PROCESS CONTROL AND DOSIMETRY APPLIED TO ESTABLISH A RELATION BETWEEN REFERENCE DOSE MEASUREMENTS AND ACTUAL DOSE DISTRIBUTION

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Abstract. The availability of the first commercial dose level indicator prompted attempts to verify radiation absorbed dose to items under quarantine control (eg for insect disinfestation) by some indicator attached to these items. Samples of the new commercial dose level indicators were tested for their metrological properties using gamma and electron irradiation. The devices are suitable for the intended purpose and the subjective judgement whether the threshold dose was surpassed is possible in a reliable manner. The subjective judgements are completely backed by the instrumental results. Consequently, a prototype reader was developed; first tests were successful.

The value of dose level indicators and the implications of its use for food or quarantine inspection depends on a link between dose measured (indicated) at the position of such indicator and the characteristic parameters of the frequency distribution of dose throughout the product load ie a box or a container or a whole batch of multiple units. Therefore, studies into variability and statistical properties of dose distributions obtained under a range of commercial situations were undertaken. Gamma processing at a commercial multipurpose contract irradiator, electron processing and bremsstrahlung applications at a largescale research facility were included; products were apples, potatoes, wheat, maize, pistachio. Studies revealed, that still more detailed information on irradiation geometries are needed in order to render meaningful information from dose label indicators.

1. INTRODUCTION

The final goal of this CRP is to establish a reliable link between a radiation absorbed dose documented outside a container and the dose distribution effected throughout the product load contained therein. The availability of such information is thought to facilitate international trade in radiation processed food. This aspect is extremely important for example in instances where the objective of radiation processing is to fulfill quarantine requirements as for fresh and dried fruits and for tree nuts. For reliable disinfestation, the insects may survive a radiation treatment and may even be able to fly but must not be capable of proliferation. The necessary radiation dose for fruit fly eradication is typically 150 Gy; however, to prevent adult emergence altogether, including the emergence of normal appearing, sterile adult insects capable of flight the generic minimum radiation dose should be 250 Gy for any species of fruit flies. Thus, the achievement of a minimum dose once set by authorities is crucial and its verification is indispensable.

Such information attached to the container could be associated, of course, with the shipping documents; preferably it could be a 'label dosimeter' (not a label indicator) permanently affixed to the container before radiation processing. This dosimeter should directly indicate the dose figure or should also be readable by some hand-held instrument. Both types are likely to be available in the near future; one prototype was included in the programme of this CRP.

The main effort of this contribution was devoted to characterization of dose distributions likely to occur under commercial practices for fruit, dried fruit and nut disinfestation by radiation processing. During the course of this CRP some label dosimeters became available and studies into their properties were added; finally, a prototype reader for such labels was developed. Thus, Part B and C below is only a supplement to the main study; a paper on the validation of some label dosimeters by subjective and objective means was already published elsewhere. Preliminary results had already been reported by participants to the Second Research Coordination Meeting of this CRP in 1996.

2. MATERIALS AND METHODS IN GENERAL

Irradiation was done with a gamma cell (cobalt-60, dose rate 0.5 kGy/h) and a linear accelerator for electrons (indirect mode (microwave) 10 MeV, 10 kW, instantaneous dose rate 10^8 Gy/s, pulse duration 12 ìs; bremsstrahlung (X rays) at 5 MeV, dose rate about 10^4 Gy/s; average dose rates depending on scanning width, repetition rate and conveyor velocity). For experiments on larger scale a commercial gamma facility was available. Treatment was always at ambient temperature and humidity. In order to cover the full dose range and sensitivity of available dosimetry films (GAFchromic DM100 and FarWest FWT6000, sealed in paper/plastic envelopes) the target doses were chosen accordingly (eg minimum dose at 5 kGy); the results can easily be transformed to the dose range of interest (eg 250 Gy) as the geometry and, hence, the dose patterns and the relative dose distribution are not affected by setting of exposure time. Film readings were taken with a colorimeter (transmission mode, CIBA CORNING 257 or FarWest Radiachromic Reader) and filters for the appropriate wave lengths. Reference dosimetry for film calibration was done by use of Fricke dosimeter cross-checked by alanine dosimetry (IDAS) at accelerator and gammacell. In the experiments in cooperation with a contract irradiator Harwell Amber 3042 and spectrophotometer readout were applied in addition to our own systems.

Also, in order to study a larger variety of geometries and bulk densities wheat, maize and potatoes were included as models for fresh and dried fruits as well as tree nuts; one study included apples on commercial cardboard trays stacked on standardized pallets; the main studies were executed on pistachio in commercial cardboard boxes containing 40 or 48 pouches, respectively, 125 g each.

SAS (Statistical Analysis System, Carry, USA) was applied for analysis of data and graphical presentation of results.

3. PART A: GENERAL ASPECTS OF PROCESS CONTROL AND DOSIMETRY

For their purposes, the radiation processing industry has already established and standardized the practice of dose mapping and process validation for each individual item accepted for treatment. This, together with records of process control measures taken, of dose measurements executed during the process, and of observations at critical control points established for the process, renders a bulk of data on random fluctuations of the process. In commercial radiation processing it is to be expected that repeating loading patterns occur regularly which can be characterized for all random fluctuations which are likely to occur and can be related to achievable dose distributions and to the measured dose at some established reference position. A study of such informations can reveal whether verification of absorbed dose administered is possible and reliable in trade. As a model, certain geometries (source product geometries) were studied for electron, bremsstrahlung and gamma ray processing.

3.1. Materials and methods

3.1.1. Gamma ray processing and products irradiated

In cooperation with a contract irradiator, a multipurpose type facility designed for source overlap was used. It could hold two pallets (one in top of the other) of dimensions 1.8 m high, 1.2 m wide and 1.0 m deep (or 2.16 m^3). The carriers moved through 4 positions around the source and were irradiated from 4 sides. Consequently, for some loading patterns the position of the minimum dose values is to be expected in the interior and not at the surface (as for other designs of irradiation facilities); this implies that the expected position of the minimum dose is not accessible for measurements during production runs. The carrier was loaded with one pallet containing 1.7 t of maize and a second pallet containing 0.5 t of apples. As the residence time had to be equal for both loads, no target dose was preset for each treatment; instead, a nominal dose suitable for both dosimeter types utilized in this study was chosen. Apples came on commercial cardboard trays and were stacked into the carrier; 4 trays per layer and 12 layers high (overall density 0.21 g/cm^3). Maize was filled in cardboard boxes ($20 \times 30 \times 40$ cm^3), 8 boxes per layer and 8 layers high (overall density 0.80 g/cm^3). Apples were in the upper, maize was in the lower hold of the carrier.

3.1.2. Electron/bremsstrahlung processing and products irradiated

The electron accelerator of the institute (technical details see above) was used in direct and in bremsstrahlung conversion mode. The products were usually put on aluminium trays (40 cm by 40 cm), the filling height depending on product density (ie 4.5 cm height for water equivalent material and 10 MeV electron treatment). Wheat and maize were filled in cardboard boxes fitting on the trays of the conveying system (40×40 cm^2 base area, layer thickness of 5.5 cm, corresponding to the range of 10 MeV electrons at a bulk density of about 0.8 g/cm^3). This arrangement was also intended to simulate a possible bulk flow processing, ie a continuously flowing bed (40 cm wide, about 5.5 cm high) in a direction perpendicular to the beam. Potatoes were placed on the trays in a single layer about 4 cm high for one-sided and in a double layer about 7 cm high for double-sided irradiation. In the latter arrangement, vacuum packaging in plastic bags was applied in order to secure maintenance of positions during turning of the stack in double-sided irradiations. For pistachio commercial size boxes ($15 \times 25 \times 40$ cm^3) were used containing 40 bags, 125 g each in 4 layers with two rows (the height of 15 cm at a density of 0.35 g/cm^3 is slightly more than the range of 10 MeV electrons); double-sided irradiation was applied for 10 MeV electrons and single-sided irradiation for X rays. In the repetition experiment on pistachio commercial size boxes ($17 \times 27 \times 40$ cm^3) of another supplier were used containing 48 bags, 125 g each in 6 layers with two rows (the height of 17 cm at a density of 0.33 g/cm^3 is equivalent to the previous series of experiments with regard to electron penetration).

3.1.3. Dosimetry

All dosimeters were placed strategically in order that each represented equal surface areas, volumes or masses, depending on whether the treatment purpose was surface or volume irradiation. This was achieved by placing dosimeters in the centre of each subvolume they represented for bulk goods or by placing 4 dosimeters in equal distances to each other on the surface of each tuber or fruit. In the case of the commercial gamma-irradiation facility, the operator in addition to the experimental dosimetry used his standard dosimetry system at the established dose mapping positions. Depending on the dosimetry system used, target dose and exposure, respectively, were chosen accordingly in order to use the full range of sensitivity of the particular dosimetry system.

3.1.4. Ddata analysis

Scale transformation and comparison with the Gaussian distribution was used to visualize inherent properties of the respective frequency distributions. 'Standardized normal distribution' is always characterized by a mean of zero and a standard deviation of one; thus, size of the standard deviation in relation to the mean value (ie 'standard error') does not play any role. Cumulative frequency distribution is the integral of the Gaussian bellshaped curve and shows a typical sigmoid pattern. Once the cumulative frequency is converted into probit units this sigmoid curve is converted into a straight line. By this scale transformation any deviation of the measured distribution from the normal distribution becomes more obvious. For ease of understanding the vertical scale in probit units is labelled instead with the corresponding percent cumulative frequency. In the horizontal grid, dotted lines parallel to the 50% or meanline indicate 1, 2 and 3 standard deviation widths around the mean. The horizontal dose scale is easily rescaled from standardized units to the real dose values and equal standard deviations then are represented by equal slopes.

Calculated frequency distributions in these experiments are area, volume and mass density functions. For gamma processing each dosimeter represented 9.7 kg of a total of 1,700 kg of maize and each set of 4 dosimeters represented (the surface of) 2.6 kg of a total of 500 kg of apples. For maize the dosimeters were put in gelatine capsules and centred for the volumes they represented during filling of the boxes. For apples the 4 dosimeters of a set were attached to the surface of the fruit in equal distance to each other, 4 apples prepared this way at representative positions on each tray; the individual orientation of the spiked fruit was at random, the main portion of the apples remaining undoped with dosimeters. For electron processing each dosimeter represented 49 g of wheat and 51 g of maize, respectively. The dosimeters were placed in 5 layers in 1 cm distance, 25 dosimeters in a five by five arrangement per layer. For potatoes 4 dosimeters were attached at equal distances on the surface of each tuber, arrangement and orientation of the individual tubers on the tray was at random. For pistachio (except the dosemapping study) dosimeters were placed at the centre of one side of each bag and on the upper and lower inner surface of the cardboard box. All electron treatments were from top, for double-sided treatment the boxes were turned around an axis parallel to the direction of the scan.

3.2 Results and discussion

3.2.1. Gamma processing of maize and apples

Both dose distributions for maize (Fig. 1) and apples (Fig. 2) irradiated at the commercial facility show great similarity and do not deviate too much from the normal distribution. The strategy in placing dosimeters was different for the experiments reported here (FRCN in tables) and for the mapping effort by the contract irradiator. This is reflected most prominently by the differences in the reported mean dose and extreme values for both experiments. The rather high max/min dose ratio for maize (Table 1) is caused by the intentional overloading of the carriers; partial loading as usual practice would have resulted in a value below 2. The difference of results for maize compared to apples is easily explained by the difference in bulk density (0.8 to 0.2 g/cm^3) and equal exposure to the radiation field.

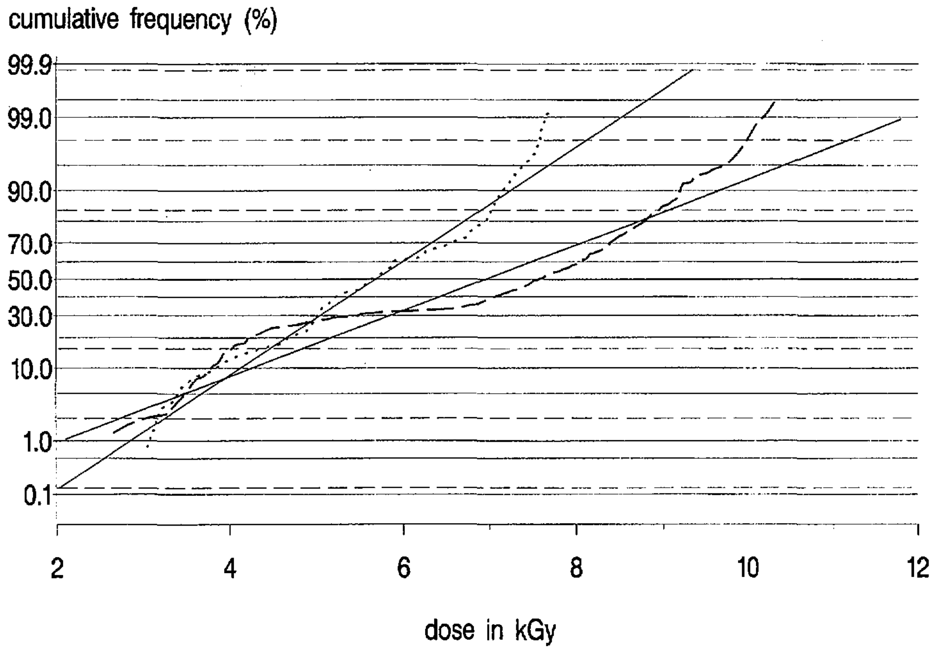

FIG. 1. Gamma processing of maize: frequency distribution of dose determined by FRCN (dotted line) and contract irradiator (dashed line); fine vertical lines: respective mean values.

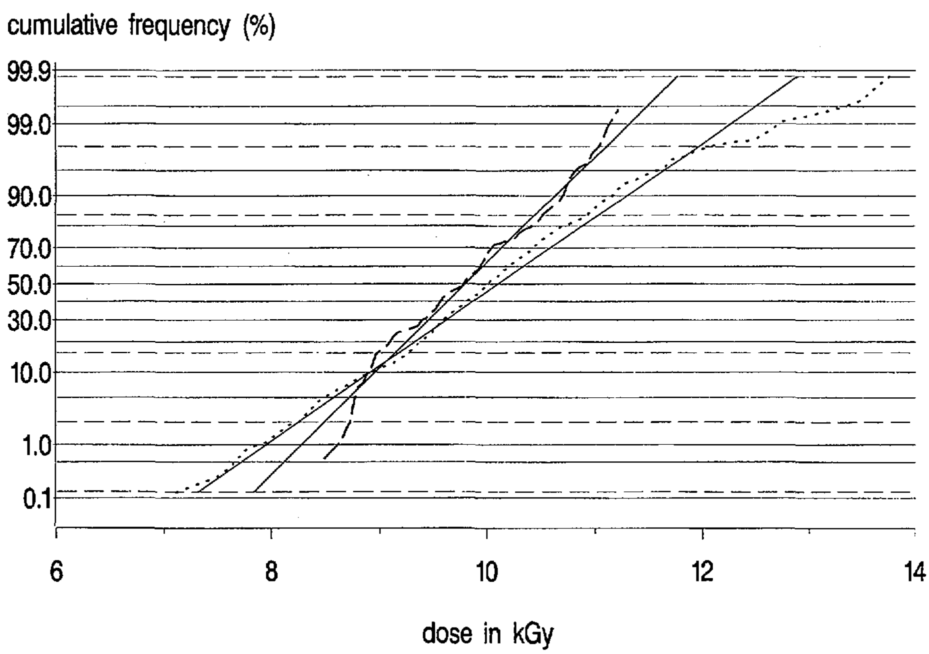

FIG. 2. Gamma processing of apples: frequency distribution of dose determined by FRCN (dotted line) and contract irradiator (dashed line); fine vertical lines: respective mean values.

TABLE 1. CHARACTERISTICS OF DOSE DISTRIBUTION IN GAMMA PROCESSING (dose in kGy)

	mean	std.	min	max	max/min
maize by FRCN	5.68	1.22	3.06	7.84	2.56
by contract irradiator	6.95	2.10	2.70	10.4	3.85
apple by FRCN	9.79	0.652	8.50	11.2	1.32
by contract irradiator	10.1	0.927	7.10	14.2	1.97

3.2.2. Electron processing of wheat, maize and potatoes

Dose distributions for wheat and for maize (Fig. 3) are very similar (Table 2); this was to be expected as geometry (size of boxes and density) were practically identical. The extremely high ratio of max/min dose is caused by the limited range of electrons. For improved homogeneity only layer thickness of about 37 mm instead of the 55 in these experiments would be acceptable. For potatoes (Fig. 4 and Table 3) the relative width of the dose distribution (standard deviation divided by mean dose) is significantly reduced by double-sided irradiation. In order to match the sensitivity of the dosimeter films used the target (surface) dose for all accelerator settings was chosen at 1 kGy, this resulted in higher minimum doses as needed for sprout inhibition. For this reason and for comparison all data were normalized to an equal minimum dose of 0.1 kGy also. The max/min dose ratios (Table 3) show that with 10 MeV electrons processing of a product like potatoes is not possible within acceptable dose ranges; even two-sided irradiation resulted only in insufficient improvement. For example, treating fruits for insect eradication with the recommended dose of 0.3 kGy would result in a maximum dose of about 4.5 kGy which would cause severe radiation damage for various fruits.

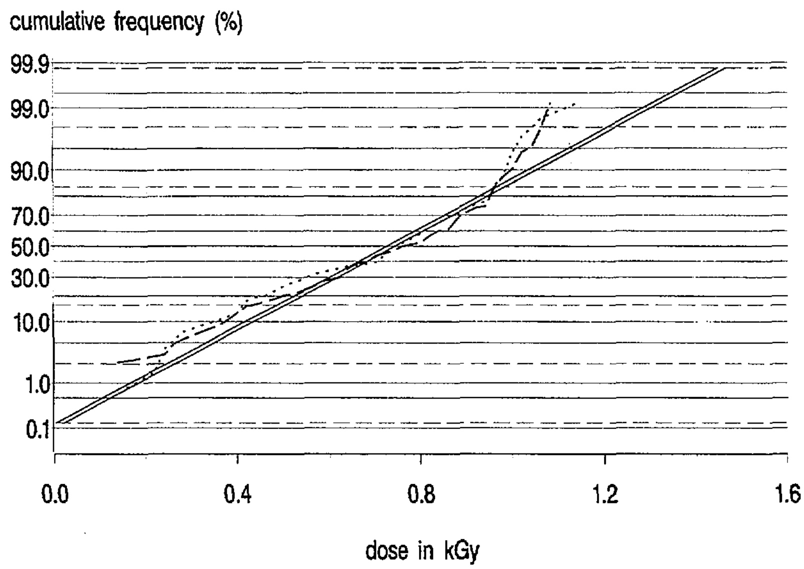

FIG. 3. Electron processing of wheat (dashed line) and maize (dotted line): 10 MeV electrons, single-sided irradiation; fine vertical lines: respective mean values.

TABLE 2. CHARACTERISTICS OF DOSE DISTRIBUTION IN SINGLE-SIDED ELECTRON PROCESSING OF MAIZE AND WHEAT (dose in kGy)

	mean	std.	min	max	max/min
maize	0.743	0.240	0.141	1.15	8.16
wheat	0.725	0.240	0.178	1.19	6.68

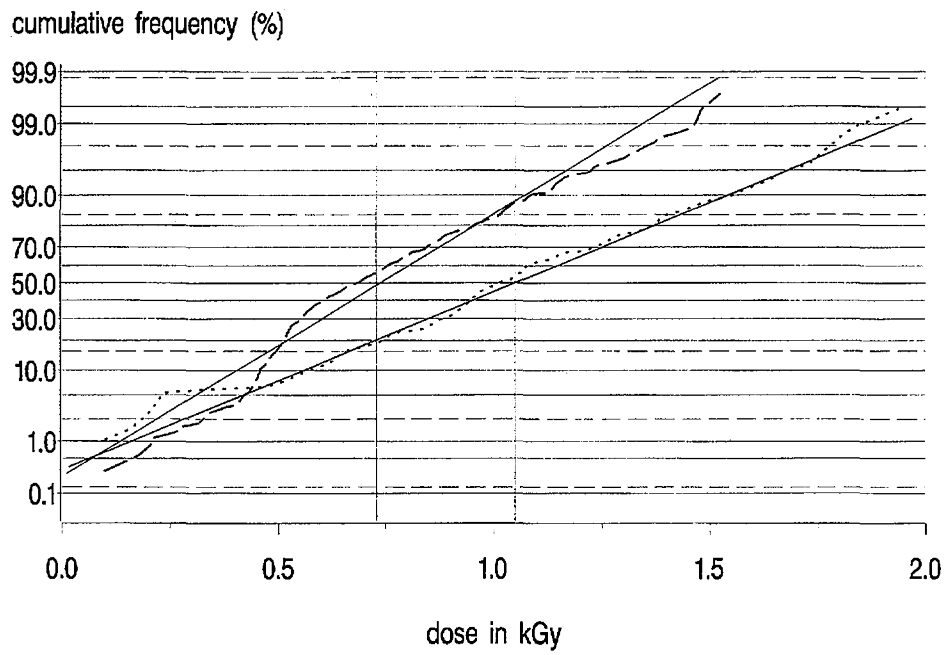

FIG. 4. Electron processing of potatoes: 10 MeV electrons, single-sided (dotted line) and double-sided (dashed line) irradiation; fine vertical lines: respective mean values.

TABLE 3. CHARACTERISTICS OF DOSE DISTRIBUTION IN SINGLE AND DOUBLE-SIDED ELECTRON PROCESSING OF POTATOES (dose in kGy)

	mean	std.	min	max	max/min
single sided	1.07	0.390	0.102	2.07	20.3
double sided	1.67	0.587	0.226	3.48	15.4

3.2.3. Eelectron and bremsstrahlung processing of pistachio

The characteristics of dose distribution for all three variants (including the repetition of the experiment with a larger number of passes and dosimeters) were similar (Table 4); single-sided electron treatment was not applied as commercial cardboard containers were too thick in beam direction for electron penetration (height 15 cm at density of 0.35 g/cm^3 results in an water equivalent thickness of 5.25 cm; practical range of 10 MeV electrons in water is 45 mm). Double-sided electron treatment is in general equivalent to one-sided bremsstrahlung treatment at either 5 or 10 MeV nominal energy. There is no significant difference between the two bremsstrahlung treatments. No extreme overdosing was observed at the surface due to any unfiltered low energy portion of the bremsstrahlung spectrum, the bremsstrahlung conversion target served as appropriate filter. The experiment on double-sided electron processing was repeated also with a larger number of items and dosimeters applied; the shape of the resulting frequency distribution of dose is still quite close to the normal distribution (Fig. 5) and only at the highdose side the practical upper limit for dose is encountered around 2.5 kGy.

TABLE 4. CHARACTERISTICS OF DOSE DISTRIBUTION IN DOUBLE-SIDED ELECTRON PROCESSING AND SINGLE-SIDED BREMSSTRAHLUNG PROCESSING OF PISTACHIO (dose in kGy)

	mean	std.	Min	max	max/min
10 MeV electrons	1.31	0.261	0.995	1.79	1.80
(repetition/normalized)	1.62	0.227	1.00	2.50	2.50
5 MeV X rays	0.793	0.121	0.661	1.04	1.57
10 MeV X rays	0.818	0.123	0.653	1.03	1.58

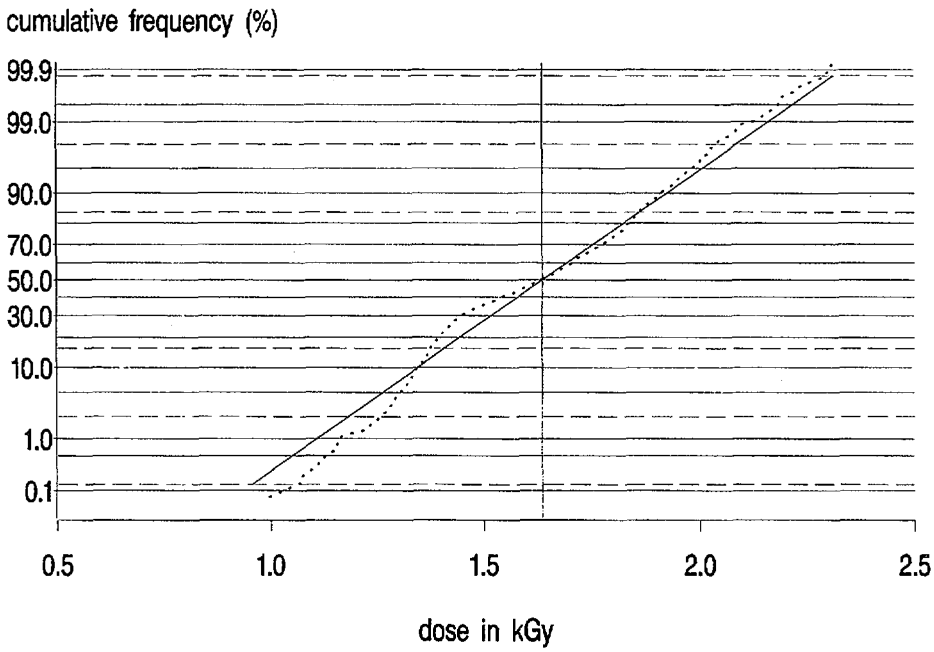

FIG. 5. Electron processing of pistachio: 10 MeV electrons, double-sided; fine vertical line: mean value (experiment with 23 repetitions and 1400 individual dosimeter readings.)

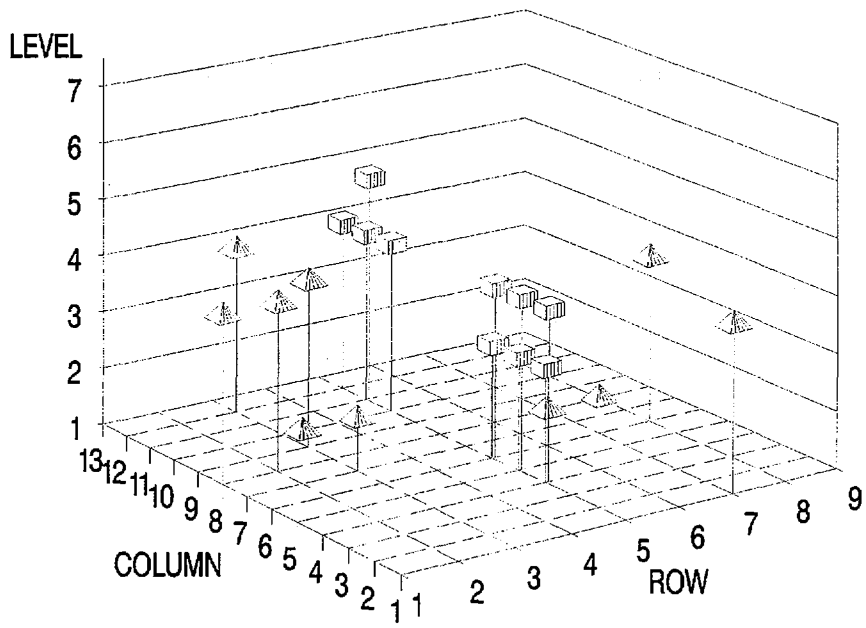

FIG. 6. Position of the 10 largest and of the 10 smallest dose values for 10 MeV electron processing (double-sided) for the repetition experiment on pistachio (dose mapping by 819 individual dosimeters): level of occurrence by row and column.

The experiment on double-sided electron irradiation was repeated in a set of 23 runs and a dose mapping run in the same geometry as described before. 6 layers of pistachio pouches allowed for 7 levels of dosimeter placement. Each layer consisted of two rows and 4 positions (resp. 9 rows and 13 positions in the mapping experiment) for the dosimeters. The dose mapping study revealed (Fig. 6) that the maximum dose mainly occurred in the centre of the boxes and along the direction of conveyor movement; the minimum doses also occurred mainly inside the box and not at surfaces. This effect was not caused by any inhomogeneity of dose distribution in scanning direction, but is due to the geometrical arrangement of the pouches in the boxes: through the centre of the pouches the beam passes most mass and the minimum dose is likely to occur in the middle of its trace; between the two rows of pouches the edges overlap, less mass is encountered and the maximum dose is due to the two-sided overlay of the largely unattenuated beam. The repetition run on electron processing was at a higher nominal dose and, therefore, for direct comparison normalized to equal minimum dose of 1.0 kGy (Table 4). The fluctuations of the overall dose distribution (standard error 14%) can be compared with the results from the reference dosimetry (dosimeters on each box in the centre facing the source and replaced when turning the boxes for the second pass); the mean reading of the control dosimeters was 1.052 kGy (standard error 2.2%), the observed lowest dose value was 1.00 kGy, and all variations of dose were in the range above the required minimum dose. Consequently, for this particular irradiation geometry it may be concluded that the surface dose of a single pass is closely related to the minimum dose and about 5% above that value. In the case of electron processing the surface dose is a particularly suited quantity for reference as it is directly linked to machine setting, ie mean current, scanning width and transport velocity. The higher max/min ratio for the repetition reflects that the geometry of pouches in the cardboard boxes during the 23 repetitions fluctuated considerably. In order to save costs the pistachio boxes were reused several times; the dosimeters had to be inserted and retrieved for which purpose the stacked pouches had to be removed from the box and laid in again.

3.2.4. *General aspects and summary*

Under this CRP from the very beginning and with respect to its duration of only five years, it was agreed that it would not be possible to collect a body of data on dose distribution occurring under a variety of practical conditions and covering all relevant geometries large enough to serve as a foundation of sound inter and extrapolation of frequency distributions of dose and of reliable estimation of the parameters of such frequency distributions from a limited set of dose measurements at some reference positions. The resolution of this problem, however, was seen as a prerequisite for the use of 'label dosimeters' in quarantine inspection of produce disinfested by ionizing radiation. The data reported in this contribution and by other participants of the CRP show at least that such approach is possible in principle. The pistachio experiment reported above shows that for special geometries here double-sided electron irradiation of boxes with a height in beam direction equivalent to electron penetration and where the dose value at the reference position is rather close to the dose value at the expected position of the minimum dose such conclusions are possible and allowable. In many practical situations an accessible position to attach some label dosimeter would not be a suitable and reliable reference position for dose measurements. Dose fluctuations on most positions at the surface of containers for the majority of irradiation geometries might be too large to allow for reliable estimation of the characteristic parameters of the frequency distributions of dose belonging to the respective setups. This would be especially true for many sided irradiation when the reference dosimeter can not be retrieved between single passes through the radiation field.

4. PART B: CHARACTERIZATION OF A LABEL DOSIMETER

In principle, there are two types of label dosimeters: The first type could consist of a barcode the optical visibility of which changes with dose and, thus, could give the dose reading; it also could be a threshold indicator for the required minimum (or reference) dose. The second type could be any conventional dosimeter which can be read by a suitable meter while still on its place on the container. Especially dosimetry films which are already in common use could be read in reflectance mode. Also such threshold dosimeters could be judged upon visual appearance.

Label indicators as used by the radiation processing industry for visual aid in inventory control are often thought to measure dose or to ensure a certain minimum dose. However, such labels do not have any metrological property. Instead, stamping every item emerging from the irradiation chamber would have the same documentary character when linked beyond all doubt to the process records of the facility. Furthermore such indicators typically are attached to the product load at positions on the outer surface of the goods and the relationship between dose at such reference position and dose at the expected position of the minimum dose is not established generally. The problem remains to establish such a link which must also be acceptable to control authorities.

Label dosimeters, on the contrary, would be instruments measuring the dose or 'indicating' that a given threshold value of dose was surpassed at the position where the 'label' resides. The metrological properties of such devices with regard to the nominal dose threshold and the reproducibility of the indication was to be studied; environmental factors also should not be neglected. Insect eradication and quarantine regulations might become a challenging application of such 'label' devices. Especially in the light of the statistical approach taken above, the value of such devices remains disputable, even after they have been proven to be real dosimeters: Securing a given dose value at an easily accessible position outside a consignment not necessarily ensures at the same time that a minimum dose throughout the product load has been met even at positions not accessible for verification. To ensure that, other measures are indispensable.

4.1. Materials and methods

"STERIN 70", "STERIN 125", and "STERIN 300" 'indicators' were used, which were made available to members of this CRP by its manufacturer (International Speciality Products (ISP) Inc., Wayne NJ, USA; similar indicators with the trade name "RADSURE" are used to a certain extent by the blood banking community). Type '125' came with a clear release film on top of each which could be peeled to expose an adhesive and to attach the indicator on the inside of a clear container; all measurements were taken through that film without peeling it. Types '70' and '300' came without such release film on the surface.

STERIN 'indicators' have a sensitive red field which darkens to black with increasing dose. In this read field the text 'NOT' in black on the red background is seen for the unirradiated indicator; as the red background darkens at a certain dose level the 'NOT' can no longer be read. The nominal dose (ie 70, 125 or 300 Gy) is also imprinted on the label indicator. Thus, the inscription initially is 'NOT irradiated at nnn Gy' which changes to 'ÛÛÛ irradiated at nnn Gy' around the threshold dose.

Colour measurements were taken with a tristimulus colormeter (Minolta Chroma Meter II 'reflectance') in CIELab coordinates. Lab coordinates were preferred as these most closely reflect human perception of equal colour differences. Furthermore, Lab coordinates allow for illumination and viewing conditions. At the same time this property is also a limitation with regard to comparison between laboratories: illumination and viewing conditions are difficult to reproduce. The tristimulus reader used here had internal conversion of measurements using predetermined factors. The conversion to other colour coordinates as YXZ or Yxy systems is not straight forward and several sets of conversion equations have been published. Despite this deficiency the results reported in the CIELab coordinates are comparable between participating laboratories and the results are in general agreement.

Subjective judgements were taken by an untrained panel of 25 participants each asked to rank a set of sample indicators irradiated at several dose levels or to discriminate the odd sample of three indicators irradiated at two dose levels (one of the three being a replica of the two dose value presented). For ranking, sets of six label dosimeters each were presented to the judges in random order; each dosimeter of the six treated with 60, 80, 90, 100, 110 and 120 % of the respective nominal dose; the judges were requested to arrange the labels in order of blackening of the 'NOT'. In a series of five triangle tests two labels irradiated at 120 % of the respective nominal dose together with an odd sample treated at 60–110 % of respective nominal dose were presented and the test was to identify the odd sample. The odd sample of the triangle sets was always presented at random position in the triplet. The indicators were labelled by three digit random numbers in both tests in order to prevent prejudice from sample naming; the triangle sets were disclosed to the judges in a gambling manner in order to prevent recognition of the two (repeating) 120 % reference samples and their codes; this was repeated for the five sets. Testing for unbiased judgement (asking individuals who had never seen a label before to read the complete text on the label and subsequent evaluation of the answers whether the 'NOT' was recognized) was not considered feasible because of burden of such extensive work. Results of the research into validation by subjective and objective means for such labels was already reported elsewhere; only a condensed summary is given below.

4.2. Results and discussion

Disputable remains the value of such label dosimeters while they have been proven suitable and reliable by the studies during this CRP. Securing a given dose at an easily accessible position outside a consignment not necessarily ensures that the minimum dose is met throughout the product load and even at positions not accessible for verification.

All label indicators showed the obvious effect: the 'NOT' became invisible when the red field turned black after receiving the indicated threshold dose or more. This effect was followed up by colour measurements in reflectance mode and by visual judgments using a test panel. The colour change in the abplane of the CIEcolour space occurred on a straight line from higher chromaticity (upperright corner) with increasing dose to the neutral chromaticity point at the coordinates' origin for all three indicator types (Fig. 7). All types of indicators were irradiated with gamma rays and electrons (the latter not for "STERIN 70"). The length of the distance passed with increasing dose from the respective starting point of the unirradiated samples is used in the following graphs; the course of CIElightness (brightness) is also given (Figs 8–10). It can be seen for all three indicator types that distance and lightness approach their asymptotic values around the respective nominal dose value; both parameters separately may be used for discrimination. Therefore lightness is used for comparison with instrumental reading by a prototype 'dose indicator reader'.

The study also included environmental factors such as temperature and UV light; humidity effects were not investigated. Storage of STERIN 'indicators' at 25 and 40 °C as well as under UV light (366 nm) did not cause significant changes even over 100 h storage/exposure; the variations are within the reproducibility of the colour readings.

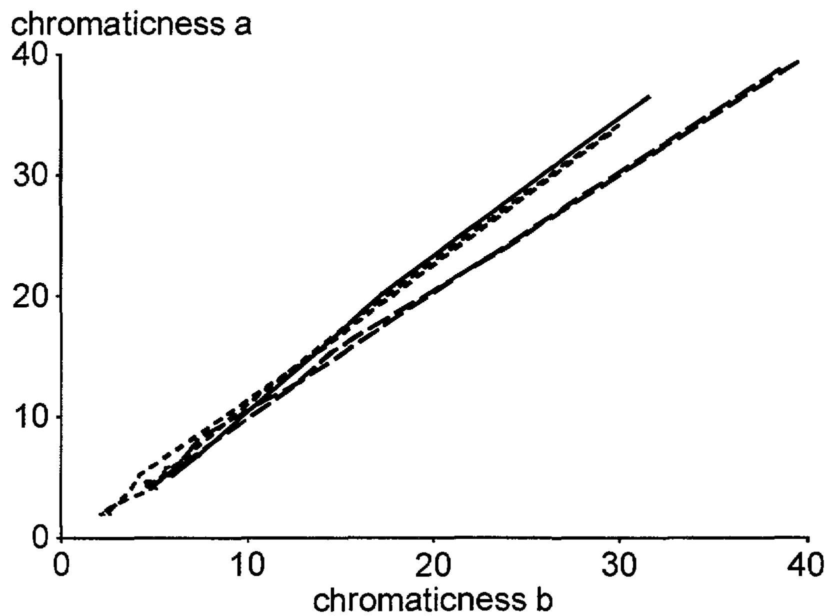

FIG. 7. Colour change for dose indicators, in CIEa and b chromaticity coordinates. increasing dose from upperright corner (red/yellow) to neutral at origin of the coordinate system (chromaticity direction to blue/green); indicator thresholds 70 Gy and 125 Gy (solid lines) and 300 Gy (dashed lines); electron and gamma irradiation (electron not measured for 70 Gy indicators) are given in identical line types as they fall close to each other.

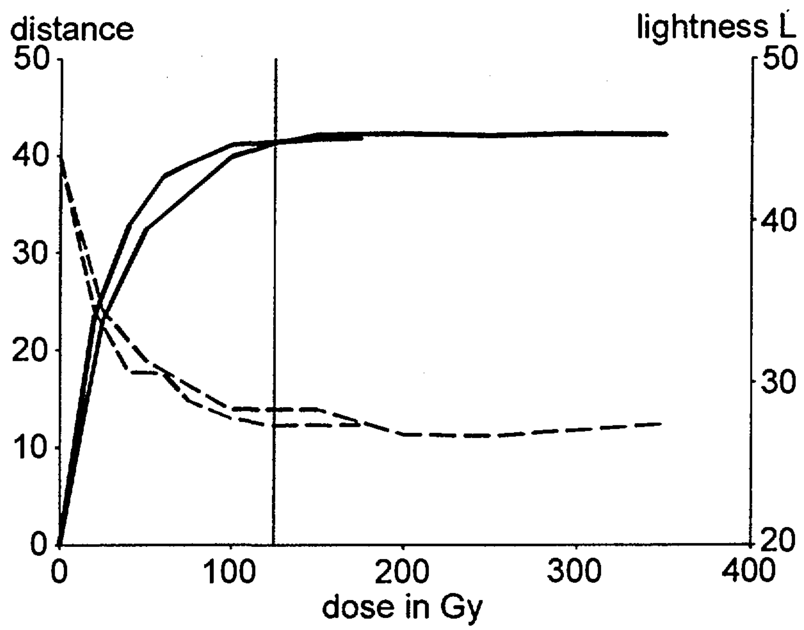

FIG. 8. As a function of dose, the arc length (see FIG. 7) traversed in chromaticity space (left scale, solid line) as well as the lightness in CIEL dimension (right scale, dashed line); treatment by cobalt-60 gamma rays; indicator threshold 70 Gy (fine vertical line).

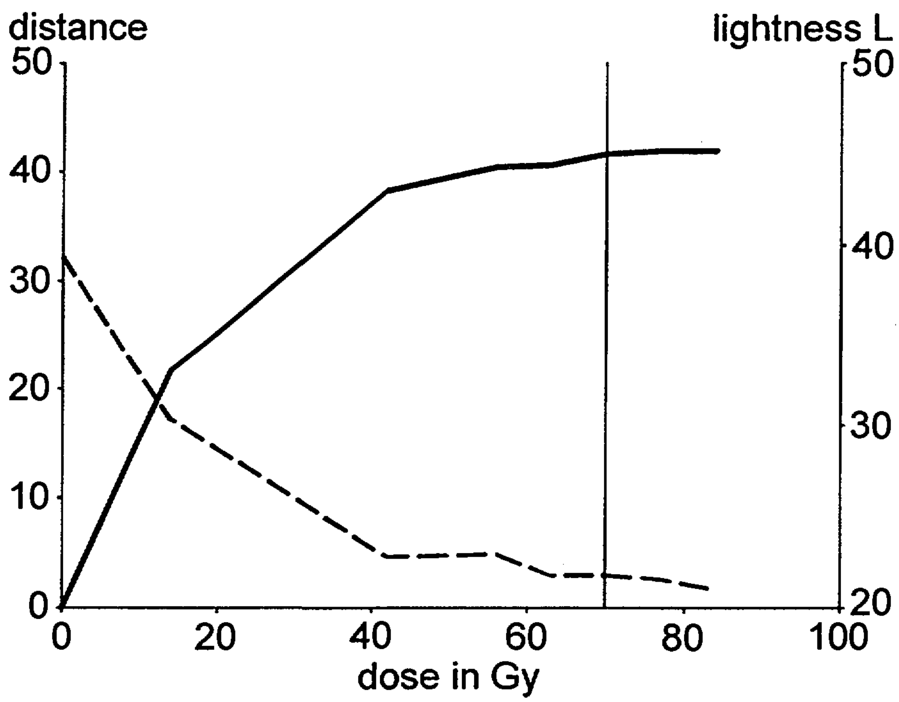

FIG. 9. As a function of dose, the arc length (see FIG. 7) traversed in chromaticity space (left scale, solid line) as well as the lightness in CIEL dimension (right scale, dashed line); treatment by cobalt-60 gamma rays and 10 MeV electrons, hence, two set of solid and dashed lines; indicator threshold 125 Gy (fine vertical line).

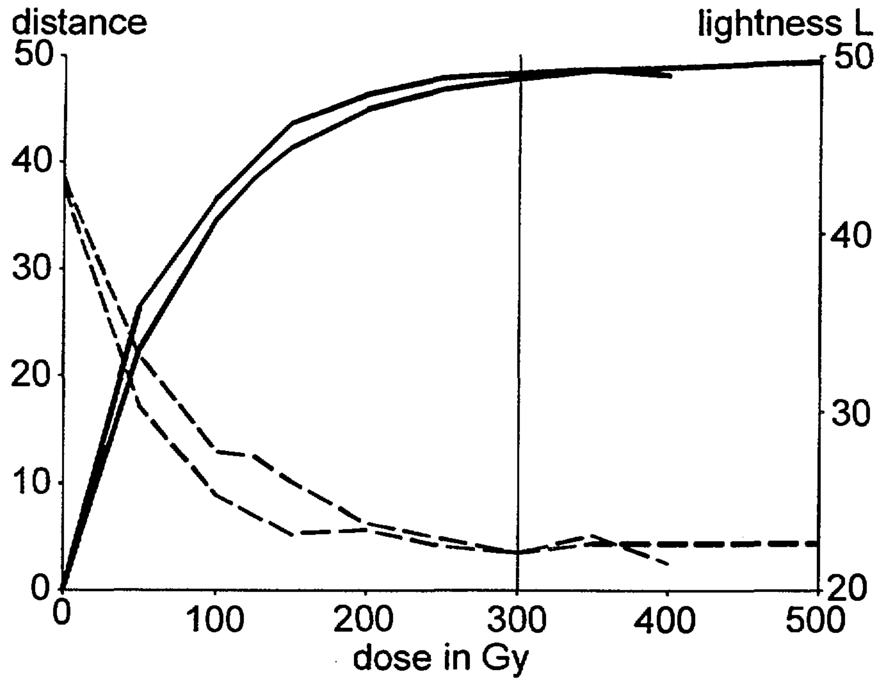

FIG. 10. As a function of dose, the arc length (see FIG. 7) traversed in chromaticity space (left scale, solid line) as well as the lightness in CIEL dimension (right scale, dashed line); treatment by cobalt-60 gamma rays and 10 MeV electrons, hence, two set of solid and dashed lines; indicator threshold 300 Gy (fine vertical line).

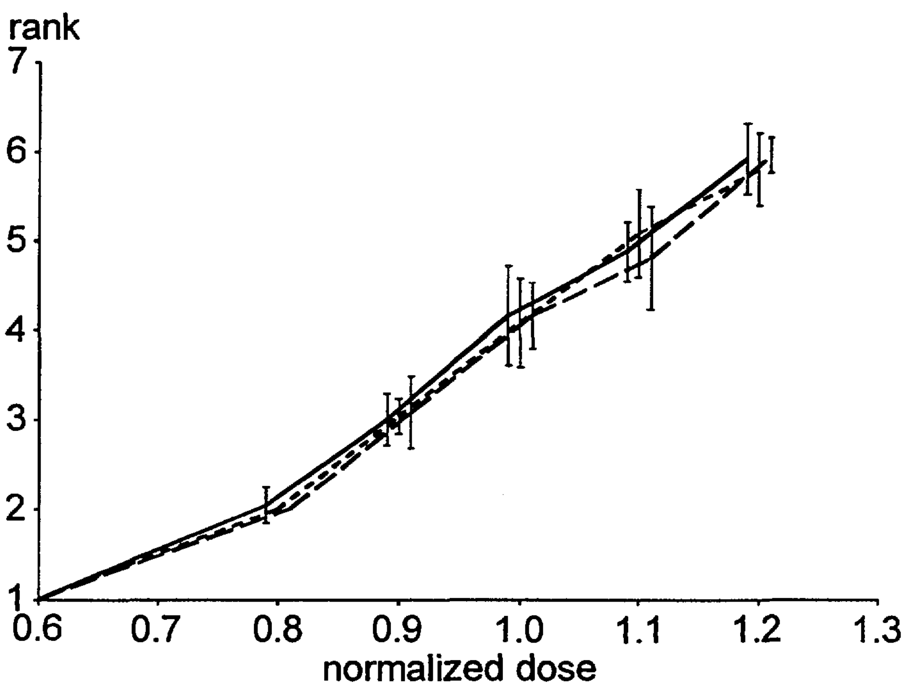

FIG. 11. Average ranks (25 judges); indicator threshold 70 Gy (solid line), 125 Gy (dotted line), 300 Gy (dashed line); 70 Gy and 300 Gy displaced to left and right, respectively, for better visibility; standard deviation indicated; dose normalized to indicator nominal dose of 70, 125 and 300 Gy.

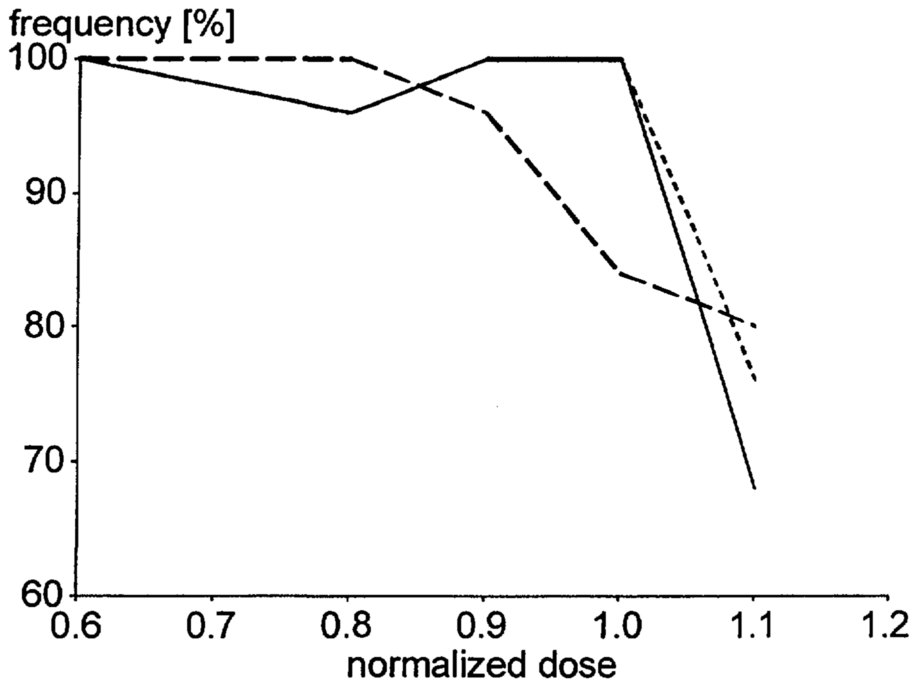

FIG. 12. Frequency of correct recognition of the odd sample (25 judges); line types for indicator nominal dose same as in FIG. 11; dose normalized to indicator nominal dose of 70, 125 and 300 Gy.

The results of the ranking test (Fig. 11) make obvious that the judges could discriminate and arrange the samples in a very reproducible manner; only directly neighbouring dose steps were sometimes arranged incorrectly which results in the rather small standard deviations for rank averages. Testing for least significant distances (á = 0.01, Duncan test) revealed that all dose steps are discriminated by their mean ranks for the three indicator types, respectively.

In triangle tests the guess probability is 1/3; if more than 17 judges out of 25 find the correct answer this is highly significant (á=0.01). A large group of judges was able to discriminate still 110 % against 120 % nominal dose for each type of labels (Fig. 12). For the labels with nominal dose of 300 Gy the decision dose is not as sharply defined as for the other two nominal doses. Practically, however, this implies that for type '300' only one judge out of 25 could not see for 90 % nominal dose the difference from the black reference ones; at 100 and 110 % of nominal dose this group increases to four to five judges among 25. All judges could see the 'NOT' being blackened away, but the shade of that black was still different enough between samples and only allowed for discrimination. However, not the shade of black but the readability of the 'NOT' is the validation criterion. It should be noted that the judges of the testpanel always tried to use any additional information (eg glossiness of label surface) for better discrimination and for improved performance in the sense of possibly expected answers.

5. PART C: DEVELOPMENT OF A LABEL DOSIMETER READER
(in cooperation with B. Bauer)

By subjective and objective judgements it could be shown that STERIN labels have suitable metrological properties; other participants of this CRP also contributed. Consequently, the idea was developed to design a simple, hand-held reader which could replace subjective judgements and which could be as reliable as a thorough spectrophotometric or colorimetric analysis. The principle approach was to replace prism, grid or filters as wavelength analysing instruments by diode illumination at suitable wavelength; 615 nm was chosen for the experimental setup (Fig. 13). The reflected light is then measured by a standard photodiode. Split glassfibre optics is used to illuminate and measure at the same spot. Simultaneously the type of the indicator imprinted close to the radiation sensitive window is read and all information are processed in some dedicated computer; the respective calibration function for several types of indicators may be selected. The whole instrument (Fig. 14) can be converted into miniature form or a hand-held device (Fig. 15). The indicator might be inserted in a reading slot or the reader might be put on top of the indicator still on its place on a carrier load.

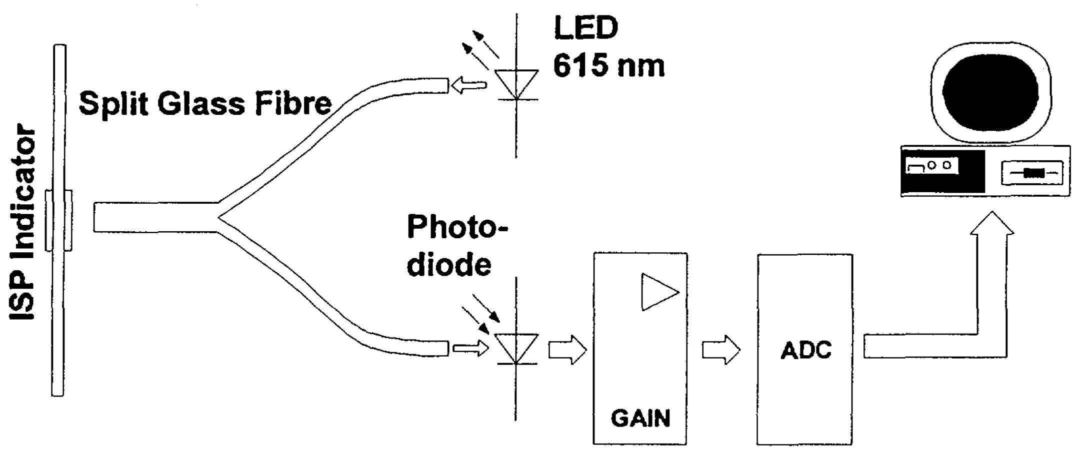

FIG. 13. Schematic diagram of the experimental setup for instrumental indicator readout.

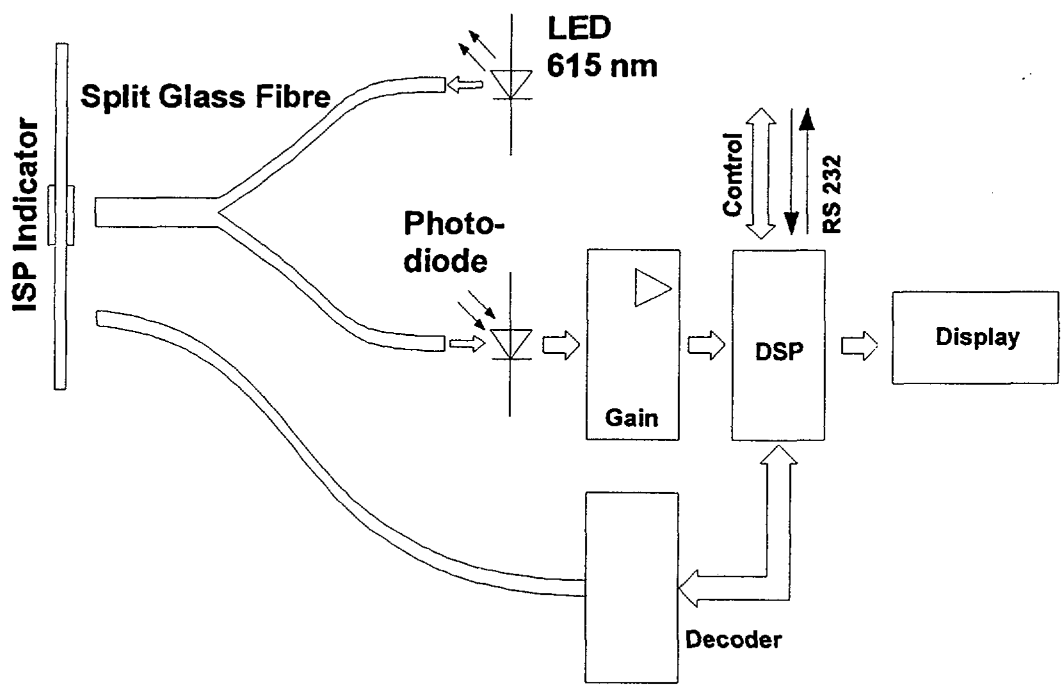

FIG. 14. Schematic diagram of the prototype reader for label dose indicators.

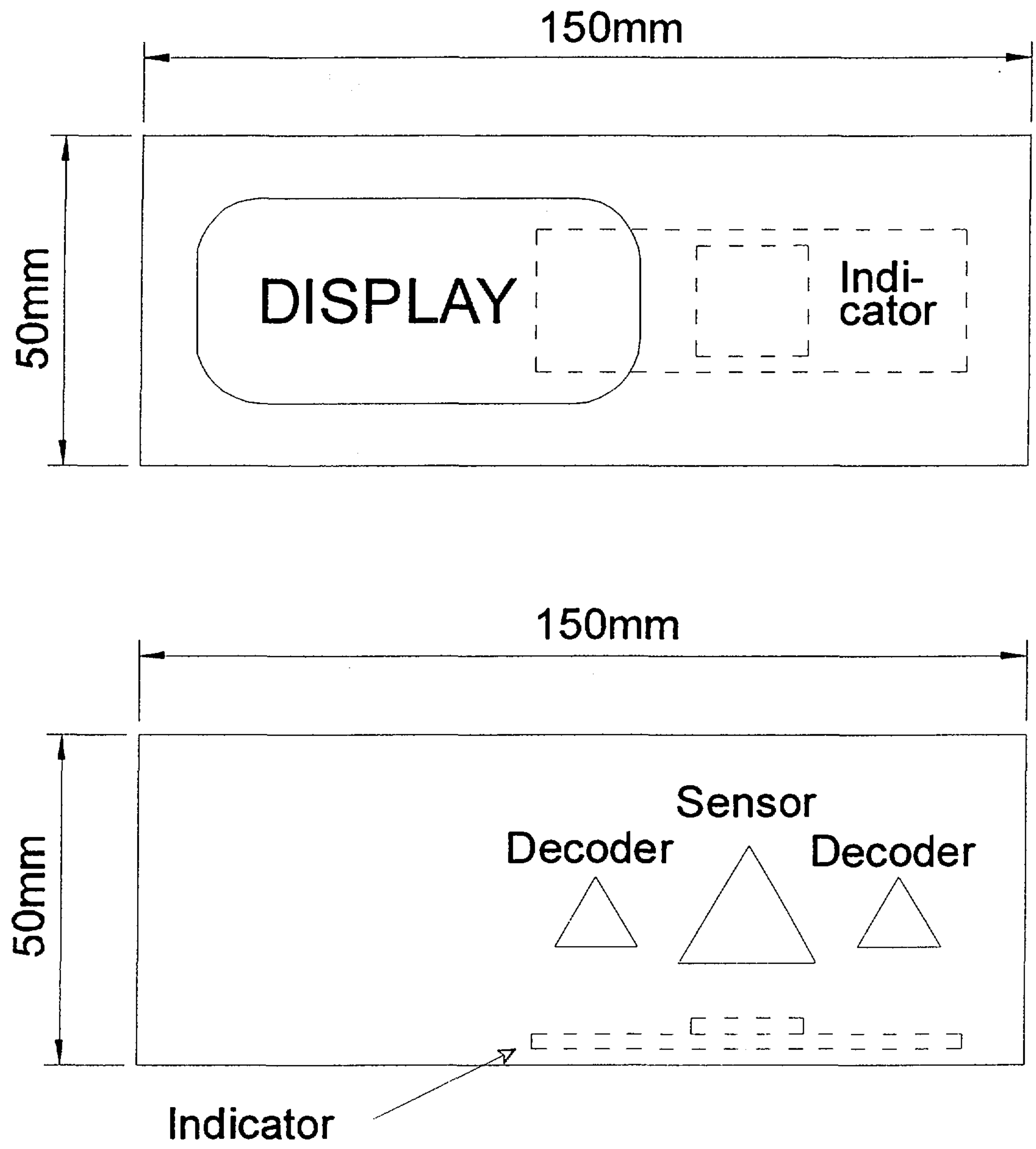

FIG. 15. Design of a miniaturized, hand-held label indicator reader for routine applications.

The experimental set up was used for reading the STERIN 125 and 300 indicators; results were compared to colorimeter readings of CIEL brightness. All data are normalized from their respective range to a common range of 1 to 0 for comparison (Figs 16 and 17); agreement between methods and for repetition is reasonable. The use of the proposed miniature reader (Fig. 15) is considered promising. Such equipment would need to identify the type of the 'label dosimeter', apply an appropriate decision function derived from the several response curves of the given sample and adjust for the required reliability of the judgement. This might become difficult as the comparison (Figs 16 and 17) of photodiode readings shows: the general trend is clear but some indicators (here STERIN 300) passes the threshold (here 300 Gy) by far too early (here 125 Gy). Much more work would be required to make such 'label dosimeters' finally acceptable for application in quarantine inspection of fruit disinfested by ionizing radiation.

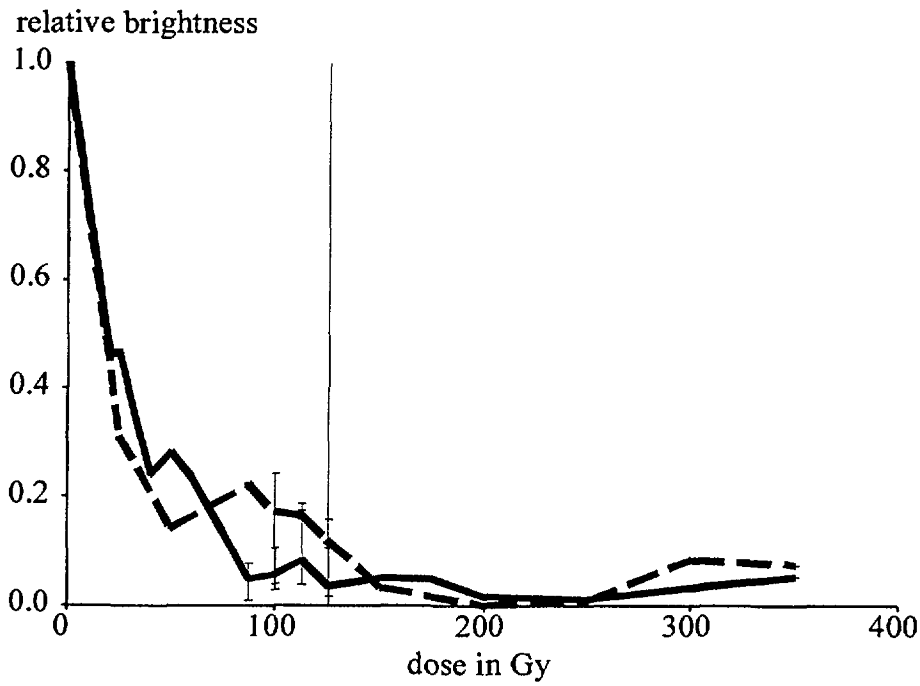

FIG. 16. Comparison of CIEL brightness (solid line) and photodiode (prototype reader, dashed line) signal for indicator nominal dose of 125 Gy; respective raw data normalized to range 0 1; fine vertical line indicator threshold; max/min of repeated measurements indicated.

FIG. 17. Comparison of CIEL brightness (solid line) and photodiode (prototype reader, dashed line) signal for indicator nominal dose of 300 Gy; respective raw data normalized to range 0 1; fine vertical line indicator threshold; max/min of repeated measurements indicated.

6. SUMMARY

The availability of the first commercial dose level indicator prompted attempts to verify radiation absorbed dose to items under quarantine control (eg for insect disinfestation) by some indicator attached to this item. The fundamental problem, however, would be the question whether a dose read at a certain reference position can be linked in a reliable and clearcut way to the respective minimum dose. Studies into variability of dose distributions under model situations reflecting commercial practices revealed the difficulty of such approach. Until now, only in situations where the position of the minimum dose is accessible and the indicator can be attached to this position a valuable dose judgement is possible. In many commercial situations the minimum position is inside the package or container and not accessible. Therefore, still more research into the statistical nature of dose distributions and into the link between measured values at reference positions to the parameters of such dose distributions is indispensable.

Samples of the new commercial dose level indicators were tested for their metrological properties. The devices are suitable for the intended purpose and the subjective judgement whether the threshold dose was surpassed is possible in a reliable manner. It should not be overlooked that the ranking and triangle tests reported here are overcritical compared to the routine situation of their intended application. Also instrumental measurements of the colour and brightness change of the indicator were executed. The subjective judgements are completely backed by the instrumental results. Consequently, a prototype reader was developed; first tests were successful.

ACKNOWLEDGEMENTS

The technical assistant Michael Knörr with support by the students Christiane Soika, Karin Weiss, Britta Mager conducted the large number of dosimeter readings and especially the subjective judgement on label dose indicators; Michael Knörr finally converted the results in fine graphical presentations; the personnel of the contract irradiator (Gammaster Allershausen) cooperated in an outstanding manner and contributed their dose readings. Gamma processing was done free of costs; ISP Wayne provided free of cost sets of prototype label indicators to all participants and extra samples for the studies reported here. This work and contribution is gratefully acknowledged; thanks go also to IAEA for accepting this research proposal without which the initiative for the reported work would have been missing and this research in principle necessary would not have been conducted.

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