

# Dose assurance service for low energy X-ray irradiators using an alanine-EPR transfer dosimetry system

*Helen J. Khoury<sup>1,\*</sup>, Kishor Mehta<sup>2</sup>, Vinicius S. de Barros<sup>1</sup>, Pedro L. Guzzo<sup>3</sup>, and Andrew G. Parker<sup>4</sup>*

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## Abstract

In recent years, X-ray irradiators have been introduced as an alternative to gamma irradiators due to the problems with the acquisition, transport and security of radioactive sources. Self-shielded low energy X-ray irradiators (such as the RS 2400) were developed to complement and eventually replace gamma sources. These irradiators are quite suitable for sterilizing insects for SIT applications, as well as for research on quarantine treatments of food and phytosanitary applications. During the irradiation of an agricultural product the absorbed dose inside the canister must be controlled to ensure that the desired effect is achieved without deleteriously affecting the product. The focus of this paper is to describe the Dose Assurance Service offered by the Insect Pest Control Laboratory of the Joint FAO/IAEA Division of Nuclear Techniques in Food and Agriculture using an alanine-EPR dosimetry system as a transfer standard for low energy X-radiation. All the administrative and technical issues are now finalized and the dose assurance service is ready for operation. The purpose of this Service is to help facilities using such irradiators to check the quality of their dosimetry systems.

Key Words: dose control, SIT applications, self-shielded X-irradiators, solid state dosimetry

Key Abbreviations: DAS, dose assurance service; EPR, electron paramagnetic resonance; IPCL, Insect Pest Control Laboratory; LMRI, Laboratório de Metrologia das Radiações Ionizantes

## Resumen

En los últimos años, se han introducido los irradiadores de rayos X como una alternativa a los irradiadores gamma debido a los problemas con la adquisición, transporte y seguridad de las fuentes radiactivas. Los irradiadores de rayos X de baja energía auto blindado (como el RS 2400) fueron desarrollados para complementar y eventualmente sustituir las fuentes gamma. Estos irradiadores son muy adecuados para la esterilización de insectos para aplicaciones de TIE, así como para la investigación sobre tratamientos de cuarentena de los alimentos y las aplicaciones fitosanitarias. Durante la irradiación de un producto agrícola la dosis absorbida dentro del recipiente debe ser controlado para asegurar que el efecto deseado se consigue sin afectar perjudicialmente el producto. El objetivo de este trabajo es describir el Servicio de Aseguramiento de Dosis que ofrece el Laboratorio de Control de Plagas de Insectos del grupo de la FAO/OIEA de la División Técnica Nuclear en la Agricultura y la Alimentación mediante un sistema de dosimetría-alanina EPR como un estándar de transferencia de radiación X de baja energía. Todos los detalles administrativos y técnicos están finalizados y el servicio de garantía de dosis está listo para funcionar. La finalidad de este servicio es ayudar a las instalaciones que utilizan estos irradiadores para comprobar la calidad de sus sistemas de dosimetría.

Palabras Clave: control de dosis, aplicaciones TIE, X-irradiadores auto blindado, dosimetría de estado sólido

Abreviaciones Clave: DAS, esquema de aseguramiento de dosis; EPR, resonancia paramagnética electrónica; IPCL, Laboratorio de Control de Plagas de Insectos; LMRI, Laboratorio de Metrologia das Radiações Ionizantes

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For radiation processing, including SIT applications and other agriculture product irradiation, effective dosimetry for dose control is essential for the quality of the irradiated product or material (Bakri et al. 2005; ISO/ASTM 2013c). Thus, the need for reliable dosimetry cannot be over emphasized, even during the research phase (ISO/ASTM 2013b). Several international standards have discussed procedures and methodology for carrying out necessary dosimetry at radiation facilities (ISO 2006; ISO 2011). After the initial commissioning of an irradiator and the calibration of the routine dosimetry system, there is a

continuous need to maintain the quality of the dosimetry at the facility. The generally accepted method for this is to compare the local dosimetry with that made by a transfer standard dosimetry system, where the transfer standard dosimeters are from a calibration laboratory. For reliable dosimetry, this standard dosimetry system should have been calibrated in an appropriate radiation field. Thus, for a facility using a low energy X irradiator, such as the RS-2400, Rad Source Technologies Inc. (Mehta & Parker 2011), it is essential that the standard dosimetry system is calibrated for that X-ray field. Recently, an alanine-EPR dosim-

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<sup>1</sup>Nuclear Energy Department - Federal University of Pernambuco, 50740-540, Recife, Brazil

<sup>2</sup>Vienna, Austria

<sup>3</sup>Department of Mining Engineering, Federal University of Pernambuco, 50740-540, Recife, Brazil

<sup>4</sup>Insect Pest Control Laboratory, Joint FAO/IAEA Division of Nuclear Techniques in Food and Agriculture, IAEA, A-1400 Vienna, Austria

\*Corresponding author: [hjkhoury@gmail.com](mailto:hjkhoury@gmail.com)

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etry system has been characterized for such low energy X-radiation in the dose range of 20–220 Gy (Khoury et al. 2015); thus, we are proposing here to use this as a transfer standard dosimetry system. Alanine-EPR has been used for many years as a transfer standard dosimetry system for gamma radiation (Co-60 and Cs-137) and also for electron beams for radiation processing applications (Mehta & Girzikowsky 2000). However, so far it has not been used for low energy X-radiation. Alanine as a dosimeter has many advantages, including its small size, robustness and little fading of the signal after irradiation. The alanine dosimeter is classified as a type I dosimeter as it is affected by individual influence quantities in a well-defined way that can be expressed in terms of independent correction factors (ISO/ASTM 2013c). The primary influence quantity that affects alanine response is the temperature during irradiation.

Besides characterizing the alanine-EPR system for low energy X-radiation, it was also characterized for cobalt-60 gamma radiation (Khoury et al. 2015). Thus, we intend to extend our dose assurance service to this type of radiation for the same dose range.

## Materials and Methods

### ALANINE DOSIMETERS

Alanine pellets were obtained from Aérial, Illkirch, France. The pellets consist of 91% alanine in a binder. The batch of pellets used had a mean mass of  $38.2 \pm 0.1$  mg, mean diam of  $4.0 \pm 0.05$  mm and mean height of  $2.7 \pm 0.02$  mm. No mass pre-selection was done, since the relative standard deviation of the mass was below 0.3%. The unirradiated as well as irradiated alanine dosimeters were stored in our EPR laboratory where the temperature and relative humidity was maintained at 20–30 °C and 30–50% RH, respectively.

### RADIATION FIELD

For calibration of the alanine-EPR transfer standard dosimetry system, we used the RS-2400 X-ray irradiator available at Moscamed, Brazil (Biofábrica Moscamed Brasil). This unit was supplied by Rad Source Technologies Inc. (Suwanee, Georgia, USA) and is currently being used for sterilization of fruit flies for SIT application. Details may be found in Wagner et al. (2009) and Mehta & Parker (2011).

### SPECTROMETER

The alanine measurements were conducted with a Bruker EMX10 EPR spectrometer, equipped with a standard cylindrical microwave resonator cavity operating in the X-band with the following parameters, based on the ISO/ASTM Standard 51607 (ISO/ASTM 2013a): microwave power 7.962 mW, modulation frequency 100 kHz and amplitude 0.977 mT. The alanine-EPR response was determined using the peak-to-peak amplitude of the most intense EPR peak, which is the middle one in the first-derivative EPR spectrum. This peak-to-peak amplitude is proportional to the alanine-derived free radical concentration in the alanine dosimeter. In order to minimize errors due to variations in the spectrometer sensitivity, a synthetic ruby ( $\text{Al}_{(2-x)}\text{O}_3:\text{Cr}_x$  with  $x = 2$  M%) was used as a reference sample. The ruby was synthesized in our laboratory and confirmed by X-ray powder diffraction.

### CALIBRATION OF THE ALANINE-EPR DOSIMETRY SYSTEM

The alanine-EPR dosimetry system was calibrated by irradiating sets of alanine dosimeters at a reference location in the X-ray field at several dose levels covering the relevant dose range from 20 to 220 Gy.

Initially, the dose rate or the dose-energy-ratio (DER) at the reference location was determined using a RadCal ionization chamber, calibrated in our Metrology Laboratory in a radiation field similar to that in the center of a canister in the RS-2400 irradiator. The spectrum in the RS-2400, calculated by Monte Carlo simulation, displayed in Mehta & Parker (2011). This particular spectrum was calculated with all the canisters filled with fruit fly pupae or a simulated material, and a tube voltage of 150 kVp.

Two correction factors were applied to the EPR response of each irradiated alanine dosimeter; these were for irradiation temperature and response of the un-irradiated dosimeter. For the correction of the temperature effect, we used a temperature coefficient of  $+0.14\%/^{\circ}\text{C}$  (F. Kuntz, personal communication). This could be a critical correction for the Dose Assurance Service if the irradiation temperature at the participating facility/laboratory is significantly different from that in our laboratory. The EPR response of an un-irradiated dosimeter (background) was subtracted from the response of each dosimeter, to arrive at the net response. The effect of humidity is mitigated by conditioning and sealing the dosimeters in water-proof poly(methyl methacrylate) (PMMA) holders during irradiation. The time interval between irradiation and dosimeter reading in the EPR spectrometer also influences the alanine response. Our protocol requires a waiting period of 3–5 d after irradiation before the measurement of the EPR response. Following these corrections, a regression analysis was performed between the corrected net alanine-EPR response values and dose values based on the RadCal ionization chamber.

## Dose Assurance Service

### GENERAL

This dose assurance service (DAS) will be offered by the Insect Pest Control Laboratory (IPCL) of the Joint FAO/IAEA Division of Nuclear Techniques in Food and Agriculture to SIT facilities that use low energy X-radiation, and will be operated by LMRI/DEN-UFPE, Brazil. The service will provide an independent check on the accuracy of the participant's dosimetry and the related procedures in use. Initially, this service will be limited to low energy X-radiation (50–300 kV), in the dose range of 20 to 220 Gy.

Procedures for the service are described here; this includes preparation and mailing of the dosimeter package to the participants, analyses of the irradiated dosimeters, certification and any follow-up action that may be necessary.

### ADMINISTRATIVE PROCEDURE

It is envisaged that the IPCL will inform relevant SIT facilities periodically that it would be desirable to have their dosimetry checked. Also, a request for participation in the dose assurance service can be initiated by the SIT facility. In this case, the request should be made in writing to the IPCL, which will make the appropriate decision. Then, when the service is needed, the IPCL will inform the head of the LMRI to schedule a service for a specific SIT facility.

Within a month of the request, the LMRI will send a dosimeter package to the SIT facility. This package will consist of 4 dosimeter holders, each containing alanine dosimeters. Of these, 1 holder will contain a set of 3 irradiated alanine dosimeters and a second will contain 3 un-irradiated dosimeters as controls, with the instruction that neither of the dosimeters is to be irradiated by the participant. The irradiated control will be irradiated at about 100 Gy, and its EPR response measured before sending. These values will act as controls to

check if the transportation conditions affected the dosimeters in any way. The package will include an instruction sheet as well as 2 technical data forms to be filled in by the participating facility.

On receiving the dosimeters, the participating SIT facility will irradiate the dosimeters as per instruction from the LMRI. It then will return the irradiated dosimeters along with the control dosimeters to the LMRI within 2 d after irradiation with the filled-in data form A (date of irradiation and irradiation temperature). At the same time, it will send the filled-in data form B to the IPCL (dose delivered to the dosimeters).

After receiving the irradiated alanine dosimeters from the participating SIT facility, the LMRI will measure the EPR response of the irradiated dosimeters as well as of the control dosimeters per its protocol. After the relevant analyses, it will forward the measured dose values along with associated uncertainty to the IPCL.

After receiving information about the dose values from both, the SIT facility and the LMRI, the IPCL will perform statistical analysis to compare the 2 sets of dose values. If the difference between the 2 dose values is greater than 5%, appropriate action will be initiated by the IPCL with the objective of locating the cause of the discrepancy. If some corrective actions are needed, re-check of the dosimetry of the SIT facility will be scheduled as soon as possible.

#### TECHNICAL PROCEDURE

Each dosimeter holder in the package will have a unique identification number inscribed on it. This number consists of 3 parts: xx-yy-z; where xx is the identification for the year (for example, '15' representing the year 2015), yy is the identification of the participant (for example, 01, 02 etc.), and z could be a, b, c or BG. The 2 holders, marked 'a' and 'b', are to be irradiated by the participating facility. The 'BG' dosimeters will be used to measure the background response to be subtracted from the responses of the irradiated dosimeters. The dosimeter holder marked 'c' (previously irradiated at LMRI) will act as a control as mentioned above. We are also planning to include 2 temperature-indicator strips in the package to provide information about the temperature exposure of the dosimeters during transport.

The LMRI will mail the dosimeter package to the participating facility by registered air-mail along with a covering letter, instruction sheet and technical data forms to be filled in by the participating facility. The LMRI will maintain the information about the corresponding dosimeter lot number.

On receipt of the irradiated dosimeters from the participating facility, the irradiated control dosimeters will be first analyzed in the EPR spectrometer by the LMRI. It is possible that its response is different than the one measured before sending them to the participating facility. The possible cause of the difference will be investigated, and if possible suitable corrections applied to the responses of the 'a' and 'b' dosimeters.

Each dosimeter will be analyzed with the EPR spectrometer and dose computed following our protocol. The irradiation temperature value that is used for correction of the EPR response will be determined based on the following information: data provided by the participating facility in the data sheet, the temperature recorded on the 2 temperature-indicator strips on the dosimeter holders, and the knowledge of the facility environment. After completing its analyses, LMRI will forward information about the measured dose values along with associated uncertainty to the IPCL.

After comparing the 2 sets of dose values, a certificate will be prepared and mailed to the participating facility by the IPCL. This will contain all relevant information, including name of the participating

facility, the participant's stated dose, DAS estimated dose, relative deviation and estimated irradiation temperature. If the relative deviation is larger than the accepted value of 5%, a letter will be included alerting to this fact. The letter may also include some information or suggestion to the participant as to the possible cause of this deviation and any corrective action. A repeat dose check will be recommended subsequent to these corrective actions.

## Maintenance of quality

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### GENERAL

Two aspects of the dose assurance service are maintained at high quality: i) the alanine-EPR transfer standard dosimetry system, and ii) the service itself. Besides the routine activities in the LMRI, this is achieved by the following 2 procedures:

- i) maintaining traceability for the dosimetry system to a national standard, and
- ii) performing proficiency testing of the dose assurance service.

### TRACEABILITY OF THE DOSE MEASUREMENT USING THE ALANINE-EPR DOSIMETRY SYSTEM

Traceability is maintained by periodic checking of the RadCal ionization chamber against the LMRI standard ionization chamber for the appropriate radiation field.

### PROFICIENCY TEST

Every 2 years LMRI will send a standard dosimetry package to the IPCL for irradiation as a participating facility. IPCL fulfills 2 important criteria necessary for this procedure, a) it has a low energy x-ray irradiator (RS-2400), and b) it has a RadCal ionization chamber, which has traceability to the IAEA Dosimetry Laboratory.

### Dose Levels

This test will be carried out for at least 2 dose levels: about 60 and 180 Gy.

### Discrepancies

If the LMRI value does not agree with the IPCL value within 5% for either of the 2 dose values, the cause of the discrepancy will be investigated.

## Discussion

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All the administrative and technical issues are now finally resolved and the dose assurance service is ready for operation, and thus to provide help to the relevant irradiation facilities for checking the accuracy of their dosimetry. Initially this service will be focused on those facilities using low energy X-radiation for SIT (20–220 Gy). However, LMRI is prepared to expand this service to other facilities that use Co-60 gamma radiation for similar dose range. All the technical aspects related to such radiation sources are also completed.

Initially, this service is limited to 20–220 Gy, however, if there is a need it can be easily expanded to cover a wider dose range, for example, for other applications besides the SIT.

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