ACTIVITY DETERMINATION BY A $4\pi-\gamma$ WELL-TYPE IONIZATION CHAMBER

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ABSTRACT

A 4π-γ ionization chamber system has beem calibrated for radioactivity determinations of liquid samples. The efficiency curve was determined experimentally for the photons in the 60 keV to 2750 keV energy range. The experimental values were fitted to an analytical function of photon energy and a good agreement was obse.ved over the entire range of energies.

INTRODUCTION

This paper describes a 4π-γ ionization chamber to be used as a reference system for radioactivity determination of liquid samples. The system was installed at the Laboratório de Metrologia Nuclear (LMN) in the Instituto de Pesquisas Energéticas e Nucleares (IPEN) of São Paulo, Brazil with the main purpose of routine calibrations.

The LMN has two $4\pi\beta$-γ coincidence systems for activity standardizations with accuracy better than 0.1%. Although these systems have quite a good performance, too long a time and exaustible work are necessary for each standardization. Therefore, it was very important for the LMN to install and calibrate a secondary system where the activities can be determined in a easier way without loosing the accuracy of the primary standards.

Several well known advantages of a 4π-γ ionization chamber system can be pointed out: low geometry dependance, high stability, easy sample preparation, short measuring time and a wide range of measurable activities; the system can also be used in the determination or check up of dilution factors and other gravimetric comparisons. Finally, this system permits the activity determination of some radionuclide specimens which are hard or even impossible to be measured by other methods such as $4\pi\beta$-γ coincidence.

To achieve an uncertainty of some tenths of a percent it was necessary to make a series of experimental studies which are described in the following sections.

PRINCIPLE OF OPERATION

The current originating in the ionization chamber is measured by the rate-of-charge method[5] with two preset voltage levels (Figure 1).

This current charges an integrating capacitor (C) placed at the feedback terminal of the electrometer (E). When the voltage in the capacitor reaches V_1 (preset by the first discriminator D_1) a control unit (CU) starts a timer (T) and simultaneously, the data indicated in a digital voltmeter (DV) and in a digital clock (DC) are transferred to a printer (P) which registers the values of V_1 and t_1 (the time of day at beginning of the measurement). At the time t_2, when the voltage reaches the value V_2 (preset by the second discriminator D_2), the timer is stopped and the values V_2 and $t_2 - t_1$ (Δt) are registered. The current is then given by:

$$I = C \frac{(V_2 - V_1)}{\Delta t}$$

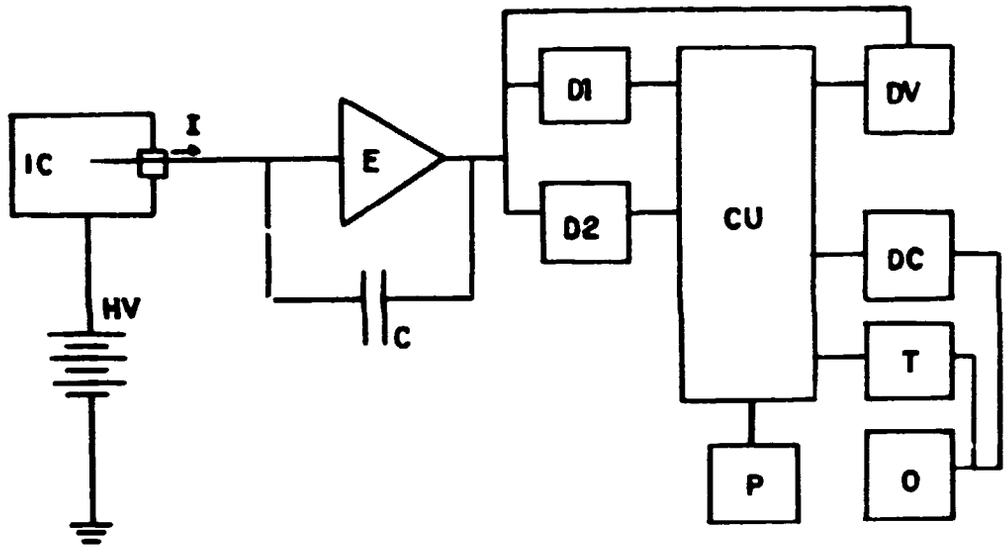


Figure 1 – Ionization chamber system – electronic diagram

In this work only relative measurements are involved, therefore it is not necessary to know accurarely the C value.

EQUIPMENT

The chamber is a 4π – Well Type Model IG12/A20 (20^{th} Century Electronics Ltd.). It is filled with 20 atmospheres of Argon and has a well diameter of 2 inches.

The electrometer is a 610 model of Keithley Instuments with input impedance greater than $10^{14}\Omega$, zero drift lower than 1 nV/day and with noise less than 5×10^{-15} A.

The digital voltmeter is the model 34701A (Hewlett Packard) with resolution of 5 digits and with accuracy near 0.03% full scale. The digital output is provided by a BCD module type 34721A (Hewlett Packard). The high voltage power supply is the model 415B (Fluke) with an stability of 0.01% and a maximum ripple of 100μV. The digital printer is a 5055A model (Hewlett Packard).

All other electronic equipments were manufactured in our own institute[19]. They include two discriminators, control unit and digital clock with timer. The discriminators can preset voltages from zero to 10 volts. The accuracy of the timer is less than 0.001% full scale and its resolution reaches 10^{-3} sec.

SOURCE PREPARATION AND HOLDER

The ampoule selected for calibration is a standard type adopted by the BIPM (Bureau International dés Poids et Mesures). It is made of commom glass with 16.4 ± 0.1 mm in external diameter, 0.65 ± 0.05 mm in wall thickness with total capacity of 5 ml. The height of solution in the

ampoule is 20 ± 1 mm. For filling the ampoule with radioactive solution several steps were performed according to the BIPM recommendations[2]. The ampoule holder is made of perpex, with 2 mm of wall thickness and with an internal diameter chosen to fit the ampoule with minimum tolerance. Its base has the same diameter as the chamber well in order to allow a good radial positioning (± 0.3 mm). The holder is placed into the well by means of an aluminium tube as shown in Figure 2.

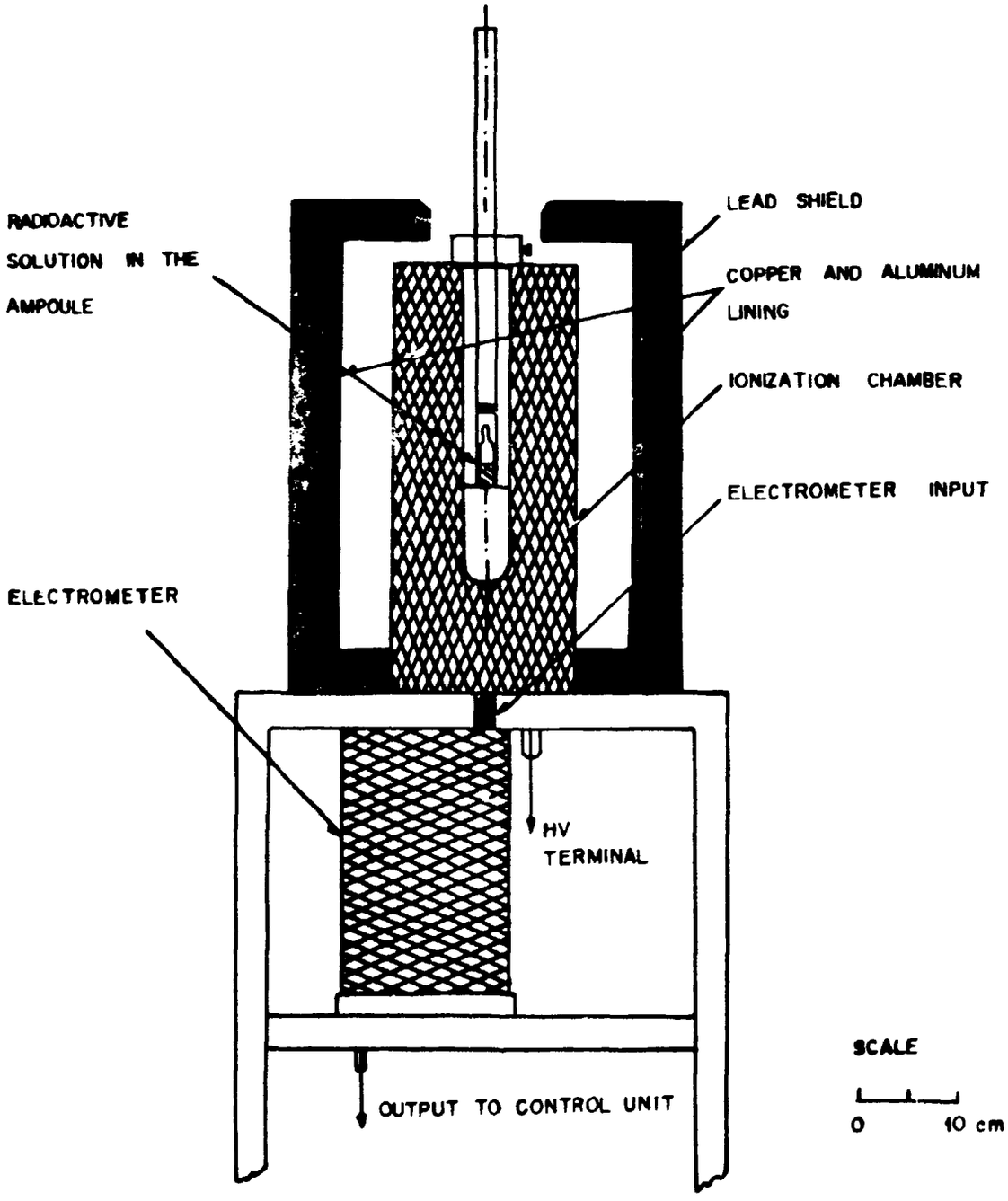


Figure 2 – Ionization chamber system – mechanical diagram

The reproducibility in the vertical positioning is ± 0.5mm. As suggested by Taylor[12] the calibration of the chamber has been repeated, lining the holder externally with Cadmium (0.7mm). This was done to absorb the X-radiation produced in electron capture or internal conversion processes. The bremstrahlung radiation turned out to be very low because it is originated essentially in the solution, glass or perspex, which are materials of low atomic number.

DETECTOR PERFORMANCE

The curve of chamber current versus applied voltage is almost linear between 200 and 1000 Volts with a slope of 0.094% per 100 Volts. The sensitivity of the chamber is 2.06×10^{-12}A per μCi of ^{60}Co. The average detector background measured during a period of several days using 4.5 cm of lead shield, resulted in $(9.9 \pm 0.3) \times 10^{-14}$A. A small backscattering has been observed due to the shield presence; the chamber current increase was 0.1% for 1.25 MeV(^{60}Co) and 0.5% for 0.411 MeV(^{198}Au). The internal face of the shield was covered with aluminum and copper in order to reduce X-radiation induction in the lead shield.

The position of the ampoule for the maximum chamber response in the well axial direction is about 21 cm below the top of the chamber. It was observed that it is about 0.5 cm higher going from 0.279 MeV(^{203}Hg) to 1.25 MeV(^{60}Co). The variation in the response around this point is small, near 0.060% per mm. The response variation of the chamber in the radial direction (about 0.1% per mm) is much higher than in the axial direction. In this case a small anisctropy has been observed which can be attributed to the off-centre position of the high voltage terminal. For the ampoule holder used, the expected total variation is about ± 0.03%.

In order to detect some loss of saturation at high current levels a sample of ^{51}V was irradiated, obtaining ^{52}V with an activity higher than 25 mCi. A least square fitting gave for the half-life of ^{52}V:

$$T_{1/2} = (3.7462 \pm 0.0020)\text{min}$$

which agrees with the expected value:

$$T_{1/2} = (3.750 \pm 0.010)\text{min}^{(16)}$$

Even for the highest experimental points, no variations around the fited curve could be attributed to loss of saturation. Therefore the value of 500 Volts of applied voltage in the chamber was satisfactory for current levels less than 3×10^{-8}A (13 mCi of ^{60}Co).

ACTIVITY DETERMINATION

In this work two methods were used for activity determination by the 4π-γ ionization chamber system. The first one, called direct metod, is applied for those radionuclides which have been standardized previously by the $4\pi(\alpha,\beta,X)$-γ absolute systems discussed elsewhere by Moura[14]. In this case, the activity is obtained by means of a Calibration Factor (F) given by:

$$F = \frac{f_n \langle \Delta V/\Delta t \rangle}{A.m}$$

Where:

A = specific activity determined by the absolute system; $\langle \Delta V/\Delta t \rangle$ = average relative currente factor, obtained for the ampoule in the 4π-γ Ionization Chamber System; m = mass of the solution in the

ampoule f_n = normalization factor, obtained by measuring a reference long lived radionuclide (^{60}Co monitor) in the ion chamber system. For other ampoules the unknown activity is given by:

$$A_x = \left(\frac{f_n \langle \Delta V/\Delta t \rangle}{F_x}\right)$$

The term $(f_n \langle \Delta V/\Delta t \rangle)_x$ is obtained by measuring the ampoule in the ion chamber and F_x is extracted from this work. The radionuclides chosen for standardization are: ^{139}Ce, ^{22}Na, ^{60}Co, ^{134}Cs, ^{54}Mn, ^{24}Na, ^{198}Au, ^{241}Am, ^{42}K, ^{57}Co[19] and ^{131}I. Two of these, namely ^{139}Ce and ^{60}Co, were standardized during International Comparisons sponsored by the BIPM[14], [15]. For ^{42}K and ^{134}Cs a minor correction was applied due to the presence of very low concentrations of ^{24}Na(0.15% in ^{42}K) and ^{137}Cs(0.06% in ^{134}Cs). The uncertainties in activity determination were about 0.2% or better. For the normalization factor ^{60}Co was used and the uncertainty amounted to 0.05%. The relative current factors were determined with uncertainties ranging from 0.05% to 0.2%.

The resulting Calibration Factors are listed in Table I. The last columm corresponds to measurements made with a cadmium liner on the ampoule holder. The uncertainties (σ_F) were calculated at the 95% confidence level. The second method for activity determination by the 4π-γ ionization chamber called **indirect method** is used for any radionuclide not standardized previously in the $4\pi(\alpha,\beta,X)$-γ System. In this case the activity is givem by:

$$A = \frac{f_n \langle \Delta V/\Delta t \rangle}{\sum_{i=1}^{n} (\epsilon_\gamma I_\gamma)_i + \sum_{j=1}^{m} (\epsilon_\beta I_\beta)_j}$$

Where:

ϵ_γ and ϵ_β are the relative gamma and beta efficiencies; I_γ and I_β are the absolute intensity of the gamma and beta radiation, extracted from the literature[2,5,6,7,8,9,12]; $\underline{n}$ and $\underline{m}$ are respectively the number of gamma and beta transitions.

The relative beta efficiency curve has been determined with pure beta emitters ^{35}S, ^{204}Tl and ^{32}P, standardized by a 4π-β proportional counter. The chamber response for bremstrahlung is very low, so it was necessary to use solutions of relatively high activities. Therefore two diluted solutions were prepared for each radionuclide. The sistematic uncertainty in the 4π-β absolute system is mainly due to self absorption. It is about 10% for ^{35}S, 5% for ^{204}Tl and 3% for ^{32}P. However, the contribution of beta radiation for the beta-gamma nuclides in most cases is less than 1%, thus this method was considered satisfactory. The interpolation in the beta curve was performed by a simple linear fit between ϵ_β and $E_{\beta max}$. The response for the X-radiation in the case of ^{204}Tl was subtracted using a preliminar ϵ_γ curve. The value of ϵ_β is given by:

$$\epsilon_\beta = a_o + a_1 \, E_{\beta max}$$

where a_o and a_1 are shown in Table II.

The current produced in the chamber is the sum of all partial currents due to individual transitions of a radionuclide. Therefore to obtain the efficiency ϵ_γ for a given gamma energy, it is necessary to subtract the other contributions by an interative procedure. The contribution of beta

radiation was subtracted first, by means of the ϵ_β curve. Using the ionization chamber response for some radionuclides with simple decay scheme as ^{241}Am, ^{139}Ce, ^{198}Au, ^{54}Mn and ^{60}Co it was possible to obtain a preliminary gamma efficiency curve by fitting the experimental points with a 2nd degree polynominal. The final curve was determined subtracting the contribution of the gamma rays of low intensity by means of the preliminary curve. The relationship chosen between the obtained relative gamma efficiency ϵ_γ and the energy (E) is the following:

$$\epsilon_\gamma = \exp\,(a\,E^b)\,[c\,E^d + e\,E^{(f\,|\,n(E/E_o)\,|)}]\,.\,E$$

Table I

Calibration Factors for IG12/A20 Ionization Chamber ($\times 10^8$ V/sec. Bq)

Nuclide	without Cd liner		with Cd liner	
	F	σF(%)	F	σF(%)
^{139}Ce	1.468	0.54	1.105	0.54
^{22}Na	15.69	0.30	15.34	0.30
^{60}Co	16.68	0.32	16.46	0.32
^{134}Cs	11.62	0.60	11.31	0.60
^{54}Mn	6.061	0.52	5.903	0.52
^{24}Na	23.92	0.40	23.60	0.40
^{198}Au	3.303	0.20	3.133	0.28
^{241}Am	0.2542	0.60	0.01577	0.68
^{42}K	1.887	0.36	1.847	0.54
^{131}I	3.120	0.64	2.950	0.86
^{57}Co	1.634	0.80	0.957	1.2

Tabela II

Coefficients for the ϵ_β Curve (V/Beta)

coefficient	without Cd liner	with Cd Liner*
a_o	$(-2.29 \pm 0.12) \times 10^{-11}$	-1.26×10^{-10}
a_1	$(1.994 \pm 0.011) \times 10^{-13}$	1.955×10^{-13}

*fit using the only two available points.

The terms inside the brackets accounts for the behaviour of the gamma absorption cross sections for Argon as a function of the gamma energy. The first one is related to the photoelectric absorption. In this case a linear relationship is assumed between log τ (photoelectric absorption cross section) and log (E). The second term is related to the Compton scatering. In this case a quadratic relationship is assumed between log σ (Compton absorption cros section) and log (E). E_o is the E value

for the maximum log σ value. Outside the brackets, the exponential accounts for the attenuation of the radiation in the chamber walls. The last term E accounts for the number of ion-pairs produced per incident gamma in the chamber gas.

The values of ϵ_γ obtained without the cadmium liner on the ampoule holder are shown in Table III and Figure 3a. Table IV and Figure 3b show the results obtained using the cadmium liner. The uncertainties in ϵ_γ were calculated at the 95% confidence level. Table V shows the parameters a, b, c, d, f, E_o obtained by a weighed least ssquare fit to the function above using the computer program SAS (Statistical Analysis System)[1]. The value obtained for the parameter d is close to the expected value of -2,8 taken from tables of τ versus E. The values of b and f are rather different from the expected values of -2,7 and -0.14. The value of 350 KeV for E_o showed a better agreement than the expected value of 500 KeV. The disagreements may be due to the simplicity of the mode l assumed. A more realistic approach, which would take into account gamma radiation scattering and absorption in the chamber structure as well as secondary electrons from the chamber electrodes, would increase the number of parameters to be fitted. This cannot be done with this restricted number of available experimental points. However, the fitted curve showed residuals smaller than the corresponding experimental uncertainties. Without further information, the uncertainty in the interpolation may be given by the uncertainty in the neighbour experimental efficiencies.

As suggested by many authors[4,11,20], some parameters such as: detection efficiency of the chamber (ϵ_D = pulses detected per emitted gamma ray); average energy transferred to the gas ($\overline{\Delta E}$) and intrinsic fluctuation of the system (σ_i) can be determined by means of the statistical fluctuation in the chamber current.

Tabela III

Relative Gamma Efficiencies (ϵ_γ) for IG12/20 Ionization Chamber (x 10^8 V/Gamma)
Holder without Cd Liner

Energy (keV)	ϵ_γ		
	experimental	fit	absolute residual
59.54	0.6944 ± 0.0052	0.6942	0.00017
123.66	1.685 ± 0.017	1.694	-0.0091
165.85	1.8342 ± 0.0085	1.8309	0.0032
364.48	3.021 ± 0.040	3.045	-0.023
411.80	3.3481 ± 0.0065	3.3501	-0.0020
511.01	4.003 ± 0.029	4.001	0.002
689.30	5.170 ± 0.052	5.147	0.022
834.81	6.062 ± 0.016	6.037	0.025
1173.22	7.928 ± 0.017	7.941	-0.013
1274.54	8.463 ± 0.031	8.471	-0.0088
1332.51	8.762 ± 0.019	8.768	-0.0058
1368.54	8.948 ± 0.044	8.949	-0.0016
2754.03	14.97 ± 0.11	14.89	0.083

For a given preset time (t_o) it is easy to show that:

$$\frac{\sigma_V}{V} = \left[\frac{\overline{\Delta E}\ e}{WC}\right]^{1/2} \cdot \frac{1}{V^{1/2}}$$

Where:

V = voltage across the v capacitor at the time t_o;

W = average energy per ion pair = 26,4 eV for Argon; C = integration capacitance.

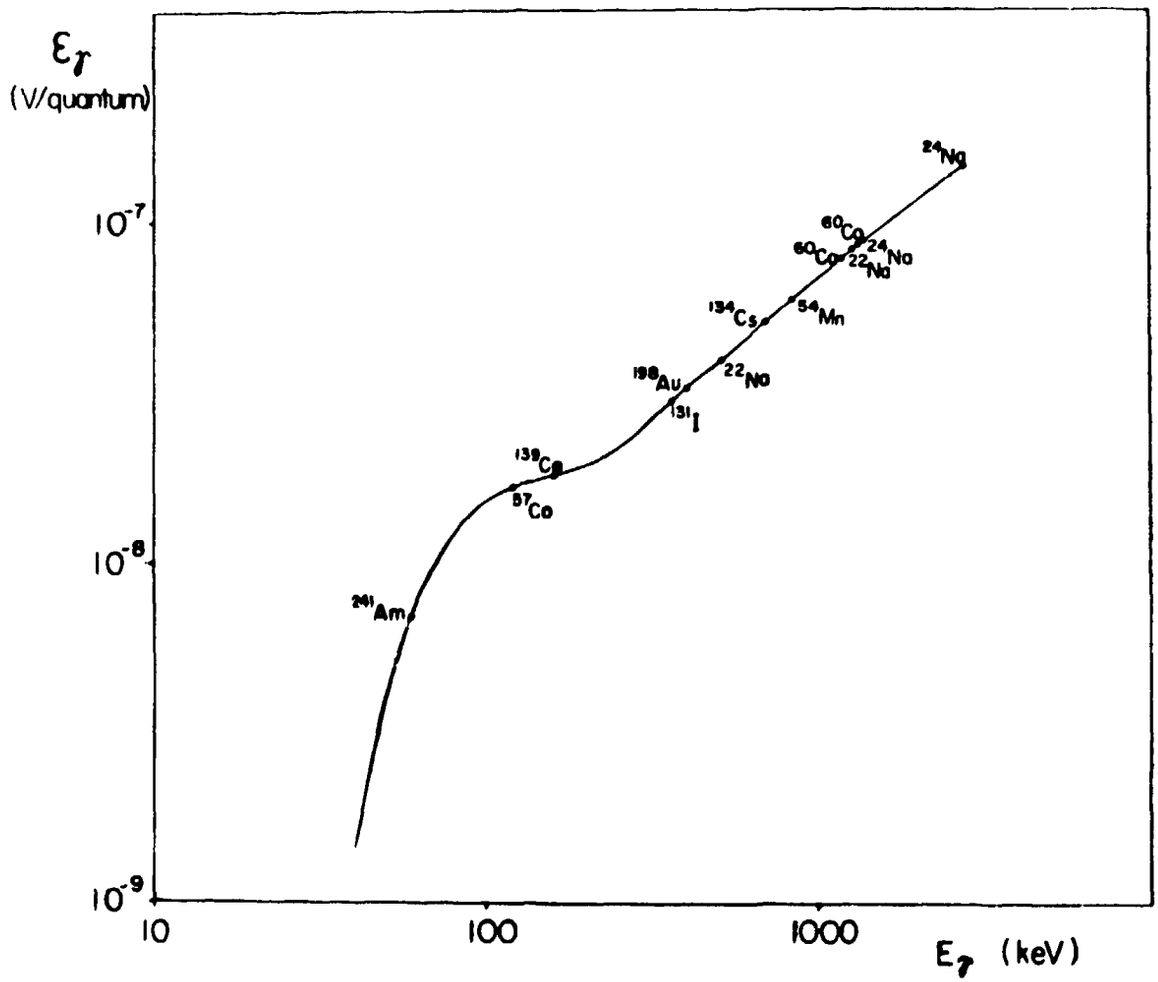


Figure 3a – Relative gamma efficiency curve – without Cd liner

Tabela IV

Relative Gamma Efficiencies (ϵ_γ) for IG 12/20 Ionization Chamber (x 10^8 V/Gama) Holder with Cd Liner

Energy (keV)	ϵ_γ		
	experimental	fit	absolute residual
59.54	0.04099 ± 0.00036	0.04099	0.00000
123.66	0.9148 ± 0.0130	0.9151	-0.0003
165.85	1.3797 ± 0.0054	1.3796	0.0001
364.48	2.862 ± 0.040	2.889	-0.027
411.80	3.209 ± 0.024	3.204	0.005
511.01	3.882 ± 0.031	3.863	0.019
689.30	5.040 ± 0.069	5.017	0.022
834.81	5.904 ± 0.012	5.911	-0.007
1173.22	7.851 ± 0.017	7.826	0.025
1274.54	8.326 ± 0.037	8.360	-0.034
1332.51	8.655 ± 0.019	8.658	-0.002
1368.54	8.791 ± 0.044	8.840	-0.049
2754.03	14.82 ± 0.11	14.81	0.01

$\overline{\Delta E}$ can be taken from the slope of the straight line obtained plotting σ_V/V vs. $1/V^{1/2}$. The extrapolation of this line to zero ($V \rightarrow \infty$) gives σ_i. In this case, the activity goes to infinite so the expected fluctuation is zero. The detection efficiency is given by:

$$\epsilon_D = \frac{V/(A\, I_\gamma\, t_o)}{\sigma_v^2 / V} = \frac{\epsilon_\gamma}{\sigma_v^2 / V}$$

Where:

ϵ_γ is the relative gamma efficiency already mentioned and σ_v^2/V is the square of the slope of σ_v/V vs. $1/V^{1/2}$. Two radionuclides, namely ^{60}Co and ^{203}Hg were chosen to determine these parameters at two different gamma energies. Using a source of long half life and changing the value of $\underline{V}$ with the aid of the discriminators, it is possible to obtain the parameters in a easy way. The results are shown in Figure 4. The values of ϵ_D and $\overline{\Delta E}$ are presented in Table VI. Although the extrapolated value gives $\sigma_i \sim 0.01\%$ for both cases, this was not observed because of instabilities at the discriminators for very low values of $\underline{V}$ or t_o. Both values of ϵ_D for ^{60}Co and ^{203}Hg agreed within the uncertainty, being a good check of consistency. Using the average value of ϵ_D it is possible to determine $\overline{\Delta E}$ for other gamma energies by the relation:

$$E = \frac{W\, C\, \epsilon_\gamma}{e\, \bar{\epsilon}_D}$$

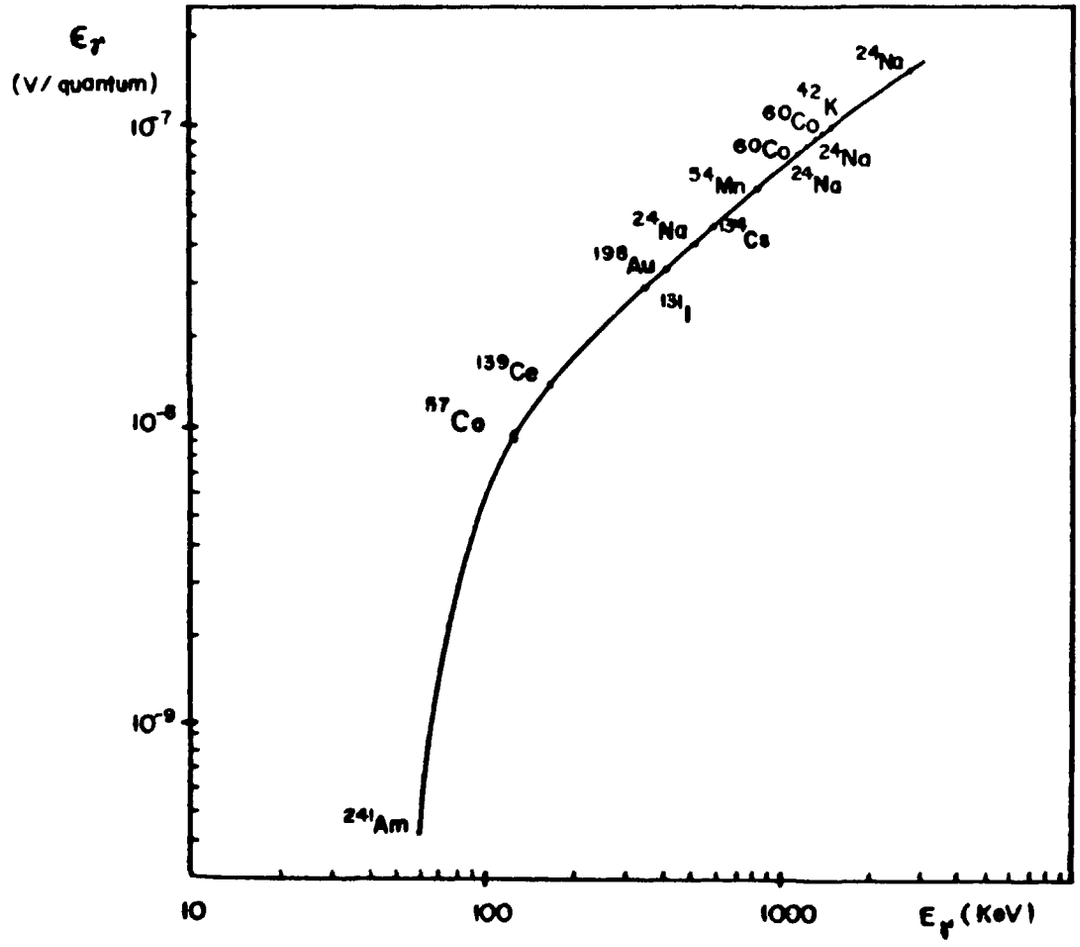


Figure 3b – Relative gamma efficiency curve – with Cd liner

Tabela V

Fitted Parameters of the ϵ_γ Curve

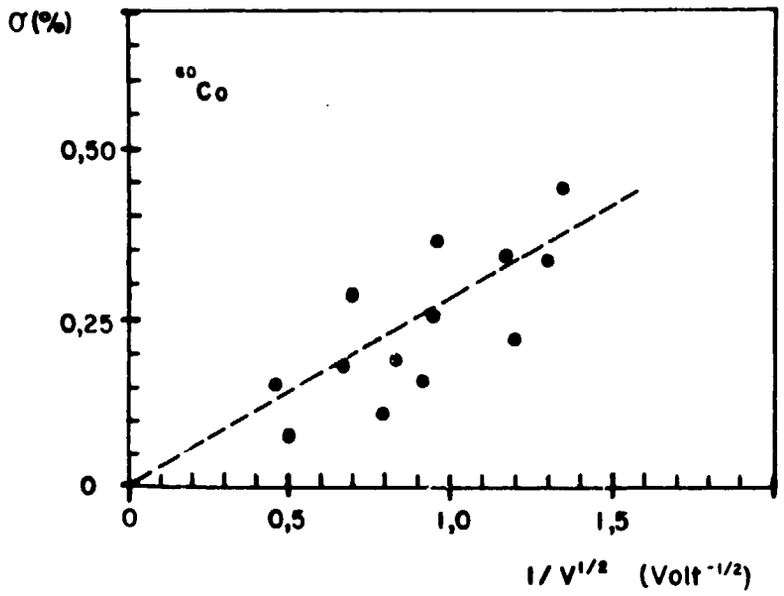
parameter	without Cd liner	wit Cd liner
a	-708.803	-2653.20
b	-1.308100	-1.47207
c	3.6163×10^{-4}	3.1619×10^{-4}
d	-2.83597	-2.73725
e	9.5695×10^{-11}	9.4263×10^{-11}
f	-3.3655×10^{-2}	-3.3082×10^{-2}
E_o	350	350
weighted residual	4.909	5.635

Tabela VI

Detection Efficiency (ϵ_D) and Average Energy Transferred to the Chamber Gas ($\overline{\Delta E}$) for ^{203}Hg and ^{60}Co

Nuclide	ϵ_D	$\overline{\Delta E}$ (keV)
^{203}Hg	$(1.05 \pm 0.05) \times 10^{-2}$	147
^{60}Co	$(1.08 \pm 0.18) \times 10^{-2}$	423

*ϵ_D = pulses detected per emitted gamma-ray



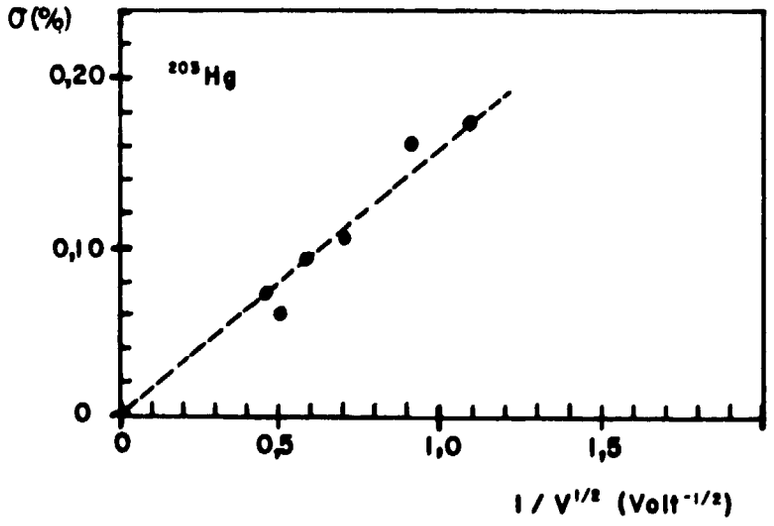


Figure 4 – Plots of $\frac{\sigma_V}{V} \times 100$ (= σ (%)) vs. $1/V^{1/2}$ for ^{60}Co and ^{203}Hg.

Table VII shows the values of $\overline{\Delta E}$ for some other energies.

Tabela VII

Average Energy Transferred to the Chamber Gas for Different Primary Gamma Energies

Energy (keV)	$\overline{\Delta E}$ (keV)	Energy (keV)	$\overline{\Delta E}$ (keV)
59.54	34.8	834.81	302
165.85	81.3	1274.54	412
411.80	166	2754.03	739

CONCLUSIONS

Using the data obtained in this work the ionization chamber system installed at the LMN can be used as a reference system for activity determination of radioactive solutions.

By the direct method the solution to be standardized is compared (by means of a monitor) with a solution measured by the $4\pi\beta$-γ coincidence system. This method can be applied for ^{139}Ce, ^{22}Na, ^{60}Co, ^{134}Cs, ^{54}Mn, ^{24}Na, ^{241}Am, ^{42}K, ^{131}I and ^{57}Co. In this case the uncertainy ranges from 0.4 to 0.8%. Other radionuclides can be measured by using the indirect method by means of the relative beta and gamma efficiency curves. In general the uncertainy is greater than in the direct method and depends upon the energy range to be used. The fitted gamma efficiency curve showed residuals smaller than the corresponding experimental uncertainties. Without further information, the uncertainty in the interpolation may be given by the uncertainty in the neighbouring experimental efficiencies. An additional check can be made by comparing the results obtained with and without Cd liner on the ampoule holder.

The lower activity limit is restricted to the background which is equivalent to 0.05μCi of ^{60}Co. There was no noticeble loss of saturation in the chamber current up to 13 mCi of ^{60}Co using an applied voltage of 500 V.

Some additional parameters of the system as detection efficiency, average energytransferred to the chamber gas and intrinsic fluctuation have been determined by measuring the statistical fluctuation of the chamber current. Consistent data were obtained at two gamma-ray energies: 1.25 MeV (^{60}Co) and 0.279 MeV (^{203}Hg). Appart from the measurements performed in this work, the ionization chamber system can be used for gravimetric comparisons, for check ups of dilution factors and masses determined previously by microbalances. In these cases the estimated uncertainty is about 0.1%.

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RESUMO

Um sistema de câmara de ionização tipo 4π-γ foi calibrado para determinações de radioatividade de amostras líquidas. A curva de eficiência foi determinada experimentalmente para fótons no intervalo de energia de 60 KeV a 2750 KeV. Os valores experimentais foram ajustados a uma função analítica da energia e uma concordância foi observada em todo o intervalo de energia.

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