ABSOLUTE MEASUREMENTS OF THE ACTIVITY OF RADIONUCLIDES

I - The Defined Solid Angle Method with Geiger Müller Counters

Dagmar C. C. Reis and Lais P. de Moura
Absolute Measurements Laboratory - Nuclear Physics Division
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RESUMO

No presente trabalho, os autores descrevem o sistema de medidas absolutas de atividades de radionuclídeos por meio de contadores Geiger Müller, com ângulo sólido definido em relação à fonte. O sistema empregado no Laboratório de Medidas Absolutas da Divisão de Física Nuclear do IEA é objeto de discussão pormenorizada e é dada ênfase especial à necessidade de fixar a geometria do sistema e o tempo morto independentemente do contador.

Apesar de sua precisão ser limitada, o sistema apresenta vantagens pela sua simplicidade e estabilidade e é suscetível de fornecer resultados de apreciável precisão desde que os diversos fatôres que afetam as medidas sejam determinados cuidadosamente.

RESUMÉ

Dans ce travail est presentée la méthode du compteur Geiger Müller avec angle solide défini, pour la mesure absolue de radionuclides. On décrit le système utilisé au Laboratoire de Mesures Absolues de cet Institut, dans lequel la géometrie et le temps mort son définis indépendamment du compteur. D'autre part, on analyse soigneusement les facteurs qui affectent l'efficacité totale du systême.

Bien que la précision de cette méthode soit limitée, elle est cependant très suffisante pour des mesures de routine , étant donnée sa simplicité.

ABSTRACT

In this paper a detailed account of the defined solid angle method with Geiger Müller counters is presented, as used at the absolute measurements laboratory of this Institute. A system is described in which both the geometry and dead time are defined independently of the counter and the influence of the different factors which affect the overall efficiency is analysed.

Because of its reliability and simplicity the method is largely used for routine measurements, in spite of its limited precision.

INTRODUCTION

There is a wide field of nuclear research in which the absolute disintegration rate of a radioactive source need only be known approximately in order to design experiments and in which only relative measurements need be made with any degree of precision.

There are, however, a few important applications which demand a precise knowledge of the disintegration rate.

As a general rule, those are the problems in which the radiations emitted by a source are used as projectiles and not merely as units to be counted. Problems such as the determination of the neutron flux, the absolute measurement of the number of neutrons or gamma rays emitted by a source, nuclear cross-sections measurements, the determination of the disintegration constants of radioactive nuclides, fission product studies and so on. In the problems of nuclear energy, where a precise knowledge of the nuclear constants of several materials used in moderators, fuel elements, structural materials and others, the influence of small uncertainties in the value of those parameters can affect seriously the results of the calculations and reactor design.

Due to the complexities involved in the precise determination of the absolute disintegration rate and the consideration that there is already a wide field of activities which require the determination of the counting rate of a source with a fairly good degree of accuracy, such as its applications in soil and fertilizer research, in measurements of the metabolic rate of isotopes or labeled molecules etc., the need has arisen of fabricating reference standard sources of several nuclides. Since through the use of such standard sources accurate measurements of the disintegration rate of an unknown source can be made with a end window Geiger Müller counter if special precautions are taken, a detailed knowledge of the physical and geometric factors which influence such measurements is necessary.

The absolute measurement of the disintegration rate of a radioactive nuclide has the main purpose of obtaining, with the maximum possible accuracy, the specific disintegration rate of the isotope under study.

Whenever a radioactive source is being measured in a counting system it is imperative that the efficiency of the system be known accurately. Such an efficiency can be evaluated by the knowledge of several factors which influence the measurements and which can be fairly well calculated when the characteristics of the measuring system and the disintegration scheme of the radionuclide are known. Whenever a very high degree of accuracy is wanted, however, the use of special and delicate techniques used in the preparation of the sources, of particle detectors specially designed for that purpose and the use of suitable electronic systems, allow measurements with an efficiency much higher than the one obtained with the conventional Geiger Müller counter system.

The most suitable method of measurement to be used for that purpose depends on the properties of the nuclide under study and a previous knowledge of its disintegration scheme is imperative.

For the measurement of beta active nuclides, the use of the method of the defined solid angle with Geiger Muller counters, described in this paper, will allow measurements to be made with an accuracy between 5 and 10% under the best experimental conditions; its main merit is, however, its extreme simplicity and the high accuracy of the results which can be obtained when only relative measurements are the main purpose of an experiment.

The use of a 4π proportional counter shows a much higher accuracy (between 1% and 3%) but the measurements require special techniques for the preparation of the sources.

When the nuclide under study emits beta rays followed by gamma rays the use of the method of coincidences between the beta and the gamma rays, as measured in a 4π beta-gamma system gives the highest accuracy obtainable to date (of the order of 0.1%),but it involves the same care in the preparation of samples which is required for the 4π counting systems.

The properties and main characteristics of the proportional 4π counting system, of a 2π beta-gamma coincidence system and the techniques used in the preparation of sources and in the preparation of thin films for source deposition, used at the IEA will be described in other publications.

GEIGER-MÜLLER COUNTER WITH DEFINED GEOMETRY

In the measurements of activities with a Geiger Müller end window counter, there are several factors which must be taken into account in order that the absolute activity of the source can be evaluated. Among those, the geometry factor is the most difficult to be evaluated due to uncertainties in the determination of the sensitive volume of the counter. One way to overcome this difficulty is to allow only a cone of particles to reach the counter in the neighbourhood of the wire(1). The geometry of the system can be very accurately known and the uncertainties due to the sensitive region of the counter eliminated by providing the counting system with a circular diaphragm provided with a central circular hole, of a diameter somewhat smaller than that of the counter, located between the source and the counter window, so as to allow only particles within a narrow cone to reach the counter window. Such a baffle plate is usually made of brass and its thickness should be sufficiently large to absorb all the beta rays emitted by the source which should not reach the counter sensitive area.

Another source of error are the losses due to the dead time of the counter, since it is known that it varies under the influence of factors such as the gas pressure inside the counter, the nature of the gases used in filling the counter, the value of the potential applied between the counter cathode and anode, the age of the counter and also the counting rate. Since the correction of those factors is difficult, the electronic counting circuit is usually provided with a gate that blocks the counter operation during a time interval not smaller than its dead time.

EXPERIMENTAL ARRANGEMENT (fig. 1)

The Geiger Müller tube used is a type 18526 (Philips), of 27 mm of diameter and provided with a mica window of 1,8 mg/cm^2. The counter is mounted inside a lead castle of 55 mm thickness, lined with 1 mm thick aluminium. The system is provided with an aluminium rack in which the baffle plate and the source holder can be placed in various positions, 1 cm apart from each other (fig.2). The 1,6 mm thick brass diaphragm is provided with a carefully machined hole of 18 mm diameter, and is located at 1 cm from the counter window (our lead castle is a standard Lemer Nantes, of the same type made for the "Commissariat à l'Énergie Atomique").

It is important to bear in mind that a precise evaluation of the geometry factor depends critically on the diameter of the diaphragm hole, an error of 0,1 mm in the value of its radius giving rise to an uncertainty of 2,5 % in the above factor.

ELECTRONIC SYSTEM

The electronic system used is schematically represented in figure 3. It consists essentially of:

- Stabilized power supply (250 V. D.C.) type TALS.
- Electronic timer, with a provision for pre-set time and pre-set count measurements (TTCP1).
- Scaler T2D1.
- Geiger circuit TEGM-1.
- Pre-amplifier for the Geiger tube (GMP 3).

The unit TEGM-1 consists of the following main circuits:

- A monostable trigger circuit to define the dead time (overall).
- A circuit for lowering the potential applied on the wire after a pulse is counted.
- A high voltage stabilized circuit.
- An output circuit to operate the mechanical register.
- A control circuit.

The main feature of this circuit is to help the quenching of discharge by lowering the potential applied in the counter wire (anode) during a pre-set time and to block the scaler input circuit during the same time interval. Since the potential drop at the tube anode is of about 400 volts, the Geiger Müller counter becomes insensitive to any ionizing radiation which might enter the tube in this time interval. The dead time can be varied at will according to the following values: 100, 250, 500 and 1000 microseconds.

In our measurements, the dead time is usually fixed in 500 microseconds, since the tube dead time is of about 200 microseconds.

CORRECTION FACTORS

The real disintegration rate of a source can be evaluated by dividing the measured counting rate of the sample by the overall efficiency of the counting system.

$$D = C \cdot \mathcal{E}^{-1}$$

$\mathcal{E}$ can be calculated$^{(9)}$ from the equation

$$\mathcal{E} = e_{\beta} \; f_G \, f_m \, f_{\tau} \, f_w \, f_b \, f_s$$

where

e_{β} is the intrinsic efficiency of the detector for beta particles.

f_G is the geometry factor for the counting system.

f_m is the factor due to multiple discharges in the Geiger counter.

f_{τ} is the factor corresponding to the losses due to the dead time of the counter.

f_w is the factor corresponding to the absorption of the beta ray energy in the air layer between source and counter window and the window absorption.

f_b is the back-scattering factor due to the source support.

f_s is the self absorption factor due to the finite thickness of the source.

There are other factors which are not taken in consideration due to their smallness when the counter system is lined with materials of a low atomic number, such as is the case in lead castles lined with aluminium or lucite and when the same materials are used in the construction of the source mounting trays and racks. For the above reason, the influence on the overall efficiency due to air scattering, scattering in the supporting frames and so on are neglected.

In the following lines the influence of the correction factors will be discussed with some detail.

a) Geiger Müller counter efficiency

For beta rays, of the energies usually found in radioisotope activity measurements, the efficiency of the Geiger counter is taken as

$$e_{\beta} = 1$$

This value is a very good approximation for counters filled with a gas pressure not smaller than 10 cm Hg of such an active length that the incoming particle travels not less than 2 cm through the sensitive volume of the counter which is assumed to show a good plateau.

b) Geometry factor

Although a source emits radiation with spherical symmetry (this is true only when both the source and its supporting film are of negligible thickness), only the rays emitted within the solid angle subtended by the source and the detector and defined by the diaphragm are detected.

The geometry factor f_G is defined as the solid angle subtended between the counter and the source.

By assuming that the emission of the radiation by the source is spherically symmetrical about the source, its value gives the fraction of the emitted rays which can enter into the sensitive volume of the counter (neglecting the influence of other factors which will be taken into consideration later).

For a point source or for a source with a negligible diameter (2), the geometry factor is given by

$$f_G = \frac{\Omega}{4\pi}$$

The solid angle can be calculated from the following expression, in which polar coordinates are used

$$\Omega = \int_0^{\alpha}\int_0^{2\pi} \operatorname{sen}\theta \, d\theta \, d\phi = 2\pi \int_0^{\alpha} \operatorname{sen}\theta \, d\theta$$

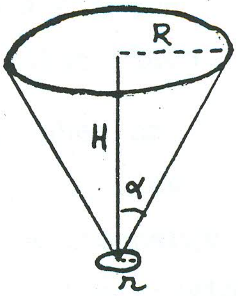
$$\therefore \; f_G = \frac{2\pi}{4\pi}\int_0^{\alpha} \operatorname{sen}\theta \, d\theta = \frac{1}{2}\,(1 - \cos\alpha) =$$

$$= \frac{1}{2}\left[1 - \frac{H}{(H^2 + R^2)^{1/2}}\right]$$

where H is the distance between the source and the circular diaphragm and R is the circular hole radius.

For sources which cannot be considered as point sources the effect of its finite radius must be taken into account. The geometry factor can be calculated by a series which converges if γ is small$^{(2)}$:

$$f_G = \frac{1}{2}\left[1 - \frac{1}{(1+\beta)^{1/2}} - \frac{3}{8}\,\frac{\beta\gamma}{(1+\beta)^{5/2}} - \gamma^2\left(-\frac{5}{16}\cdot\frac{\beta}{(1+\beta)^{7/2}} + \frac{35}{64}\cdot\frac{\beta^2}{(1+\beta)^{9/2}}\right) - \gamma^3\left(\frac{35}{128}\cdot\frac{\beta}{(1+\beta)^{9/2}} - \frac{315}{256}\,\frac{\beta^2}{(1+\beta)^{11/2}} + \frac{1155}{1024}\cdot\frac{\beta^3}{(1+\beta)^{13/2}}\right)\right]$$



where $\beta = \frac{R^2}{H^2}$ $\quad \gamma = \frac{r^2}{H^2}$

In fig. 4, one can see the variation of the geometry factor with the distance between the source and the diaphragm.

c) Factor due to multiple discharges in the Geiger Müller tube

The factor f_M takes into account the increase in the counting rate due to multiple discharges in the Geiger Müller tube. It is known that this factor increases with the excess of voltage above the counter threshold, with the counter age (due to the decomposition of the quenching gas molecules under electrical discharges) and with the counting rate, since it is of a statistical nature; it is the main factor responsible for the slope of the Geiger counter plateau. New counters with plateau showing a negligible slope should be used and the high voltage applied to the counter anode should be as stable as possible, in order to minimize the influence of those spurious discharges; under the above hypothesis we could take

$$f_M = 1$$

as a very good approximation. The counting rates of the source should not be so large as to introduce errors due to its dead time, since the observation shows that, under those conditions, the probability of multiple pulse formation is negligible.

d) Factor due to the dead time of the counter

The dead time of a counter is defined as the minimum time interval required by the counter to start a new avalanche after a discharge initiated by another particle has taken place.

During the time in which the electron avalanches are occuring, the counter is obviously insensitive to any ionizing particle which traverses its sensitive volume. After a discharge has taken place, the counter anode potential raises to its previous value exponentially with the time and it may occur that the field will not be fully restored before another particle produces a few ion pairs in the counter. Due to the presence of the positive ions and/or the time required for the anode to reach its former value, the pulse formed under those conditions will have a smaller amplitude - so that the counting rate registered will be dependent on the gain of the amplifying system. In order to avoid this source of error, it is usual to provide the counter with a quenching system which will lower its anode potential during a time larger than its intrinsic dead time.

It is also important to bear in mind that there are other parts of the counting circuit which show a finite resolution time which should be taken into account. In order to eliminate such sources of correction factors, it is usual to use time constants in the electronic circuits smaller than the total dead time of the counter. Under those hypothesis, the only correction required is the one due to the finite dead time of the counter.

Let D dis./sec. be the true counting rate of the source (that is, the real number of particles emitted by the source under the solid angle subtended by the counter; this number would be equal to the number of particles registered if no losses were present).

The time during which the counter is insensitive is $C\tau$ per second. The number of particles lost in each second is $DC\tau$; but this number is equal to D-C(4)

$$D - C = D\,C\,\tau$$

where C is the number of registered particles. We have then

$$D = \frac{C}{1 - C\tau}$$

There are several ways by means of which the resolution time of the system can be measured; among those deserve special consideration the methods of direct measurement of the dead time by means of an oscilloscope, the method of paired sources, the method of the areas and the method of the radioactive decay of a strong source of short half-life. The last two are the most interesting since they afford a simple way to determine the resolution time for sources of different disintegration rates.

I. Method of the Radioactive Decay of a Source

Since the activity of an isotope decays exponentially with the time, a plot of the logarithm of the recorded intensity against time should appear as a straight line. However, if the source intensity is high enough, the recorded counting rates are smaller than what they should be owing to the finite resolution time of the equipment. As a result, the observed points lie below the straight line extrapolated towards the time $t = 0$. The dead time of the system can be computed by taking into account the differences between the observed and the true (extrapolated) counting rates.

In order to determine the resolving time by this method, one should use a strong source (of the order of one microcurie) of an isotope of short life. Fig. 5 shows the results obtained with the decay of I^{132}.

II. The Method of Increasing Areas

If one considers a source uniformilly spread over an area, the specific counting rate (that is, the counting rate per unit of area) should be a constant. Therefore, by increasing the area of the source by known amounts, its activity should be proportional to the total area of the source.

The recorded counting rate, however, follows this rule as a first approximation only, since a correction due to the finite size of the source should be taken into account.

In order to perform those measurements, it is important to use radioactive sources with a fairly long half life, such as UX2 in equilibrium from a uranium oxide source. The results obtained with this source are represented in fig. 6.

Since the dead time of the counter depends on its counting rate and can vary with the time, it is a regular procedure to use an electronic circuit to make it a constant (usually slightly larger than the maximum dead time observed by one of the above methods).

e) Absorption factor due to the air layer between the source and the counter and the influence of the counter window.

Before entering into the sensitive volume of the counter, a particle emitted by the source will travel through the intervening air layer and the counter window before it can give rise to a pair of ions in the counter filling gas.

Since the total equivalent thickness is small, the absorption can be calculated with a good approximation by the formula

$$f_W = e^{-\mu s_t}$$

where μ is the mass absorption coefficient (in cm^2/mg) and s_t is the total thickness of the absorbers (air plus window)(in mg/cm^2). The absorption coefficient can be evaluated from empirical expressions:

$\mu = 0{,}022\ (Em)^{-1{,}33}$ $\quad 0{,}5 < Em < 6$ Mev ref. (13)
$\mu = 0{,}017\ (Em)^{-1{,}45}$ $\quad 0{,}15 < Em < 3{,}5$ Mev ref. (1)
$\mu = 0{,}0155\ (Em)^{-1{,}41}$ ref. (8)
$\mu = 0{,}0119\ (Em)^{-1{,}83}$ ref. (12)

where Em is the maximum energy of the beta rays in Mev.

For measurements in which a higher precision is required, however, it is better to measure f_w directly by interposing aluminium absorbers, since the mass absorption coefficient (in cm^2/mg) is practically the same for aluminium, air and mica. In those measurements it is important to place the absorbers as near to the counter window as possible in order to avoid errors due to the scattering of the beta particles in the aluminium absorber.

If a semi-log plot of the counting rate against the absorber thickness is made, the activity due to zero absorber thickness can be obtained by extrapolation. (fig. 7)

The f_w values calculated by the approximated expression low $f_w = e^{-\mu s_t}$ and the values obtained from the absorption curves for energy β-emitters show a significant difference, as can be observed in the following table:

		S^{35} (Em = 0,167 Mev)		Co^{60} (Em = 0,312 Mev)		$Sr^{90} + Y^{90}$ (Em = 0,545 Mev, 2,26 Mev)	
shelf	air + + window thickness (mg/cm^2)	f_w calc.	f_w from curve	f_w calc.	f_w from curve	f_w calc.	f_w from curve
3	5,414	0,276	0,397	0,571	0,611	0,863	0,861
5	7,823	0,156	0,239	0,445	0,463	0,813	0,805
7	10,233	0,088	0,147	0,346	0,389	0,766	0,764

f) Back scattering factor

As the source under measurement is distributed evenly on a film support, it may happen that particles which normally would not reach the counter due to its direction of emission, can be scattered by the source support and penetrate the counter (Rutherford scattering).

The backscattering factor is, therefore, larger than one (usually its value is between 1 and 2, depending on the thickness and on the atomic number of the source supporting film).

Since f_b changes rapidly with the support film thickness, it is usual to employ either films of negligible thickness in order that $f_b = 1$, or infinitely thick (as compared with the particles range in the material) in order that the saturation backscattering factors found in tables can be used.

Backscattering is estimated from the results of Zumwalt(9) and Cervellini(14) whose curves are reproduced in fig. 8.

If one assumes for those corrections that the backing material is thin, then the percentage of the backscattered beta rays is a function only of its thickness in terms of an absorption half-thickness and, under those assumptions, the known results for polystyrene can be applied to any material.

g) Factor due to self-absorption

Since the sources always have a finite thickness, there are losses due to the absorption of emitted rays by the source material located between the disintegrating atom and the counter. This influence is greater when the maximum energy of the beta rays is small.

Let C_o be the counting rate per unit time under the hypothesis that the self absorption can be neglected. If one supposes that the thickness s of the source is uniform, the activity which goes through a layer of thickness dx at a distance x from the source will be:

$$dc = \frac{C_o \quad e^{-\mu x} \quad dx}{\rho}$$

The activity of the source at its surface will be

$$C = \int_0^{\rho} dc = \frac{C_o}{\mu\rho}\left(1 - e^{-\mu\rho}\right)$$

$$\therefore \; f_s = \frac{C}{C_o} = \frac{1}{\mu\rho}\left(1 - e^{-\mu\rho}\right)$$

f_s can be directly measured by a number of diluted samples in which the inactive material and the source material are mixed in such a way that the total activity is maintained.

For absolute measurements, however, it is recommended that special source preparation techniques be used so as to keep $f_s = 1$: the use of carrier-free sources and a small amount of a wetting agent such as insulin will usually provide sources of a high specific activity with a negligible self absorption. If a thin support film is used, the influence of the two last factors can be neglected.

PRELIMINARY MEASUREMENTS

In order to check the conditions of operation of the counting system, there are some measurements which should be made periodically.

Plateaux The working condition of the Geiger Müller tube can be controlled periodically by determining its plateau (the plot of the counting rate against the applied high voltage on the counter wire) and observing its length and slope (fig. 9). As it is well-known, counters with deteriorated quenching mixture or showing inhomogeneities in the wire surface will give rise to a short plateau and a high slope due to the formation of multiple discharges.

Statistical distribution A source of long half-life is used and several measurements are made (obviously, the larger the number of such measurements the better will be the meaning of this test). When a source is not very active, the observed data will fit a Poisson distribution, whereas for strong sources a Gauss distribution is found. If a deviation of the statistical distribution is found, the equipment is not working properly.

Background All the counting systems show a certain background counting rate due to a number of causes such as cosmic rays, room and air or walls radioactivity due to natural radio-emitters, and contamination in the support, trays and walls of the counter.

The background counting rate is usually decreased by shielding the counter and source with a lead castle, lined with aluminium, lucite or other materials of low atomic number whose function is to absorb any ionizing particles from the lead contamination and to minimize scattering.

Periodical tests of the background counting rate will show the casual presence of radioactive contamination in the counter system or a disturbance in the operating conditions of the pulse amplifying and registering systems.

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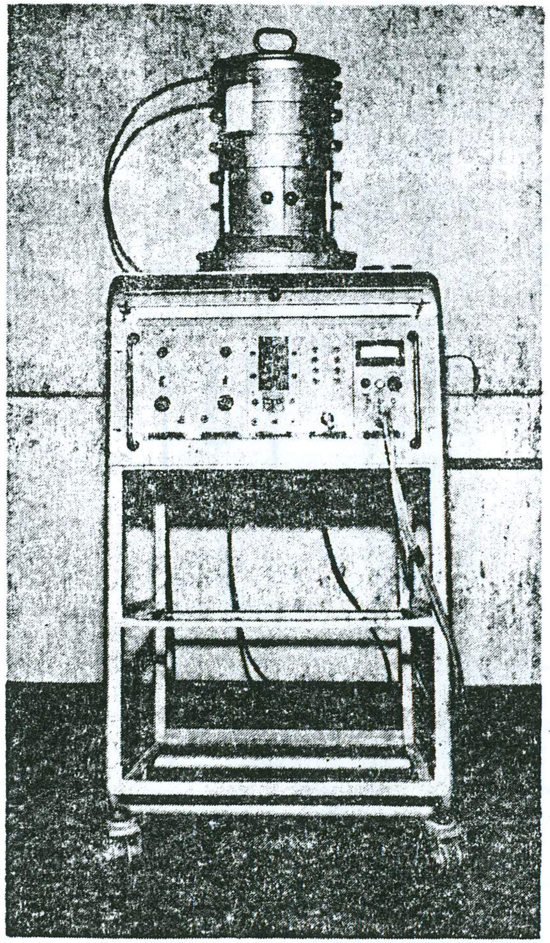


Fig 1) GEIGER MÜLLER ASSEMBLY WITH DEFINED GEOMETRY

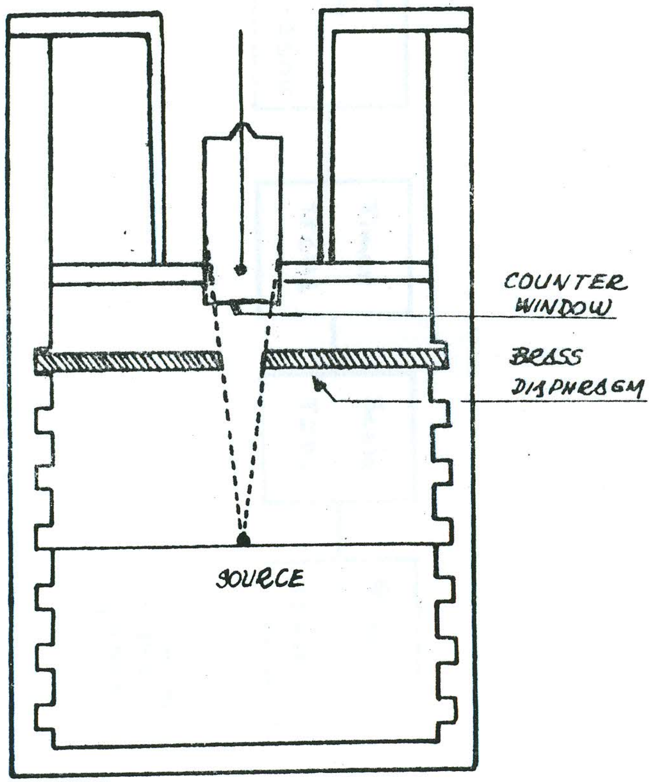


Fig 2) DESIGN OF THE SYSTEM

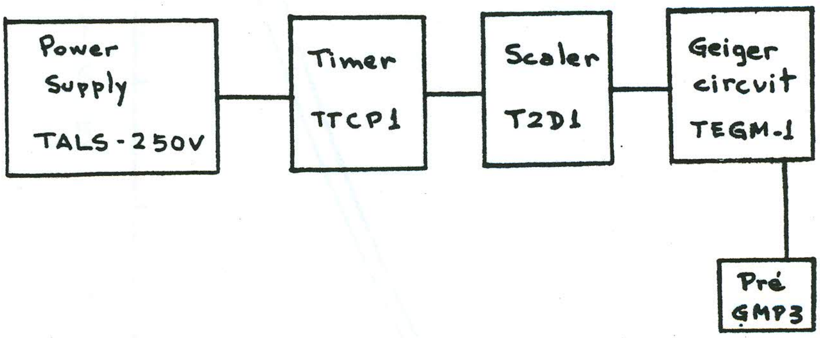


Fig. 3a) Block diagram of the electronic system

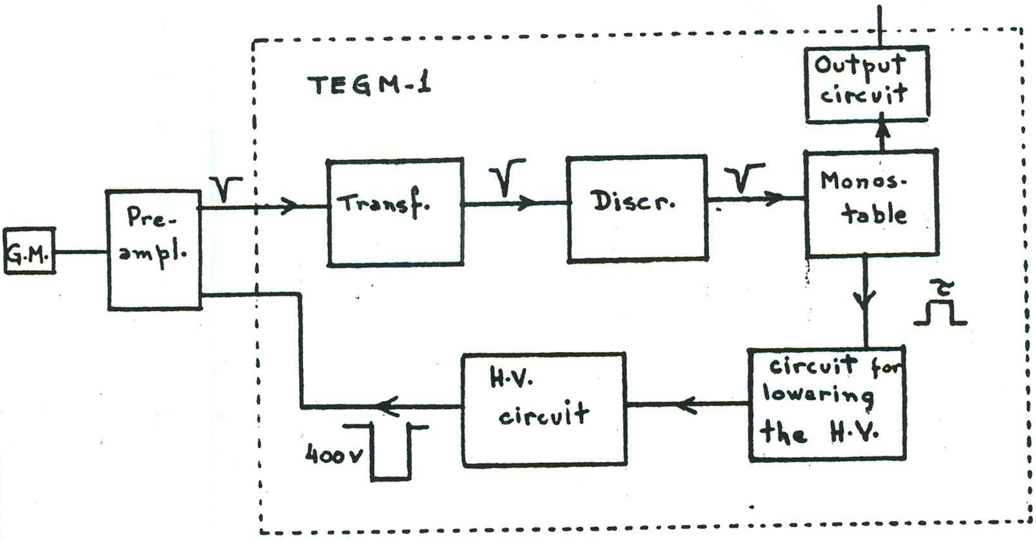


Fig. 3b) TEGM - 1 unit

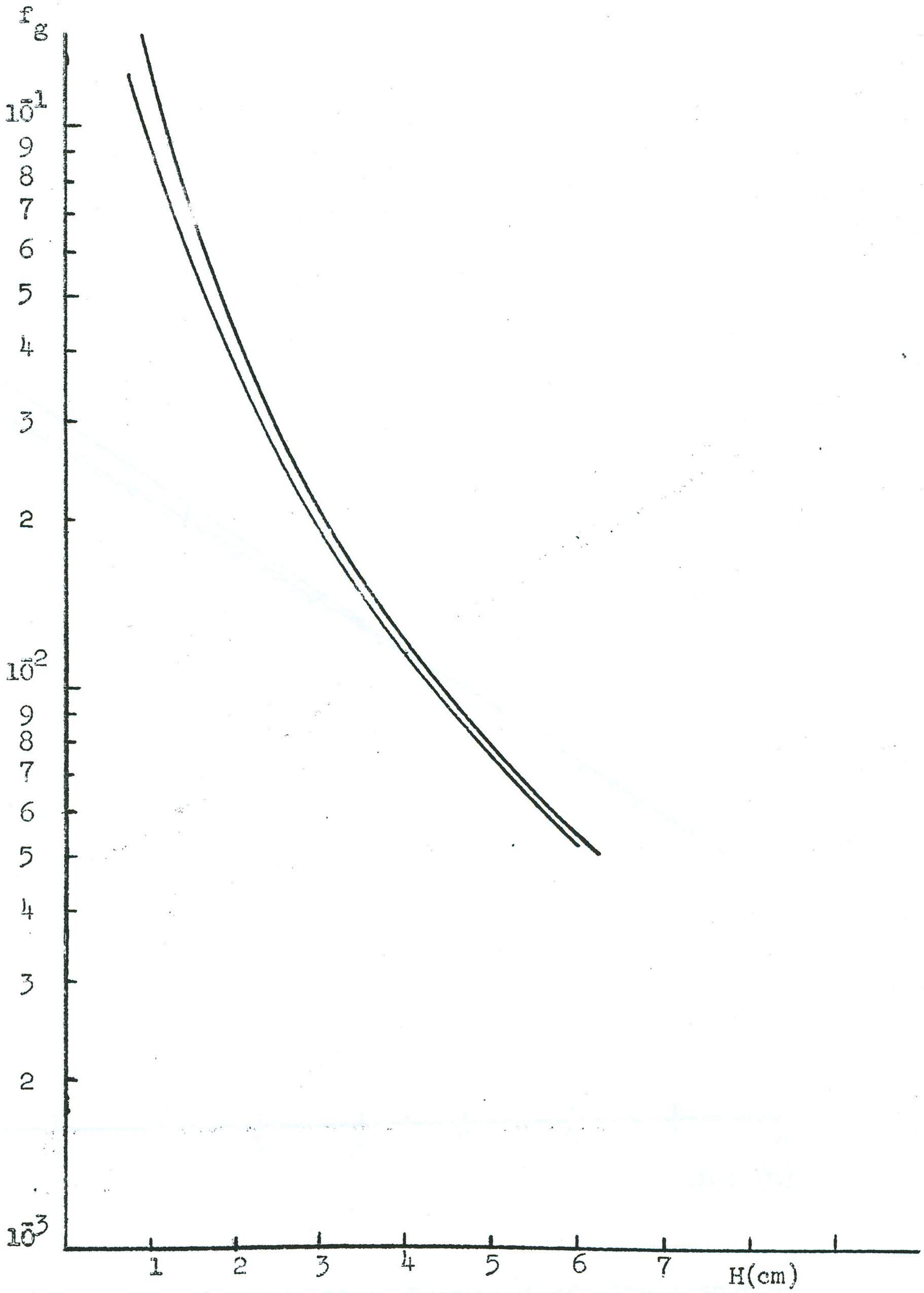


Fig. 4) Geometry factor against distance between the source and diaphragm (for different source radii)

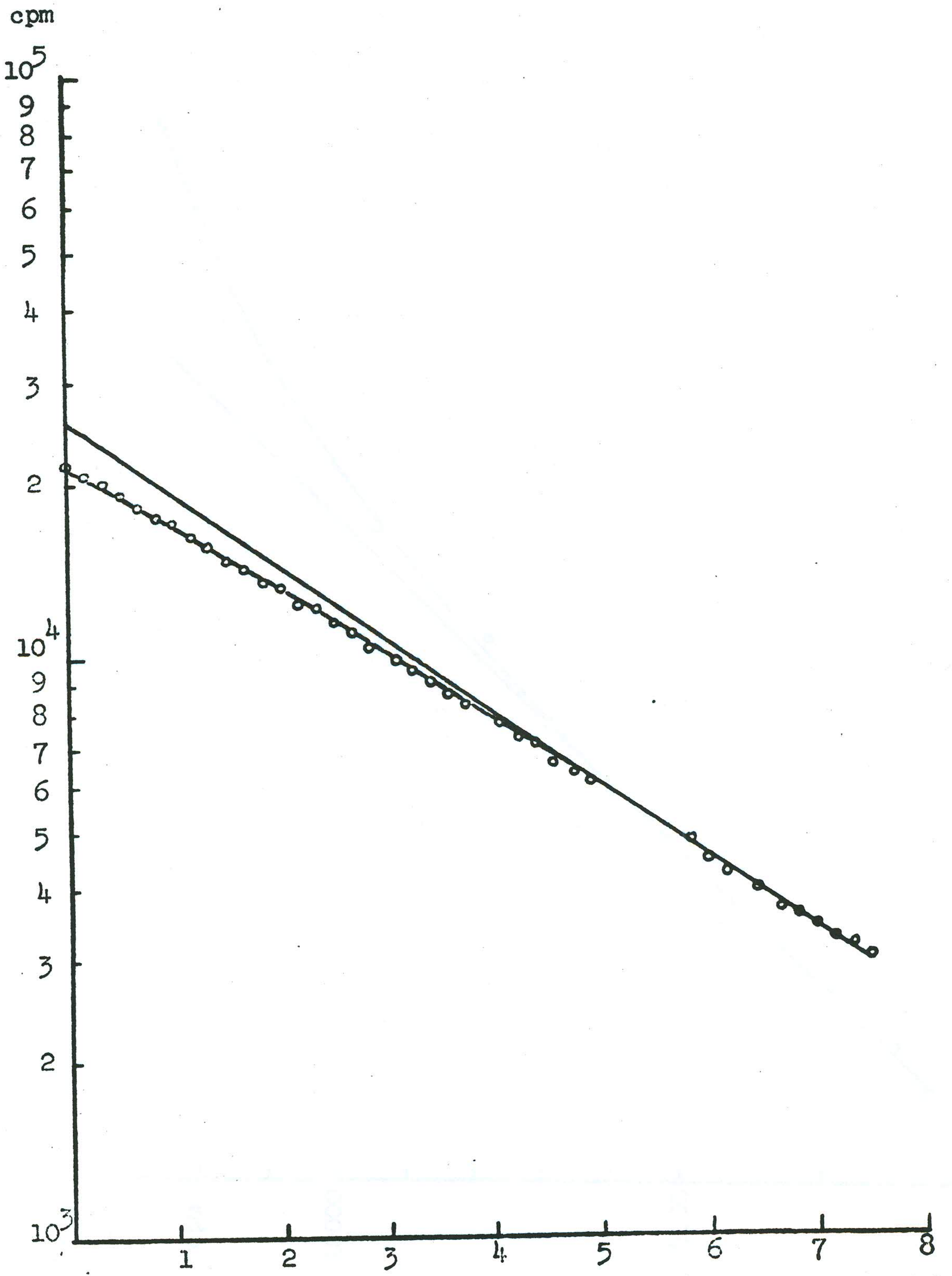


Fig. 5) Dead time determination. Decay method with a source of I^{132}

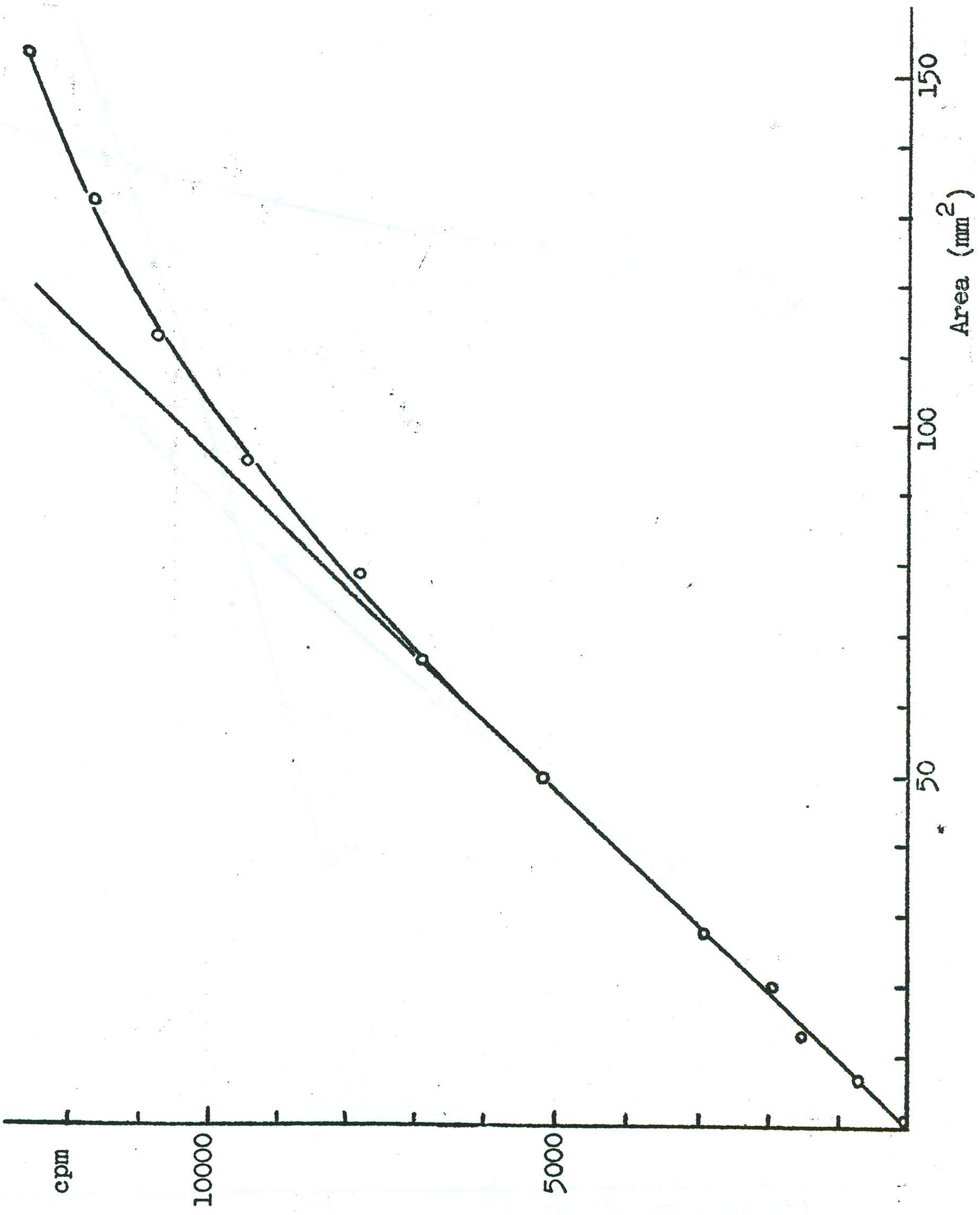


Fig. 6) Dead time determination. Method of the areas.
Sources: uranium oxide

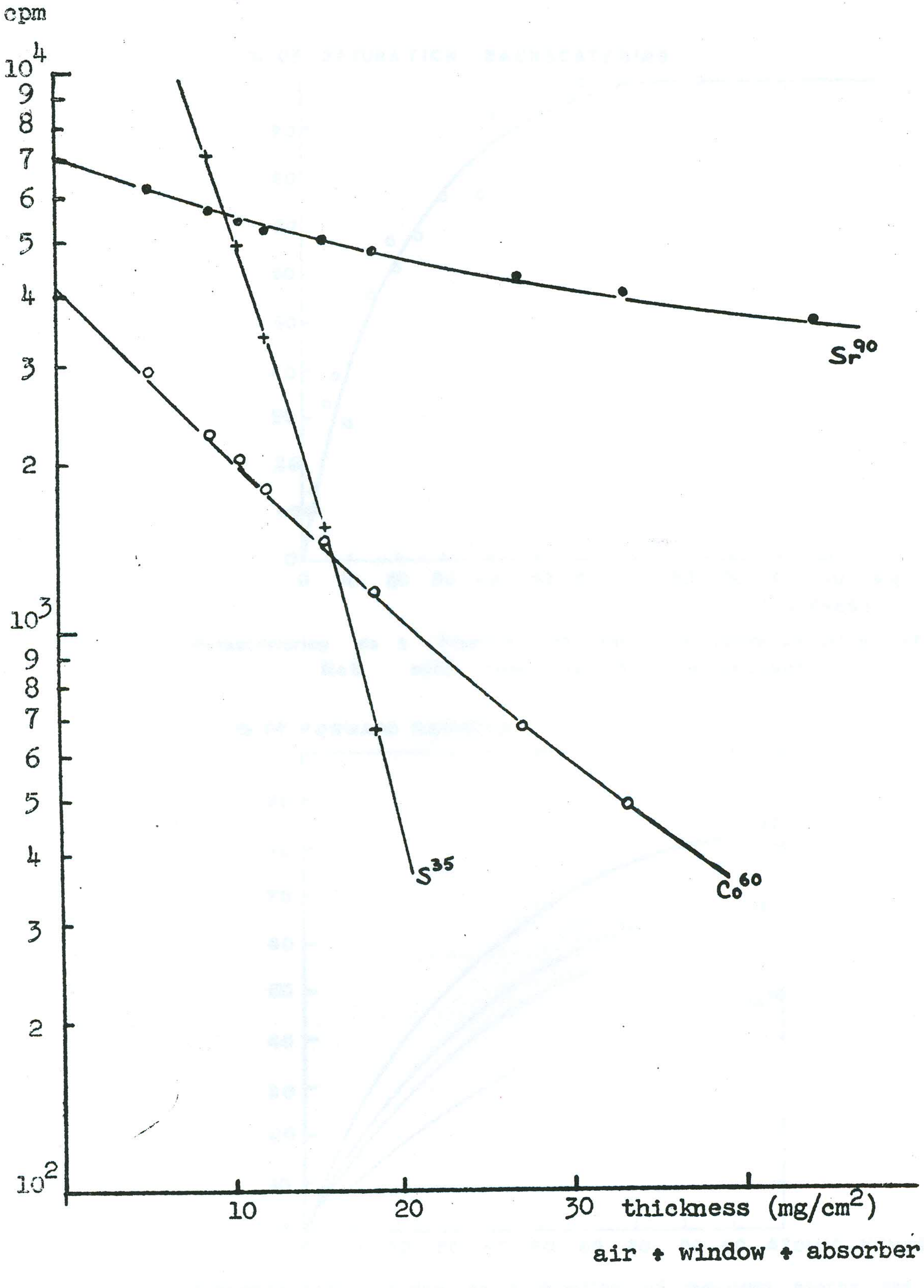
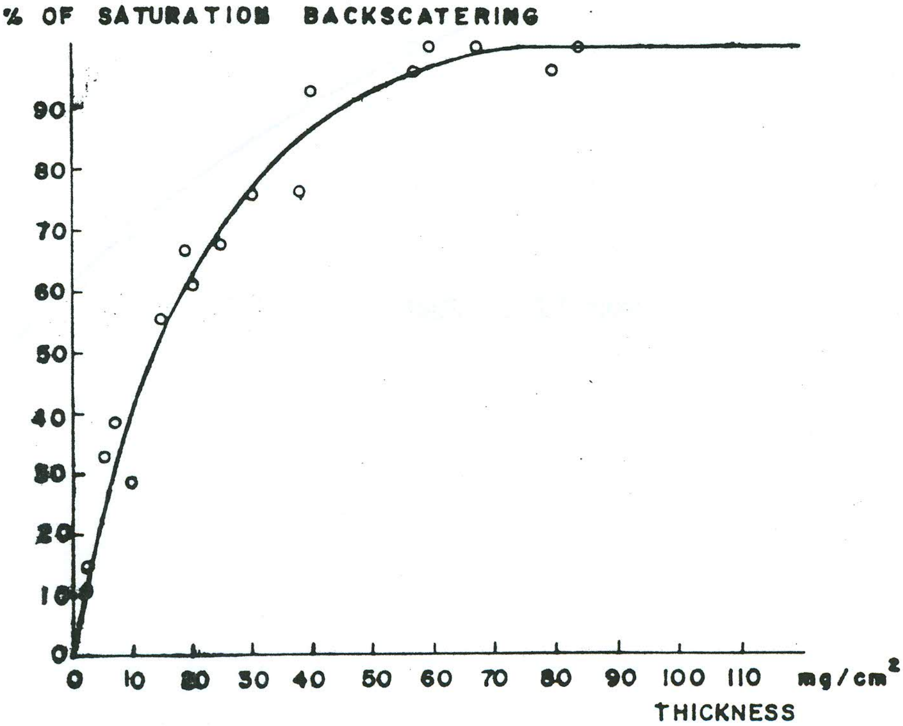
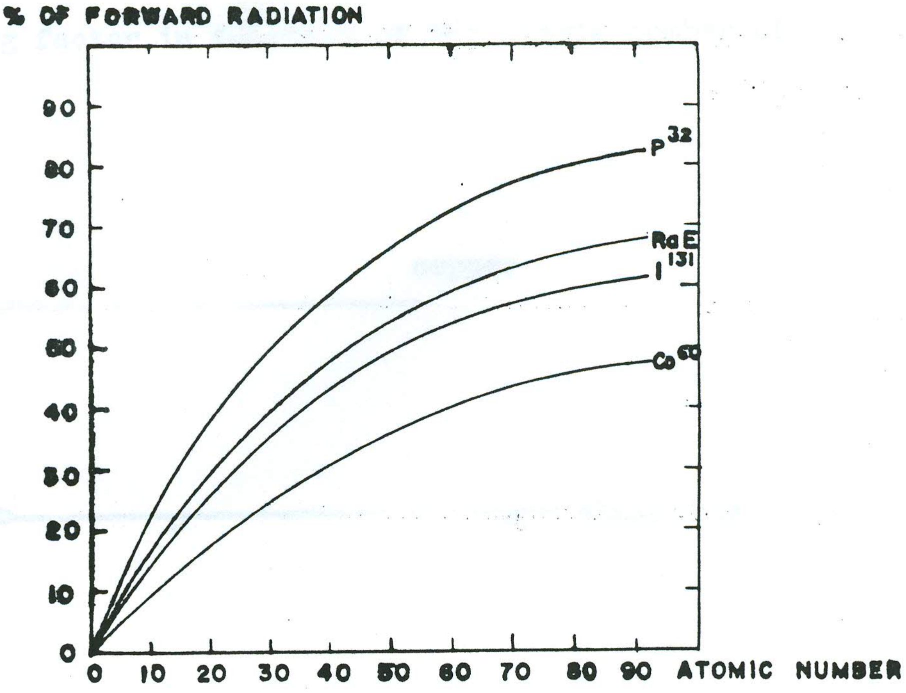


Fig. 7) Absorption curves for the determination of the absorption factor in the window + air

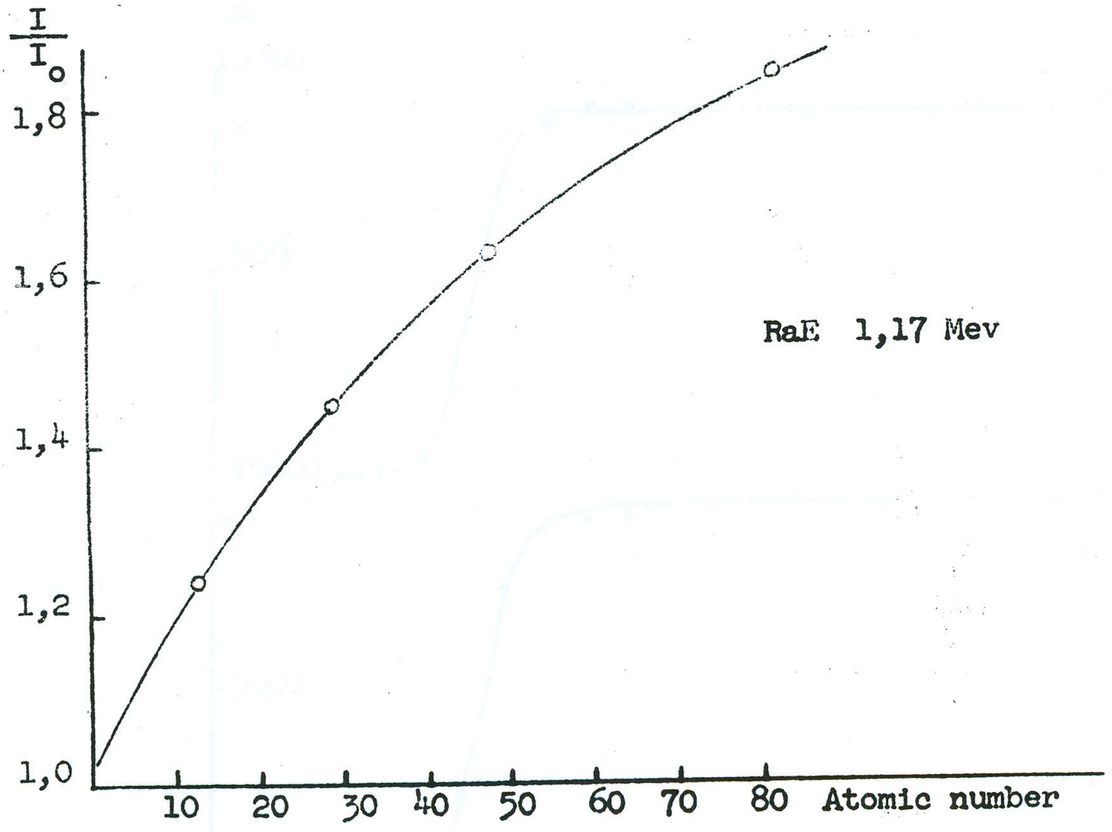


Backscattering as a function of thickness (backscattering of RaE beta particles by polystyrene)

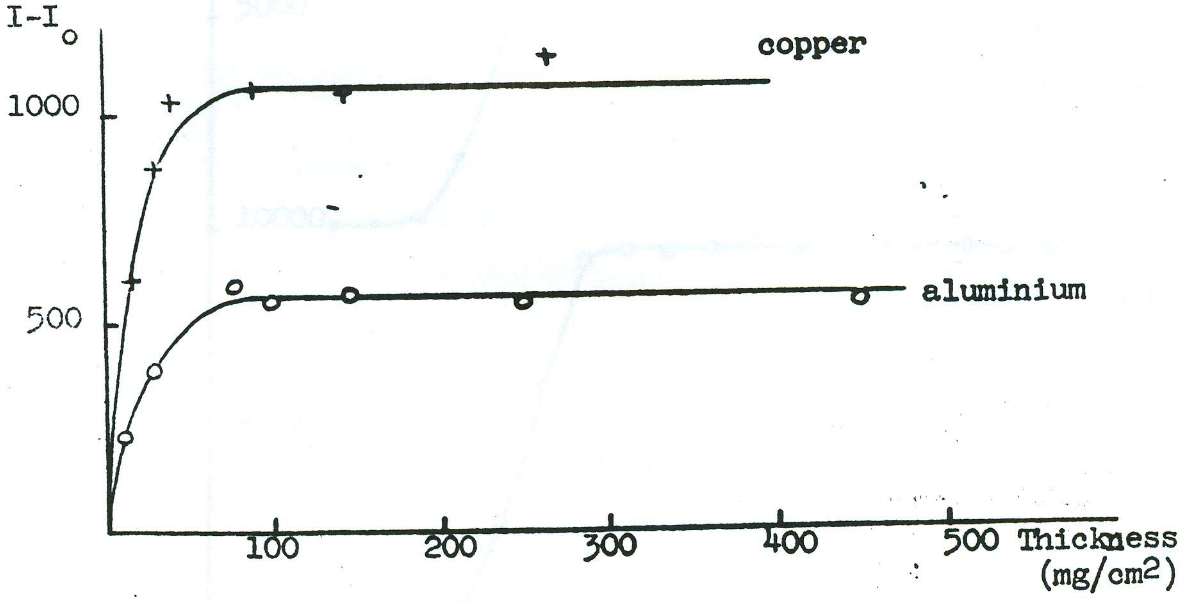


Saturation backscattering as a function of maximum energy and of atomic number of backscattering material

Fig. 8a) Backscattering of beta particles (after L.R. Zumwalt)



Backscattering factor in fonction of the atomic number of backscatterer



Saturation curves of the backscattering radiation

Fig. 8b) Backscattering of beta particles (After A. Cervellini)

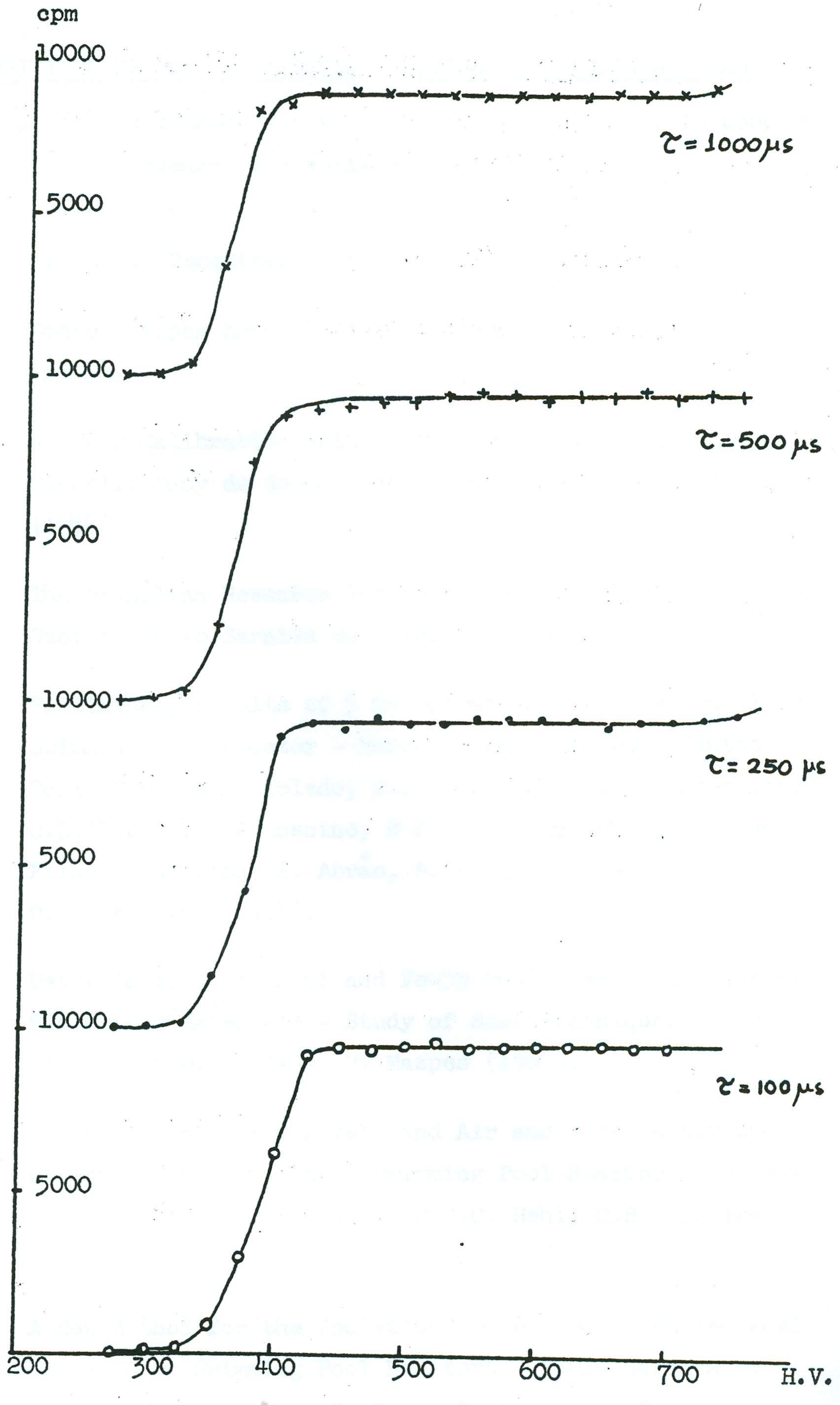


Fig. 9) Plateaux for the different pre-stablished dead times

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List of papers published by the D.F.N. (including paper in cooperation with other research divisions of the I.E.A.)

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nº 5. A Power Calibration Method Using the Xenon Poisoning - Marcello Damy de Souza Sanots, Paulo Saraiva de Toledo (1958).

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- Utilisation d'un Eclair Lumineux pour la Stabilisation d'un Spectrometre de Scintillation - Manoel Américo Nogueira de Abreu (1966)