *ethanol-chlorobenzene* solutions (10^2 to 10^5 Gy), can be used for both transfer and routine dosimetry, with readout by electro-chemical potentiometry of the ceric-cerous sulfate solution (the so-called "COMPU-DOSE System") (Doyle, 1980) or with readout by high-frequency potentiometry of the ethanol-chlorobenzene solution (HF "oscillometry") (Ražem *et al.*, 1985; Kovács *et al.*, 1985). It should also be pointed out that aqueous acidic *dichromate* solutions have proven to be one of the most successful reference transfer dosimetry systems for the sterilization dose range (10^3 to 5×10^4 Gy), as operated by NPL (Sharpe *et al.*, 1985).

Considerable advancements and developments are being made in several areas involving the use of mass-produced materials for routine dose measurements and for the mapping of dose distributions in radiation processing (McLaughlin, 1991, 1993a). Some of the most promising of these for high-dose dosimetry applications are as follows:

Diamond crystals and films. Nitrogen-doped synthetic diamond microcrystalline films or chips are useful as real-time dose-rate monitors for long-term use in radiation fields (Keddy *et al.*, 1988; Nam *et al.*, 1991). These systems consist of a conglomeration of microcrystalline material attached to electrical leads (e.g., fine gold wires), and, with a small DC voltage bias (up to 100 V), various radiation dose rates up to 10^3 Gy/min can be read in terms of direct-response current in the range pA to μA (typical dark current at 100 V bias is 5×10^{-12} A) with linearity between current and dose rate. At NIST, a single N_2-doped diamond detector with gold-wire connectors has been used as a continuous off-axis electron monitor (1 to 3 MeV) over a total dose estimated at 10^8 Gy with no appreciable loss or gain in sensitivity. One limitation of these detectors is the variability of sensitivity from one detector to another, which requires individual calibration of response. They also require a conditioning irradiation treatment of about 10 Gy before real-time use, and the temperature dependence of each specimen must be evaluated.

Lithium fluoride optical-quality crystals. One of the most useful dosimeters for measuring very large doses (10^4 to 10^7 Gy) is the colorless optical-quality, pure LiF crystal dosimeter, which usually consists of a polished single crystal having dimensions of 2 mm × 2 mm × 6 mm (Spindel *et al.*, 1994; Chen Fanxiu *et al.*, 1993). A series of electron-filled traps (as negative ion vacancy arrays) exists due to irradiation to very high doses. These occur as color centers, namely the M-center (443 nm absorption band) (dose range 10^3 to 10^5 Gy), the N_1 and N_2 centers (517 and 547 nm, respectively) (10^5 to 10^7 Gy), and an X-center (785 nm) (5×10^5 to 10^8 Gy). The radiation-colored LiF optical-quality dosimeter is measured spectrophotometrically at the appropriate wavelengths. The response shows no appreciable dose-rate dependence (10^0 to 10^{10} Gy s^{-1}) and only a small temperature dependence during irradiation (McLaughlin *et al.*, 1979), but there is appreciable instability after irradiation, for periods up to 24 h. The dosimeters after irradiation and readout may be annealed to a colorless state by an elevated-temperature treatment (1 h at 550 °C), and then reused over many irradiation-annealing cycles. As with the diamond detectors, each LiF specimen must be calibrated individually. The optical surfaces must also be protected against abrasion, scratches, and dust. Pre-irradiation readout and thickness measurement of each dosimeter are recommended.

Semiconductors. Silicon diodes and metal oxide semiconductor field effect transistor (MOSFET) detectors can be used either for "real-time" current reading (with or without DC voltage bias) as a function of absorbed dose rate, or for post-irradiation readout of integrated voltage changes at specified currents due to permanent radiation damage effects (Nablo *et al.*, 1994). A typical new MOSFET system has applications for relatively low doses in the range 0.1 to 200 Gy (blood irradiation or insect irradiation) and dose-rate independence up to 10^8 Gy s^{-1} (Holmes-Siedle and Adams, 1986; Holmes-Siedle *et al.*, 1992; Reece and Thomson, 1988). The system is also independent of relative humidity and can be used over a broad temperature range (0 to 50 °C). The integrated radiation effect that is used for the measurement is the shift in threshold voltage due to trapped charge in the multilayered device. This threshold voltage is evaluated in the measurement of the channel ("drain") current as a function of gate voltage at a constant supply voltage to the device.

Some encapsulated silicon diode detectors can also be used as integrating or dose-rate detectors at relatively low doses (10^0 to 10^4 Gy) and dose rates (up to 10^2 Gy s^{-1}) (Knoll, 1989). Permanent changes are measured after irradiation as radiation-induced increase in voltage at a constant reverse current (Holmes-Siedle and Adams, 1993).

New developments in radiochromic films. Major advantages of radiochromic films and plastic chips are their broad range of response (10^0 to 10^6 Gy), their wide availability in large, relatively inexpensive batches, and their ability to map both gamma-ray and electron dose distribution with high spatial resolution. Disadvantages of some of these systems include dependence of response on temperature and relative humidity during irradiation, certain variabilities of response at high dose rates, sensitivity to ultraviolet light in the case of some systems, and batch-to-batch variability of response characteristics in some cases.

One of the most promising new systems for dose mapping applications is the GafChromic™ type (Saylor *et al.*, 1988; McLaughlin *et al.*, 1991, 1994b), which is now supplied with two dose ranges of response, 10^0 to 10^3 Gy (GafChromic MD-55™) and 10^1 to 10^4 Gy (GafChromic DM-1260™). They are read spectrophotometrically at certain wavelengths corresponding to multiple broad radiation-induced absorption bands and on their shoulders, which allows dose readings over a relatively broad dose range. Figure 4 shows, for the more sensitive of the two, GafChromic™ film MD 55™, on the left, a series of absorption spectra for unirradiated and irradiated film, and, on the right, the response curve when measured at the indicated wavelength. The system exhibits a marked dependence of response on temperature during both irradiation and readout, but the dependence on relative humidity is small. The films are also considerably less sensitive to ambient ultraviolet light (e.g., white fluorescent light) than the widely-used Nylon-based radiochromic film types. New versions of these dosimeters are being developed to extend the useful dose ranges downward to 0.5 Gy and upwards to 5×10^4 Gy.

The radiochromic films are useful for measuring high-resolution absorbed-dose distributions of electron-beam profiles (see Fig. 5) and depth doses, as well as dose distributions in thin layers, such as on surfaces of irradiated materials. The orthographic and topographic contours in Fig. 5 are actually values of relative optical absorbance at 632.8 nm wavelength and are proportional to isodose contours, according to a film calibration of absorbance versus dose.

Another useful new radiochromic system is a colored polymethylmethacrylate (Perspex) dosimeter, referred to as GAMMACHROME YR™ (10^2 to 3×10^2 Gy) which upon irradiation changes in color from yellow to red (Whittaker, 1990). It is analyzed spectrophotometrically at 530 nm wavelength.

A color-changing film designed for a higher dose range (10^4 to 10^6 Gy) is a dye-coated cellulose diacetate film (DY-4.2™) (McLaughlin *et al.*, 1988a). This film is yellow and bleaches upon irradiation, with the radiation-induced decrease in optical density being measured by a

densitometer equipped with a broad-band pass filter (390 to 440 nm). This dosimeter shows a marked temperature dependence of response, and requires calibration of each batch.

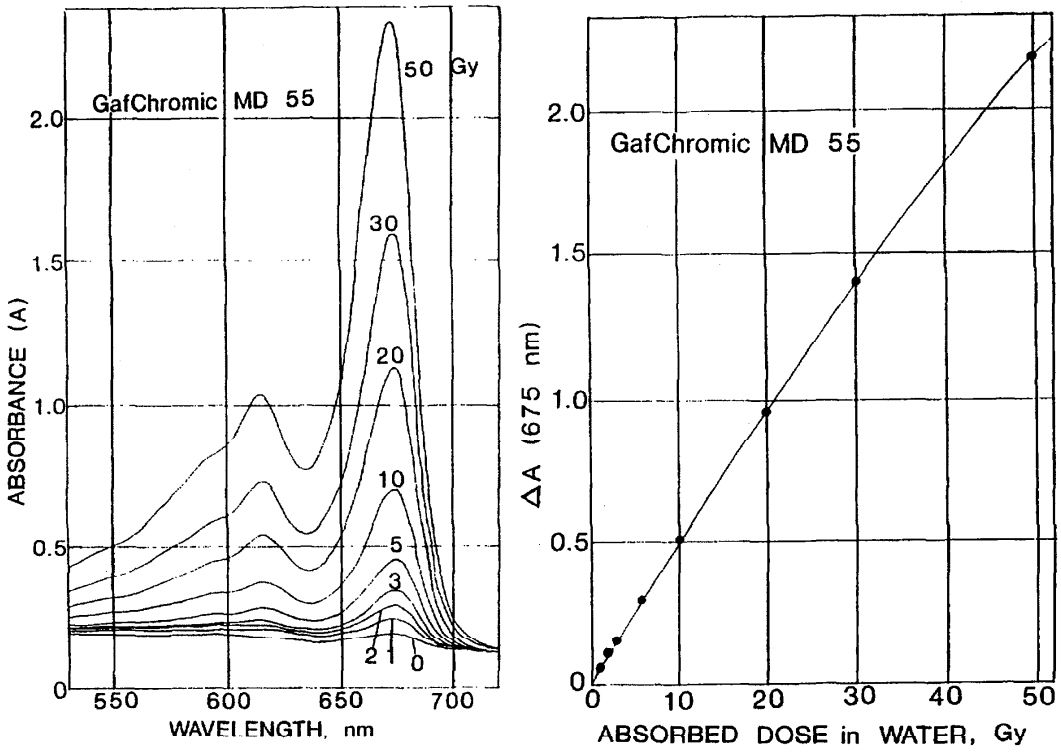

Fig. 4. Gamma-ray response of GafChromic dosimeter type MD-55™. *Left*: Absorption spectra of unirradiated and irradiated specimens. *Right*: Response curve measured at the absorption band peak.

A useful radiochromic system is based on a biological stain that when irradiated changes from the colorless form (triphenyl tetrazolium salts) to the red-colored dye (triphenyl formazan) (McLaughlin *et al.*, 1991). It can be used in liquid solutions (alcohol solvents) or in thin films (polyvinyl alcohol) (Kovács *et al.*, 1994).

An important advancement supplemental to the well-established Nylon-base radiochromic film, traditionally referred to as FWT 60-00™, is the new form called FWT-460™, with the addition of a background yellow color (Rickey and Humpherys, 1993a; Rickey *et al.*, 1994; McLaughlin *et al.*, 1994c). This uniform stable color has an absorption peak (430 nm) away from the blue color absorption band (600 nm) induced by irradiation, so that spectrophotometry at the two wavelengths after irradiation makes unnecessary the pre-irradiation read-out of optical density and obviates the need for film thickness measurement. The response range for this film is 3×10^3 to 6×10^4 Gy. Another new radiochromic film for very high doses (10^4 to 5×10^5 Gy) consists of pararosaniline cyanide and *p*-nitrobenzoic acid in polyvinyl butyral (Uribe *et al.*, 1993).

A novel approach to real-time radiochromic dosimetry is the use of laser telemetering to analyze remotely radiation-induced changes in dosimeter transmitted or reflected color (Walker *et al.*, 1990).

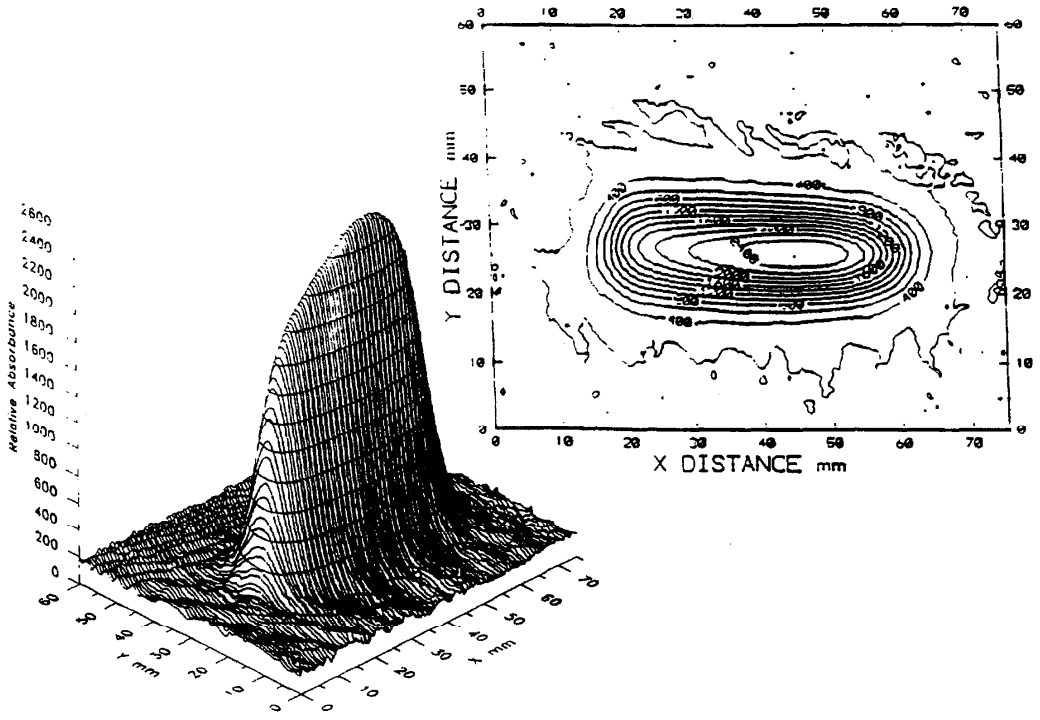

Fig. 5. Dose distribution across the profile of a 10-MeV electron beam at oblique incidence on a calibrated radiochromic dosimeter film held in polystyrene slab phantom as measured by a laser-scanning microdensitometer (Walker *et al.*, 1992). *Top*: Orthographic projection of the isodose contours as computed from the radiochromic image profile using SURFER™ software. *Bottom*: Topographic isodose contours of the beam image.

Optical waveguide dosimeters. Two types of radiochromic liquid-core optical waveguide dosimeters are especially useful for food irradiation dosimetry McLaughlin *et al.*, 1989b; Rickey and Humpherys, 1993b). These devices consist of light-transmitting cores of a radiochromic dye-gel solution in a polymer tubing (5-cm long and 0.25-cm outer diameter), sealed at both ends by lens-like glass beads. These dosimeters are read-out in terms of increase in absorbance, A (optical density), at a given optical wavelength, λ, ΔA_λ, as a function of absorbed dose, D, using a specially-designed "optichromic" reader (densitometer) with different wavelength settings. The absorbed dose ranges are specified between 10 and 10^4 Gy, depending on dosimeter type and the analytical wavelength. Although humidity dependence is not a problem, there is temperature dependence during irradiation that must be taken into account (McLaughlin *et al.*, 1989b).

SUMMARY

Improvements in existing high-dose dosimeters and systems now under development should advance the use of reference, transfer, and routine measurements for validation verification, and for quality control of radiation processing. Calorimeters are proving to be valuable both as beam

monitors and beam-calibrating standards, as well as devices for calibrating dosimeter response to electron beams in the energy range 1 to 10 MeV. EPR spectrometry of several materials, in which stable free-radicals are formed, has become a useful method for reference transfer dosimetry and for regulatory purposes over broad dose ranges. Certain aqueous and organic solutions are also well-established reference-transfer dosimeters.

A number of new developments in several types of routine dosimetry systems (real-time or integrated) are now becoming established. It is encouraging that some of these advances may help simplify, improve, and make less expensive the day-to-day, routine, radiation processing plant dosimetry, as well as the process-validation and dose-mapping procedures.

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