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# Determination of the Absorbed Dose to Water in Cs-137 Research irradiator Chambers Using Fricke Dosimetry

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**Astract**: The IDQBRN/CTEx has a Cs-137 research irradiator to promote research development in the field of material irradiation, assisting in obtaining data and scientific publications in the area. Currently, tests are being conducted with pellets and plates of materials placed at the base of the irradiator chambers, requiring verification of the absorbed dose to water in the central areas of the chambers, where the plates and pellets are positioned, ensuring the dose delivered in processes, as well as ensuring a corresponding average value for each material positioned within this measured area. By using the Fricke chemical dosimeter, it was possible to determine that for the central position closest to the door of the upper chamber of the irradiator, the highest absorbed dose in water found in the measurements conducted was a value of 5.10% above the expected value compared to the last dosimetry performed with a ceric-cerous sulfate chemical dosimeter. The results show that using the Fricke dosimeter it was possible to characterize the irradiator obtaining complete dosimetry and doses according to the positioning of the material within the chamber to subsequently create a simulator that could be used for dosimetry of the irradiation volume.

**Keywords:** research irradiator, dosimetry, Fricke solution, chemical dosimetry.

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# Determinação da Dose Absorvida na Água em Câmaras de irradiador de pesquisa de Cs-137 utilizando o Dosímetro Fricke

**Resumo**: O IDQBRN/CTEx possui um irradiador de pesquisa de Cs-137 para promover o desenvolvimento de pesquisas na área de irradiação de materiais, auxiliando na obtenção de dados e publicações científicas na área. Atualmente são desenvolvidos testes com pastilhas e placas de materiais colocados na base das câmaras do irradiador, sendo necessária a verificação da dose absorvida na água nas áreas centrais das câmaras, local onde são posicionadas as placas e pastilhas, garantindo a dose entregue nos processos, além de garantir um valor médio correspondente para cada material posicionado dentro desta área medida. Utilizando o dosímetro químico Fricke foi possível determinar que para a posição central mais próxima da porta da câmara superior do irradiador, maior dose absorvida na água encontrada nas medidas realizadas, foi encontrado um valor de dose absorvida 5,10% acima do valor esperado em relação à última dosimetria realizada com dosímetro químico de sulfato cérico-ceroso. Os resultados mostram que com a utilização do dosímetro Fricke foi possível caracterizar o irradiador obtendo a dosimetria complete e as doses de acordo com o posicionamento do material dentro da câmara para posteriormente crias um simulador que poderá ser utiliizado para dosimetria do volume do irradiador.

**Palavras-chave:** irradiador de pesquisa, dosimetria, solução Fricke, dosimetria química.

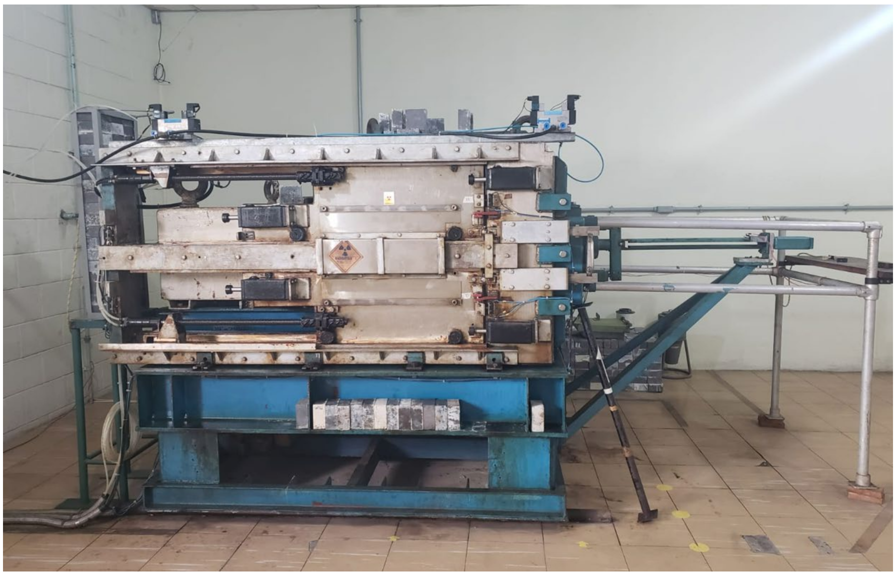
## 1. INTRODUCTION

There are some necessary precautions when using a researche irradiator, among them is the need to ensure the dose delivered by the device during its operation [1]. Depending on the material to be irradiated, the dose rate can be a contribution factor in modifying the structural characteristics of the material, a necessary consideration in the irradiation of polymers, for example [2].

IDQBRN/CTEx (*Instituto de Defesa Química Biológica Radiológica e Nuclear/Centro de Tecnologia do Exército*) has a Cs-137 research irradiator, illustrated in figure 1, which contains two chambers for material insertion, with dimensions of 20.00 cm in height, 68.00 cm transversely, and 137.00 cm longitudinally [3]. Its activity (A) in kCi as a function of the irradiation year (t) can be described by equation 1 [4].

$$A = 108 * e^{-0.023*(t-1969)} \quad (1)$$

**Figure 1:** IDQBRN/CTEx Cs-137 research irradiator



The current activity of the irradiator is A= 1.15 PBq. The high dose rate of the Cs-137 irradiator requires a dosimeter that can be exposed to obtain a reliable response regarding process dosimetry. The Fricke chemical dosimeter has reliability in the dose range of 20 to 70 Gy, achieving better than 2% accuracy for a dose of 2 Gy/min [5].

The advantage of the Fricke dosimeter over others used in high dose rate dosimetry is that it is an absolute dosimeter, recognized by the ISO/ASTM E-51026-2015; ISO/ASTM 51939:2017; ICRU REPORT 90; ICRU REPORT 80 protocols, without needing the use of another reference dosimeter [6-8].

Considered the best chemical dosimetry system, the Fricke chemical dosimeter can obtain more precise values in the absorbed dose range of 40 to 420 Gy [9-10], being ideal for the dosimetry of industrial or research irradiators.

The aim of the initial Fricke dosimetry measurements was to determine the absorbed dose to water $(D_w)$ along the chambers, understanding the radiation behaviour at the lowest height within the irradiator chambers, namely at the base of both chambers, and how the source movement could contribute to the increase in absorbed dose for the materials positioned in the drawers during the irradiation processes.

The determining of absorbed dose to water values at the base of the chambers, without the use of a phantom, is justified by the demand for irradiation of materials in the form of pressed pellets or plates of 3D printed materials, whose compositions vary. The objective is to verify the damage caused to the material according to the absorbed dose to water to which these materials are exposed, enabling the identification of new materials with potential for shielding or for use in new phantoms.

# 2. MATERIALS AND METHODS

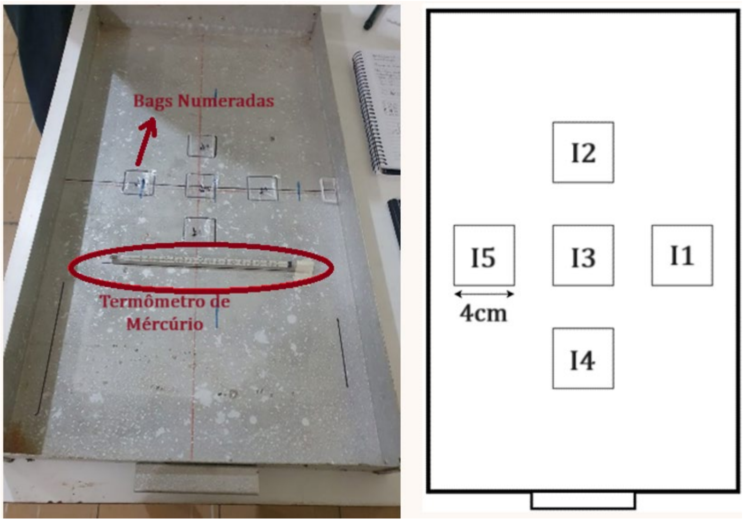
## 2.1. Research Irradiator of Cs-137

The Cs-137 research irradiator at the IDQBRN/CTEx facilities consists of two irradiation chambers, with a total useful volume capacity of 100 litres [3].

It is a cavity irradiator, with pneumatic control that positions the source at the irradiation site and returns it to a shielded barrel after the irradiation is completed. The gamma source has a density of 3.00 g/cm$^3$ of CsCl plus a binder [3].

To better position the materials, the irradiator has an aluminium drawer, designed by the team responsible for handling the equipment. Figure 2 shows the irradiator drawer with two mercury thermometers positioned near the centre of the drawer used to verify the temperature reached during the procedure.

**Figure 2:** Positioning the bags and thermometer in the aluminium drawer of the irradiator



## 2.2. Fricke Dosimeter

The preparation of the chemical material is carried out in the Fricke Laboratory of the Radiological Sciences Laboratory (LCR/UERJ) according to the literature for the preparation of a standard solution of ammoniacal ferrous sulfate hexahydrate ($FeSO4$) [11].

For its preparation, the following are used: ammonium iron (II) sulphate hexahydrate [(NH4)2Fe(SO4)2**6H2O] (99%), sodium chloride [NaCl] (99.5%), and sulfuric acid [H2SO4] (95.0-99%). In a 2 L volumetric flask, a solution is composed of 44 ml of diluted sulfuric acid in 250 ml of ultrapure water. This solution is pre-irradiated with 10 Gy to eliminate possible reducing agents present in the acid using an X-ray irradiator, and after 1 hour, 0.120 g of NaCl and 0.784 g of ferrous sulfate are added. To complete the volume of 2 L, high purity water is added. The volumetric flask is then wrapped with protection against ambient light and stored for 24 hours before undergoing Quality Control and being put to use [5, 12-13].

At the end of the process, the density of the Fricke solution is obtained using a calibrated pycnometer, with an average value of 1.023 g/cm³ at a temperature of 25ºC [5].

The Fricke dosimeter is placed in sterilized polyethylene bags containing approximately 1.4 g of solution, as illustrated in figure 2 [6].

The bags are divided into three groups:

- irradiated bags: positioned in the irradiator drawers and exposed for 9 minutes;
- control bags: used for background radiation measurement; and
- transit bags: used to determine the transit dose, i.e., the time and dose associated with source transfer, counting from its exit from the safe to its return.

## 2.3. Irradiation of Dosimeters

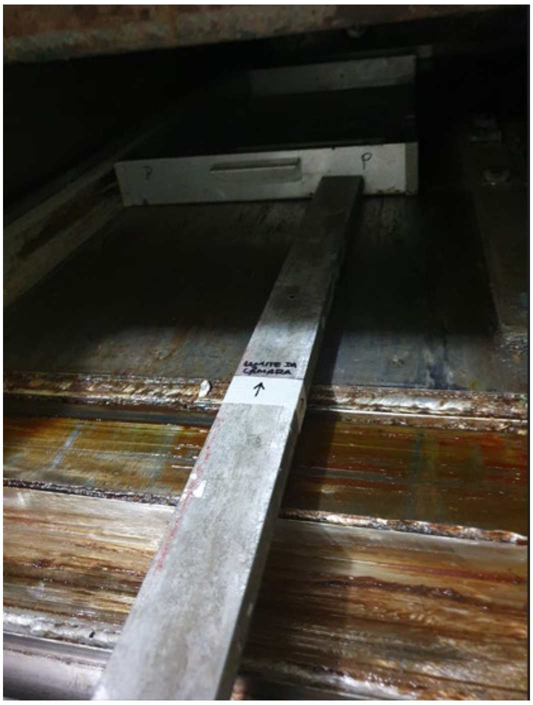
The dosimeters were exposed for a duration of 9 minutes, and the experiment was repeated 3 times with the position remaining the same and the dosimeters being exchanged.

Control bags, not irradiated, were used throughout the procedure to enable the extraction of background dose values.

Another factor to consider is the direct dependence of temperature related the exposure time that the material is subjected to within the irradiator chambers, as the interaction of thermal radiation will increase with the increase in exposure time. Therefore, all irradiations were conducted with calibrated mercury thermometers positioned in both drawers (upper and lower). The thermometers (HG - Brasil, Nº 2552, Total Immersion) can be seen inside the aluminium drawer, as shown in figure 2.

In figure 3, the placement of the aluminium drawer in the upper chamber of the irradiator can be seen, positioning this drawer on the left wall of the irradiator and at a fixed distance from the front part of the door.

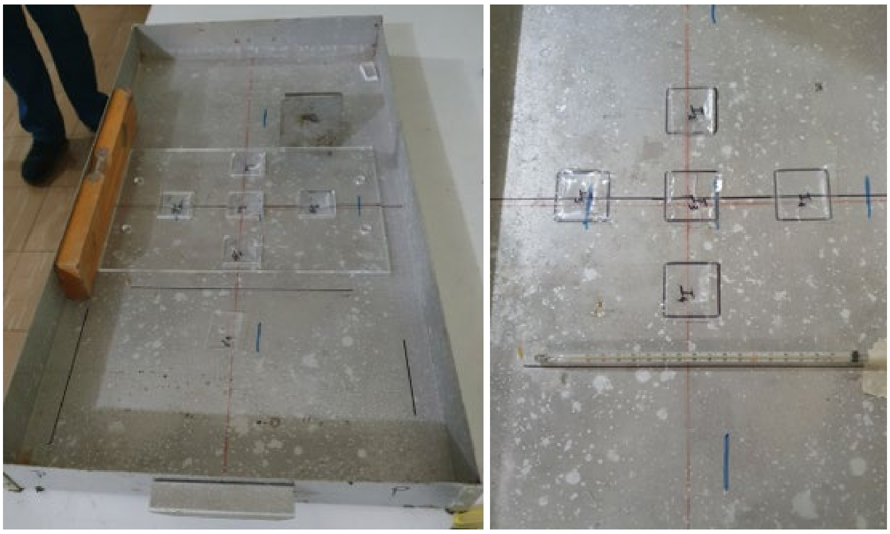
**Figure 3:** Positioning the drawer with the dosimeters in the chamber



To facilitate the reproduction of the position, the standard measurement of 36.00 cm from the front edge of the chamber was used to positioning the drawers in the irradiator, both for the upper and lower drawers. The drawer was always kept against the side where the door of the safe containig the source opens, as shown in figure 4.

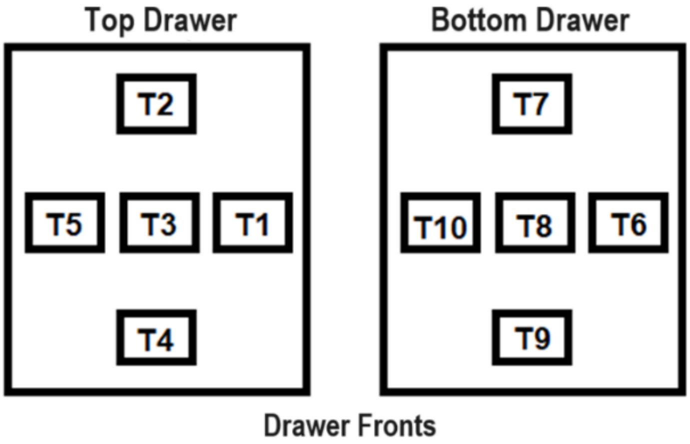
In this position, it was possible for the central bags, T3 and T8, to be positioned 69.00 cm from the chamber entrance (longitudinal direction), and 15.00 cm from the side where the safe with the source is positioned (transverse direction).

**Figure 4:** Acrylic mould used to reproduce the positions



The distribution of the bags followed the same positioning pattern for all irradiations performed, both in the upper and lower drawers. In figure 4, the acrylic mold used for repeating the positioning of the bags can be visualized.

**Figure 5:** Position of the bags in the drawers



## 2.4. Reading Fricke Dosimeters

The solutions, irradiated and non-irradiated, are withdrawn from the bags and placed in the cuvette of the Varian Cary 50 BIO spectrophotometer (Varian Australia Pty Ltd.), which reads through the direct interaction of the light beam with the cuvette and the deposited liquid, absorbance. For this purpose, a wavelength of 304 nm and a resolution of 1nm are used. Thus, it is possible to obtain the absorbance (Abs) measurement directly from the spectrophotometer.

From these data, it is possible to acquire the Fricke Dose, which will be obtained through the absorbance variation values divided by the fixed parameters related to the Fricke chemical solution [5].

To acquire the absorbed dose to water ($D_w$), it is necessary to convert the Fricke Dose ($D_F$) using equations 2 and 3 [14].

$$D_F = \frac{\Delta OD}{G(Fe^{3+}).l.\rho.\varepsilon} \tag{2}$$

Where $\rho$ is the density of the Fricke solution, whose value obtained for this work was 1.023 g/cm$^3$ at 25°C, calculated by the Fricke Laboratory of LCR; $\Delta OD$ is the absorption difference between the irradiated solution and the non-irradiated control sample; and the molar absorptivity coefficient of ferric ions (ε), at 304 nm, which is 2174 $M^{-1}.mol^{-1}$ [14].

For the conversion of Fricke dose to absorbed dose to water ($D_w$), equation 3 is used.

$$D_w = D_F.f.p_{wall}.k_{dd,F} = \frac{\Delta OD}{G(Fe^{3+}).l.\rho.\varepsilon}.f.p_{wall}.k_{dd,F} \tag{3}$$

Where $f$ is the conversion factor due to the difference in dose deposited in the same volume of water; $p_{wall}$ is the wall correction factor where the solution is deposited; and $k_{dd,F}$ is the correction factor due to non-uniformity of the lateral dose profile over the area presented by the container containing the solution and the variations in the beam depth distribution in the direction parallel to the beam axis.

The relationship found for calculating the absorbed dose to water ($D_w$) in relation to the value found in the Fricke dose is presented in equation 4 [8], value found in the unit of measurement Gy.

$$D_w = \left(\frac{\mu_{en}}{\rho}\right)_{watter,Fricke} .\, (p)_{watter,Fricke} .\, D_F \tag{4}$$

Where:

$\left(\frac{\mu_{en}}{\rho}\right)_{watter,Fricke}$ – is the ratio of mass absorption coefficients of energy of water and Fricke ; and

$(p)_{watter,Fricke}$ – is the bags disturbance factor, which will be neglected in the calculation as it is made of thermoplastic polymer [8].

When developing the calculation, the corresponding value for conversion will be given by equation 5 [8].

$$D_w = 1{,}004.\, D_F \tag{5}$$

Thus, it is a good approximation to consider the values found in the Fricke dose as the absorbed dose to watter.

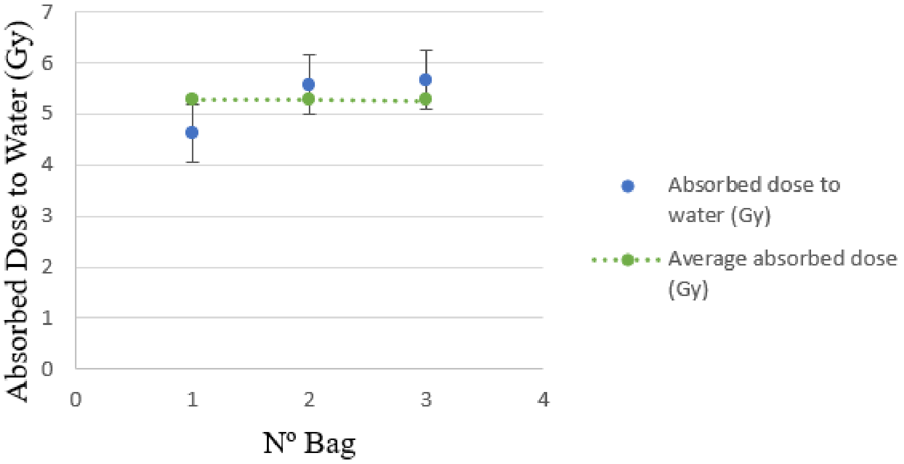
# 3. RESULTS AND DISCUSSIONS

## 3.1. Transit Dose Values

Data for transit doses were acquired, reproduced on three different days, with the purpose of finding an average value for the transit time and for the average value of absorbed dose to water at the center of the drawer. The average transit time found was (32.67 ± 2.52) seconds.

The data obtained, in terms of absorbed dose to water, were reproduced in figures 6 and 7, with the type A uncertainty bar of the measurements.

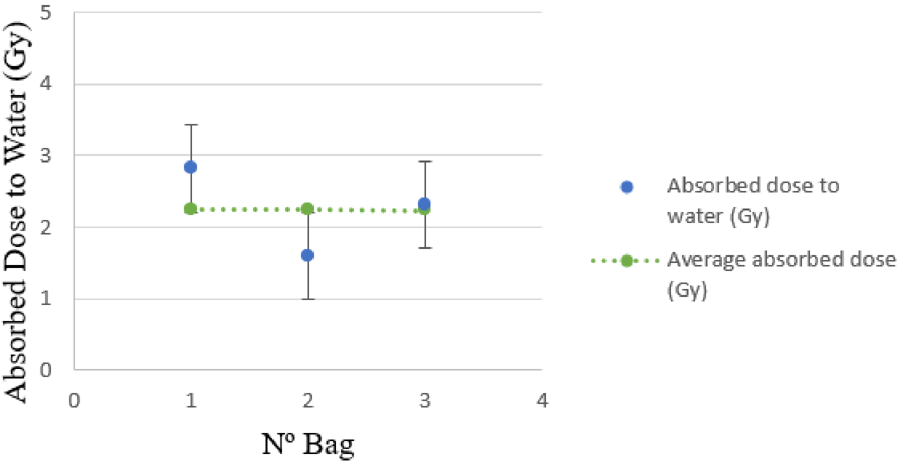
**Figure 6:** Transit bag in position T3



The average transit dose found in this central position of the upper chamber was (5.28 ± 0.58) Gy.

For the lower chamber, the average transit dose found was (2.24 ± 0.61) Gy.

**Figure 7:** Transit bag in position T8



## 3.2. Irradiation Values for the Two Chambers

An average was taken between the bags in the same position to verify the type A uncertainties associated with the measurements between them, and thus obtain a good basis for mapping the doses at these 5 points in each chambers.

The irradiator's programming allows the entire process to be completed within the time planned by the irradiator's own schedule, thus, within the schedule 9 minutes of irradiation, the (32.53 ± 2.52) seconds of source transit time are included. Comparative data for the 3 irradiations were described in table 1.

**Table 1 :** Comparative average

| BAGS COMPARED | AVAREGE DOSE (Gy) | TYPE A UNCERTAITY (Gy) |
|---|---|---|
| I1, I11 and I21 | 224.43 | 18.47 |
| I2, I12 and I22 | 213.47 | 2.61 |
| I3, I13 and I23 | 214.69 | 9.67 |
| I4, I14 and I24 | 227.83 | 9.45 |
| I5, I15 and I25 | 215.94 | 8.16 |
| I6, I16 and I26 | 119.32 | 4.39 |
| I7, I17 and I27 | 123.85 | 6.51 |
| I8, I18 and I28 | 128.14 | 6.24 |
| I9, I19 and I29 | 122.03 | 7.69 |
| I10, I20 and I30 | 117.63 | 4.98 |

The times obteined in the irradiations of each set of 10 bags, with 5 in the upper chamber and  in the lower chamber, were respectively: 9 minutes, 9 minutes and 7 seconds and 9 minutes and 10 seconds.

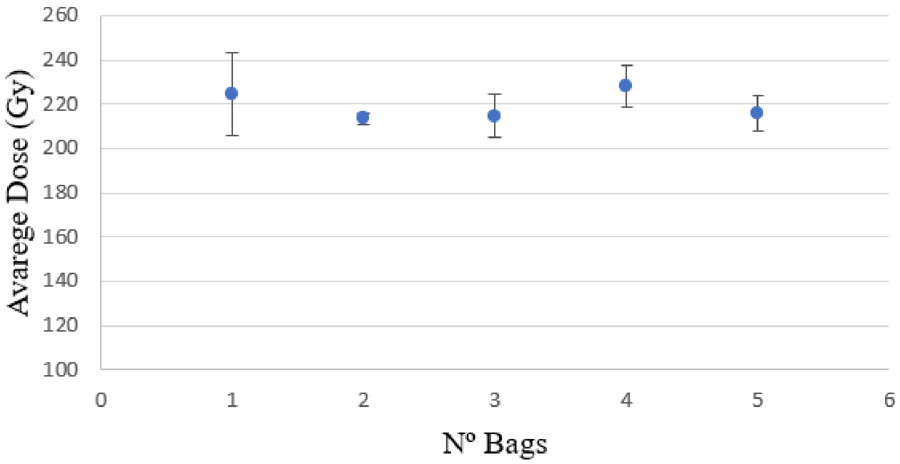
### 3.3. Data Compared for Irradiations in the Chambers

For better understanding, graphical analysis of the averages of the bags by positions in the upper and lower chambers are presented in graphs 2 and 3.

In figure 8, it was possible to show the averages relative to the 5 bags positioned in the upper drawers, in the order of:

- in position 1 are bags I1, I11 and I21; in position 2 are bags I2, I12 and I22; in position 3 are bags I3, I13 and I23; in position 4 are bags I4, I14 and I24; and in position 5 are bags I5, I15 and I25.

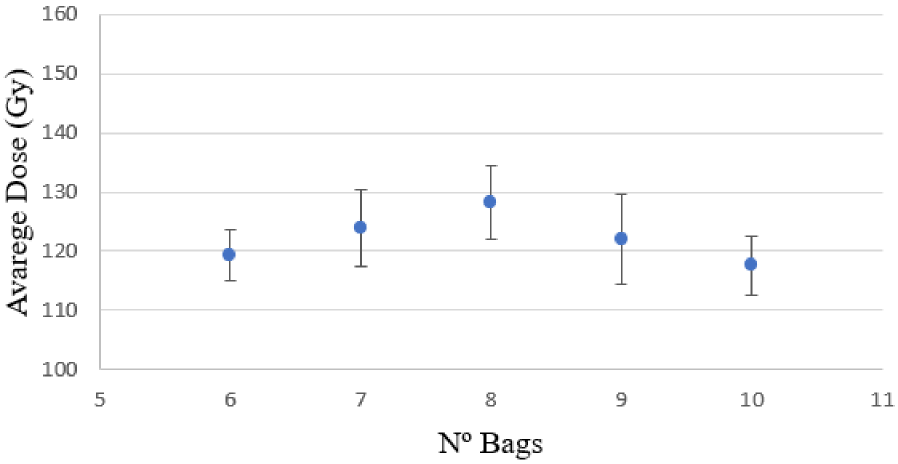
**Figure 8:** Average absorbed dose to water X Positions in top drawer



In figura 9, it was possible to show the averages relative to the 5 bags positioned in the lower drawers, in the order of:

- in position 6 are bags I6, I16 and I26; in position 7 are bags I7, I17 and I27; in position 8 are bags I8, I18 and I28; in position 9 are bags I9, I19 and I29; and in position 10 are bags I10, I20 and I30.

**Figure 9:** Average absorbed dose to water X Position in bottom drawer



The $D_w$ at each position will depend directly on the angle of the radiation beam in relative to the distance at which each of the Fricke solution bags is located, justified by the inverse square law relationship. Since the source is not point-like and moves along a track, these two factors also need to be taken into account.

The bags positioned further to the left of the irradiator are closer to the location where the source exits, and therefore have the hidhest doses in the in the chambers.

The lower doses in the lower chamber result from the distance betwee the source movement location and the height of 18.7 cm from the position where the bags were inserted.

As the source is not point-like, variations are generated which, in an analytical comparison of values, need to be taken into consideration, such as the increase in absorbed dose due to source movement.

Due to dependence on a compressor for source positioning, different exposure values occur in some irradiations, which results in an increase in dose for cases where the source takes longer to position and/or return to the safe.

These dose increase values generated by the time difference in the irradiation process time were not removed during the work because it was necessary to determine in which position the source stayed longer. Thus, the total values of 9 minutes in the first irradiation; 9 minutes and seven seconds in the second irradiation; and 9 minutes and 10 seconds in the third irradiation were obtained.

After determining the distribution of the absorbed dose in the chamber floor, the project for the development of a PMMA phantom was initiated, which would be easy to handle and with cavities for insertion of liquid state chemical dosimeters.

## 3.4. Comparison with previous dosimetry

Dosimetric mapping was carried out in the year 2000 using ceric-cerous sulfate dosimeters produced by MDS Nordion. In the year the measurements were taken, the activity of the irradiator source was 1.9 PBq [15,4].

According to the data obtained in the year 2000 through dosimetric mapping, it was possible to verify that the central position of the upper chamber of the irradiator, at height $z = 0$ cm (chamber floor), had a dose rate of 37.27 Gy/min, being the highest dose rate value presented for the volume region verified in this dosimetry [4]. The value found in the same position for the dosimetry conduced in the year 2023, if considered in dose rate, at height z = 0 cm, was approximately 23.50 Gy/min, being the highest dose rate found on the chamber floor of the upper chamber.

From the year 2000 to 2023, the dose rate decreased by 36.94%, although the expected value for the computational simulation specific to IDQBRN/CTEx indicated a decrease value for the year 2023 of 40.41%. An important observation that may justify the difference found is that in the dosimetry for the year 2000, the contribution values of scattering generated by computational simulations (Monte Carlo) carried out by IDQBRN/CTEx. For the current work, it was taken into account that all the scattering generated by the internal material of the irradiator should be maintained as an important part of the absorbed dose contribution by the materials that are irradiated. Thus, the value withdrawn by the uncertainty calculated in this project takes into account the type A uncertainty, obtained by the type A standard deviation of the sample readings, adding the ttype B uncertainty value of the dosimeter which is previously determened by the Fricke Laboratory of the Radiological Sciences Laboratory (LCR) of the State University of Rio de Janeiro (UERJ) and calculated together with the conversion for the acquisition of the Fricke dose values.

## 4. CONCLUSIONS

As the pneumatic system of the source exhibited non-continuous behaviour during the first dosimetry of the year 2023, varying by a few seconds in the transit of the source, it was necessary to verify the feasibility of continuing with a dosimetry in the irradiator volume or waiting for further adjustments so as not to incur excessive material cost in the manufacture of dosimeters, as the Fricke dosimeters were provided by the Fricke Laboratory of the LCR/UERJ as research cooperation between the institutions.

With the data from this first dosimetry on the ground, it was possible to locate and dimension the source, and observe that there is a contribution from the spread of the internal irradiation material to decrease the edge effect caused by the source geometry, as well as an increase in absorbed dose in the central part closest to the irradiator door.

It was possible to determine the average absorbed dose to water for each of the five positions of each chamber using the Fricke dosimeter. All the values found, considering that the dose were measured point by point, have a type A uncertainty of less than 3% when removing the time difference in seconds and the spread of the internal irradiation material, stipulated by Monte Carlo in the dosimetry performed prior to this work. The values for the time variation were not removed from the values of absorbed dose to water so that the generated statistics could be faithful to the equipment operation, thus translating data closer to the behaviour of the absorbed dose by the materials exposed in each of the position.

One of the conclusions reached was infeasibility to comparing the doses of the chambers in general, comparing the responses and finding a relationship between the absorbed dose to water for the positions of the lower chamber in relation to the same positions of the upper chamber, as they provide non-proportional responses between them. Thus, the best way to verify the doses is to consider them as separate systems and generate the report for each of the irradior chambers, in order to obtain a point-by-point statistics and interpolation for intermediate values for the preparation of a dose map.

With the data obtained, it is possible to identify the need to automation of the control system for source transit because the main reason for the increase in time measurements stems from the occurrence of locking in displacement at various times, caused by the use of a compressor associated with a pneumatic system that pushes the source to the irradiation site and retrieves it at the end of the exposure time. This also responds to the fact that the use of absorbed dose to water is the best response in relation to the dose rate response.

Regarding the linearity of the absorbed dose in the irradiator chambers, it was possible to verify the non-linearity in the behaviour of the dose distribution of the measured space, which was already expected due to the source not being punctual and the measured area being very extensive.

The Fricke dosimetry with liquid-phase chemical solution proved to be ideal for dosimetry at industrial irradiation levels due to its responses amidst dose variations that exceed the dose range of many other dosimeters known in the literature, thus making it possible to continue the volumetric dosimetry project with the possibility of exposure for varying times.

## ACKNOWLEDGMENT

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## CONFLICT OF INTEREST

All authors declare that they have no conflicts of interest.

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