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AUSTRALIAN ATOMIC ENERGY COMMISSION
RESEARCH ESTABLISHMENT
LUCAS HEIGHTS

STANDARDISATION OF RADIOISOTOPES AT LUCAS HEIGHTS BY $4\pi\beta$ COUNTING AND $4\pi\beta$-γ COINCIDENCE COUNTING

by

D.F. URQUHART

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ABSTRACT

The principles of $4\pi\beta$ and $4\pi\beta$-γ coincidence absolute counting methods are discussed, with particular reference to the corrections required. The practical aspects of source preparation, instrumentation, instrumental adjustments, and counting procedure are described in some detail. Examples are given of the standardisation of several commonly used isotopes and the results obtained are compared with standards obtained from overseas laboratories. The examples include Tl-204 which was standardised in this laboratory while participating in an international intercomparison of this isotope.

ERRATA - AAEC/TM 239

Please make the following amendments to AAEC/TM 239, a report issued by the A.A.E.C. Research Establishment, Lucas Heights, Sutherland, N.S.W. Australia.

Page 13 Line 21 should read:

Let σ_β = fractional standard deviation in the net beta count, due to background only.

The last equation should read:

$$\sigma_\beta = \frac{t_s}{N_\beta} \left[\left\{ \frac{1}{t_s} \left(\frac{B_\beta . t_s}{t_b} \right)^{\frac{1}{2}} \right\}^2 + \left\{ \frac{1}{t_b} (B_\beta)^{\frac{1}{2}} \right\}^2 \right]^{\frac{1}{2}}$$

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The first line should read:

$$= \frac{t_s}{N_\beta} \left(\frac{B_\beta}{t_b t_s} + \frac{B_\beta}{t_b^2} \right)^{\frac{1}{2}}$$

The second line should read:

$$= \frac{\left[B_\beta \; r \; (r + 1) \right]^{\frac{1}{2}}}{N_\beta}$$

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1. INTRODUCTION

The first radioisotope standard was set up in Paris in 1911. It consisted of a sealed glass tube containing 21.99 mg of pure anhydrous radium chloride and was accepted as the international primary standard. This and similar radium standards, established later, served until the advent of controlled nuclear fission when the large scale production of artificial radionuclides created the need for other standards.

In the early radium standards radioactivity was measured in terms of curies, the curie being defined as the quantity of radon in equilibrium with 1 g of radium. This quantity of radon was thought to emit alpha particles at the rate of 3.7×10^{10} per second. Later measurements have shown this to be $3.608 \pm 0.028 \times 10^{10}$. In order to make this unit applicable to all radioactive materials, the Sixth International Congress of Radiology in London in 1950 redefined the curie as the quantity of any radioactive material in which the number of disintegrations is 3.700×10^{10} per second.

At a conference at Harwell in 1951, the U.S.A., Canada, and the U.K. agreed to exchange, for intercomparison purposes, samples of nine isotopes: C-14, Na-24, P-32, Co-60, Br-82, Sr-90/Y-90, I-131, Tl-204, and Au-198. Other countries have since joined in this scheme and the responsibility for the establishment of national standards in each country has generally been vested in the existing standards authority, such as the National Bureau of Standards in the U.S.A., and the National Physical Laboratory in the U.K. The Australian National Standards Laboratory has not entered this field.

Most artificially produced isotopes are short lived and are generally difficult to produce in an isotopically pure state. This precludes the setting up of weighed quantities as was adopted for the radium standards. It is therefore necessary to determine the absolute activity of "working standards" in curies by direct counting of the disintegration rate.

The Australian Atomic Energy Commission has set up its own radioisotope standards laboratory responsible for the accuracy of radioisotope standards used at the Research Establishment. The main requirements for such standards are:

(a) Certification on request of the activity, in curies, of radioisotopes distributed by the Isotope Production Group.

(b) Absolute determination of neutron fluxes by measurement of the activity induced in target elements.

(c) Calibration of monitors and sub-standard measuring instruments.

For the most part an accuracy of ± 5 per cent. is adequate and ± 2 per cent. is occasionally necessary.

Following discussions by a committee of section heads in 1959 and a visit to several overseas standardising laboratories by Dr. J.K. Parry in 1960, it was decided to form a standards group which was to begin by setting up and testing a $4\pi\beta$ proportional counter and a $4\pi\beta$-γ coincidence counter. The work was begun by the counter development group in the General Physics Section in January, 1961 and was handed over to a newly formed standards group in 1962.

This paper describes the two methods, the equipment now in use at Lucas Heights and the results obtained from absolute measurement on several commonly used isotopes.

2. THE $4\pi\beta$ PROPORTIONAL COUNTER

For absolute beta counting it is essential to know accurately the overall detection efficiency. This is most readily achieved by making the detection efficiency 100 per cent. A gas flow counter is suitable since the intrinsic efficiency is 100 per cent. for particle energies down to about 200 eV, and 4π geometry can be easily obtained. High geometrical efficiency can also be achieved using liquid or plastic scintillators but ionizing particles of energy less than about 1 keV are not detectable with presently available photomultiplier tubes.

A gas flow counter operating in the proportional region is the most suitable counter for this application since the long dead time of Geiger counters places an undesirable limit on the maximum counting rate.

The 4π proportional counter has proved to be satisfactory for standardisation of beta sources of end point energy greater than 1 MeV. An absolute accuracy of ± 1 per cent. or better can be obtained. For lower energies the source self-absorption correction becomes uncertain and may introduce errors of up to 10 per cent. for isotopes such as S-35 and Ni-63.

2.1 Principle of Operation

The operation of proportional counters has been described by many authors, for example Sharpe (1955) and Rossi and Staub (1949). Four aspects relate particularly to absolute counting.

Firstly, since the energy distribution of a beta source extends down to zero energy a fundamental limitation to the intrinsic counter efficiency is imposed by the average energy W required to produce one ion pair. For argon, W is about 30 eV so there is a high probability that a β particle with energy less than this will not be detected. Assuming a Poisson distribution for the number of ion pairs produced, one can calculate the probability that at least one ion pair will be formed. Pate and Yaffe (1955b) have shown that the probability for one ion pair is 99.9 per cent. for a β energy of about five times W. For most isotopes, virtually all of the β energy distribution will lie above this level and the intrinsic counter efficiency will be very nearly 100 per cent provided the gas gain is sufficient to detect one ion pair. Pate and Yaffe show for example that the intrinsic efficiency possible, is greater than 99.9 per cent. for Ni-63 (end point energy 67 keV).

Secondly, short clipping times are used in the amplifier following the proportional counter; consequently the output pulse is due almost entirely to the collection of electrons. The collection time is less than 1 μsec, compared with about 100 μsec for the heavy positive ions. If a beta particle enters the gas before the positive ion cloud from the preceding particle has drifted away from the anode, the output pulse may be reduced in amplitude, owing to the reduction of the field strength in the neighbourhood of the anode by the space charge effect of the positive ion cloud. This effect places a limitation on the maximum source strength which can be used in the counter.

A third aspect of particular importance in this application, is the occurrence of "after pulses". Radiation from the molecules of the counter gas excited by an ionizing event may result in one or more minor output pulses in addition to the main pulse. Engfer and Buttlar (1962) have observed the time distribution of "after pulses" in argon-methane gas. They report a distribution with a maximum at about 200 μsec after the main pulse and extending to about 3 msec. Since a dead time of less than 20 μsec is generally required for standard counting, the passage of a single beta through the counter gas could result in more than one count.

The fourth aspect is the time delays introduced by the detector. Although the electric field strength in the region close to the small diameter wire anode (where all the gas multiplication occurs) is high, the field is much lower over the greater part of the volume of the counter. An appreciable time is therefore required for the negative ions, produced at a distance from the anode, to drift to the region where gas multiplication occurs. This results in a variable delay between the instant the beta is emitted from the source and the appearance of the pulse at the anode. These delays (up to 1 μsec) are important when the proportional counter is used for beta-gamma coincidence counting.

2.2 Description of 4πβ Counter in Use

The counter in use is an A.E.R.E. type 1364A (Figure 1). It is a cylindrical counter with loop anodes

(0.6 cm radius loops of 0.001 inch dia. molybdenum wire). The sample drawer is effective in restricting entry of air to the chamber when changing sources and the chamber can therefore be flushed quickly after introduction of a source.

The source is deposited on gold coated VYNS foils as shown in Figure 1. The preparation of foils and the deposition of the sources is described in Section 6.

Three counting gases have been used successfully; type A, 90 per cent. argon + 10 per cent. methane, Type B, 90 per cent. argon + 10 per cent. propane and Type C, "pure" propane. All give similar plateaux, 200 to 300 volts long with slopes of less than 0.5 per cent. per 100 volts, for Co-60 betas. Operating voltages of 1,800 to 2,000 volts are required for types A and B and 4,000 to 4,500 volts for type C.

Type A is expensive and has to be imported. Specified mixtures of argon and propane can be obtained locally as well as a special high grade propane guaranteed to contain 85 to 95 per cent. propane and a negligible proportion of air.

Type A produces faster rising output pulses than the other two types and is preferred when the proportional counter is used in a β-γ coincidence arrangement, because shorter coincidence resolving times may be used.

Figure 2a shows examples of plateau curves for these gases.

Differential and pulse height distributions for argon-methane gas are shown in Figure 2b. It can be seen that the gas gain at the voltage used (1920 volts) is sufficient to produce output pulses above the amplifier noise level for beta energies down to about 50 electron volts. The occurrence of "after pulses", however, sets the useful lower limit of detection at 200 to 300 electron volts. The beta energy scale in Figure 2b was determined by using the 5 keV X-ray from Cr-51. as a reference line.

2.3 The Amplifier

For a beta source, the proportional counter output covers a wide spectrum of pulse heights. An amplifier gain of up to 93 db may be required to count the minimum output pulses and at this gain those maximum energy betas which lose most of their energy in the counter gas may produce output pulses which overload the amplifier by as much as 50 times. An amplifier with good overload characteristics is therefore necessary to prevent loss of counts at high count rates.

The main factors which contribute to long overload recovery in conventional pulse amplifiers are:

(a) Shift in d.c. levels in the amplifier due to pile-up of large pulses at high counting rates.

(b) Interstage coupling capacitors charge quickly through low resistance grid circuits while grid current is being drawn but discharge slowly through the high grid leak resistors.

In an amplifier designed by Fairstein (1956), (a) is minimised by the following means: the amplifier contains two differentiating time constants producing bi-polar output pulses. This symmetrical pulse results in a zero d.c. component.

Problem (b) is overcome as follows:

(i) The input of each feed-back group in the amplifier is a cathode-coupled pair with the operating point so chosen that the maximum possible driving pulse cannot cause grid current to flow.

(ii) The grid leak resistances are made small compared with the preceding plate loads. By this means the charging and discharging time constants for coupling networks can be made nearly equal. The use of bi-polar output pulses introduces a difficulty in the design of the output stages where large plate current swings are required. A special output stage employing a "White" cathode follower overcomes this difficulty.

The amplifier used is a Type NE.5202 made by Nuclear Enterprises (G.B.) Ltd. and is based on a design by Fairstein. Its performance is generally satisfactory but a positive overshoot occurring on overload pulses is troublesome for low level input pulses. The overshoot has a very long "tail" and effectively extends the overload recovery time to about 40 μsec. This can be overcome by using a long dead time (for example 50 μsec), but for low energy particles an amplifier with improved performance is desirable for fast counting applications.

The main amplifier is preceded by a cathode follower preamplifier which is mounted directly on the back of the lead castle, with short leads to the counter to minimise stray capacity.

The Nuclear Enterprises pre-amplifier was found to contribute noise pulses when connected to the proportional counter. This was due to stray leakage currents at the input caused by the 2 kV polarising voltage on the counter. This was overcome by replacing the input coaxial socket and the input condenser with better quality high voltage components.

3. THE $4\pi\beta$-γ COINCIDENCE COUNTER

The $4\pi\beta$-γ coincidence counter is the most successful absolute counter for those isotopes in which beta decay is accompanied by gamma emission. This counting method may be regarded as an extension of the $4\pi\beta$ method by which a subsidiary measurement is made to determine the loss of efficiency due to source self absorption of low energy betas. The counter may also be regarded as a particular form of the beta-gamma coincidence counter. In its usual form two low efficiency detectors are used and the simplicity of the basic principle is largely offset by the difficulty of accurately evaluating the many corrections required in practice. Campion (1959) has shown that they become small as the efficiency of one of the detectors approaches 100 per cent. He considers all the corrections likely to be required and shows the significant reduction in the magnitude of these corrections which occurs when a high efficiency detector such as a $4\pi\beta$ proportional counter is used for the beta detector.

3.1 The Principle of the $4\pi\beta$-γ Coincidence Method

If a beta and gamma emitting source disintegrating at N disintegrations per sec. is observed simultaneously by a beta detector (efficiency E_β) and a gamma detector (efficiency E_γ), the count rates observed from the two detectors (N_β and N_γ) are related to the disintegration rate by the following equations:

$$N_\beta = N \, . \, E_\beta \qquad \ldots\ldots \; (1)$$

$$N_\gamma = N \, . \, E_\gamma \, . \qquad \ldots\ldots \; (2)$$

If we also observe the rate at which betas and gammas due to the same disintegration are counted by both detectors, we find that the coincidence rate N_c is related to the disintegration rate by the equation:

$$N_c = N \, . \, E_\beta \, . \, E_\gamma \qquad \ldots\ldots \; (3)$$

Solving these equations for N gives:

$$N = \frac{N_\beta \, . \, N_\gamma}{N_c} \, . \qquad \ldots\ldots \; (4)$$

The source disintegration rate is thus obtained from a simple ratio of directly observed counting rates, independent of the efficiency of either detector.

Alternatively, these equations may be used to find the efficiency of either detector; for example, from Equations 1 and 4,

$$E_\beta = \frac{N_c}{N_\gamma} \, . \qquad \ldots\ldots \; (5)$$

The application of the β-γ coincidence technique using low efficiency detectors has been described by Dunworth (1940), Barnothy and Farro (1951), Siegbahn (1955) and Bay (1956) and has been used at Lucas Heights for the absolute measurement of foils used as neutron flux monitors in HIFAR (Nicholson and Quealy 1961).

3.2 Corrections

Campion (1959) has examined the corrections which must be made in the practical application of Equation 4 and has derived general expressions for them. Many amount to several per cent when both detectors have a low efficiency and evaluation is difficult without making many tedious and often uncertain subsidiary measurements. He shows that the corrections generally amount to less than 0.1 per cent. when a $4\pi\beta$ proportional counter with an efficiency approaching 100 per cent. is used. His observations and conclusions are summarised below.

3.2 1 Correction for finite source size

Equation 4 applies only to a point source. For a finite source the efficiency for beta and gamma detection may not be the same for radiations originating from different parts of the source. Putman (1953) has shown that

$$N = \frac{N_\beta \cdot N_\gamma}{N_c}\left[\frac{\overline{E_\beta \cdot E_\gamma}}{\overline{E_\beta} \cdot \overline{E_\gamma}}\right] , \qquad \ldots . (6)$$

where $\overline{E_\beta \cdot E_\gamma}$ is the mean of the product of the β and γ detector efficiencies integrated over the extension of the source. If either detector is equally sensitive to all parts of the source, the term in the square bracket reduces to unity. In general, source activity will not be uniformly distributed. For example, evaporated sources tend to form clumps concentrated around the perimeter of the source.

Campion makes an order of magnitude estimate of this correction for a $4\pi\beta$-γ coincidence counter. Taking an extreme case in which the beta efficiency over the extent of the source varies from 100 to 85 per cent. owing to variations in self absorption he shows that the correction amounts to less than 0.01 per cent.

3.2.2 Effect of instrumental drift

The coincidence equation(4)is insensitive to changes in the sensitivity of either or both detectors, since a change in either or both terms of the numerator $(N_\beta . N_\gamma)$ will be accompanied by a corresponding change in the denominator N_c. The stability of proportional flow counters is exceptionally high and it can be shown that as long as one detector is stable, large fluctuations in the sensitivity of the other detector can be tolerated without the need for any correction to the equation.

3.2.3 Corrections for β-γ angular correlation

Small corrections for β-γ angular correlation may be necessary when low efficiency detectors are used. However, when a 4π proportional counter is used for the β-detector, this correction becomes zero or negligible.

(a) If $E_\beta = 1.0$ there is clearly no correction necessary.

(b) If E_β is less than 1.0, the loss in efficiency, being due only to self-absorption, is not likely to be more than 15 per cent. The angular correlation becomes less at low β energies and it is only the low energy betas which are affected by self-absorption. In general, β-γ angular correlation can be disregarded for this type of counter.

3.2.4 Correction for internal conversion

Consider a simple decay scheme with 100 per cent. β emission and a coincident gamma ray. If the gamma ray is internally converted for a fraction F_c of the total number of disintegrations, then:

$$N_\beta = N\left[E_\beta + F_c \cdot E_e\,(1-E_\beta)\right]$$

$$N_\gamma = N \cdot E_\gamma \cdot F_\gamma$$

$$N_c = N \cdot E_\beta . E_\gamma . F_\gamma \quad .$$

From these equations:

$$\frac{N_\beta . N_\gamma}{N_c} = N\left[1+\frac{F_c . E_e(1-E_\beta)}{E_\beta}\right] = N\left[1+\frac{\alpha(1-E_\beta)E_e}{E_\beta(1+\alpha)}\right] \quad \ldots (7)$$

where:

E_e = efficiency of beta detector for the conversion electrons.

F_γ = fraction of unconverted gammas, and

$\alpha = \frac{F_c}{F_\gamma}$ = conversion coefficient.

For a low efficiency β-detector, this correction may become quite large and is subject to uncertainty in the value of E_c and α. For $E_\beta = 1$, however, the correction factor becomes unity.

When some loss of β efficiency occurs due to self absorption, the term $1-E_\beta$ is usually less than 0.15 and the correction is therefore small. The correction is evaluated for some particular cases in Section 7.2.

3.2.5 Υ-sensitivity of the β detector and Υ-Υ coincidences

The β detector may not have zero efficiency for Υ-rays. In this case Υ-rays may contribute to the "β count" and Υ-Υ coincidences may occur due to Compton scattering or to the presence of a second Υ-ray in the spectrum.

Campion shows that:

$$\frac{N_\beta . N_\gamma}{N_c} \approx N \left[1 + \frac{(1-E_\beta)}{E_\beta} \left\{ (E_\beta)_\gamma - \frac{E_c}{E_\gamma} \right\} \right] \qquad \ldots (8)$$

where

$(E_\beta)_\gamma$ = Sensitivity of the beta detector for Υ rays.

E_c = Probability that a coincidence will be detected when the β particle is undetected.

When E_β is small this correction can become large. When $E_\beta = 1$, no correction is required. When a 4π proportional counter is used for the β detector, E_β is seldom less than 0.85 and E_α is small. It is found in practice that this correction is usually less than 0.1 per cent. It is evaluated for a particular case in Section 7.2.

3.2.6 Bremsstrahlung correction

It is generally easy to prevent betas from reaching the Υ detector. However, bremsstrahlung photons may contribute to the Υ count. It is shown by Campion that:

$$\frac{N_\beta \cdot N_\gamma}{N_c} = N \left[1 - \frac{(1-E_\gamma)(1-E_\beta)N_{\gamma\beta}}{E_\gamma E_\beta N} \right] \qquad \ldots (9)$$

where $N_{\gamma\beta}$ is the bremsstrahlung counting rate in the gamma channel. When $E_\beta = 1$ the correction becomes unity. The ratio $\frac{N_{\gamma\beta}}{N}$ can be found experimentally using pure beta emitters. However, this quantity is only significant for high energy betas in which case $E_\beta \simeq 1.0$ for a 4π proportional counter.

3.2.7 Corrections for complex decay schemes

If a nuclide decays by two β branches (A and B), it can be shown that:

$$\frac{N_\beta . N_\gamma}{N_c} = N \left[1 - \frac{ab(E_{\beta a}-E_{\beta b})(E_{\gamma a}-E_{\gamma b})}{aE_{\beta a}\ E_{\gamma a}+bE_{\beta b}\ E_{\gamma b}} \right] \qquad \ldots (10)$$

where

a = fractional intensity of branch A,

b = fractional intensity of branch B,

$E_{\beta a}$ = beta efficiency for branch A,

$E_{\beta b}$ = beta efficiency for branch B,

$E_{\gamma a}$ = gamma efficiency for branch A,

$E_{\gamma b}$ = gamma efficiency for branch B.

The correction becomes unity if $E_{\beta a} = E_{\beta b}$, or if $E_{\gamma a} = E_{\gamma b}$. If any of the γ rays are internally converted the correction is zero only when the two β efficiencies are equal. For multiple branching the correction is more difficult to evaluate. The use of the $4\pi\beta$ counter therefore provides an advantage over low efficiency detectors since when $E_\beta = 1.0$ for all branches, the correction becomes 1.0 in all cases. When E_β is less than 1, the correction should be evaluated for each particular detector system and each decay scheme, but provided E_β is greater than 0.85 for every branch, the correction is less than 0.1 per cent. even for complex decay schemes such as iodine-131. (see Section 7.2).

3.2.8 Correction for accidental coincidences

Hayward (1961) showed that the use of one high efficiency detector complicates the correction for accidental coincidences. When both detectors have low efficiency the true coincidence rate can be measured directly by subtracting the accidental rate (as measured by the use of two unrelated sources) from the observed total coincidence rate. However, when the efficiency of one detector approaches 100 per cent. an appreciable fraction of the chance coincidences will be masked by true coincidences. In this case it is necessary to derive the correction in the following way (Campion 1959).

Let N_c^{1} = total coincidence rate, that is, "true" + "accidental" rate (less background),

N_c = true coincidence rate,

N_β^{1} = observed β count rate including background,

N_γ^{1} = observed γ count rate including background,

T_R = coincidence resolving time.

Those counts in the β channel for which no true coincidence pulse is recorded in the γ channel are $(N_\beta^{1}-N_c)$ and the counts in the γ channel for which no true coincidence pulse is recorded in the β channel are $(N_\gamma^{1}-N_c)$.

Therefore the accidental coincidence rate:

$$N_c^{1}-N_c = T_R N_\gamma^{1}(N_\beta^{1}-N_c) + T_R N_\beta^{1}(N_\gamma^{1}-N_c) \ ,$$

and

$$N_c = \frac{N_c^{1}-2T_R N_\beta^{1} N_\gamma^{1}}{1-T_R(N_\beta^{1} + N_\gamma^{1})} \ . \qquad \ldots (11)$$

In practice, for values of E_β greater than 0.85 and low values of E_γ (for the counter to be described here E_γ is approximately equal to 0.02), the correction for accidental coincidences (for T_R 2 μsec) is generally less than 0.5 per cent, and can be evaluated to an accuracy of about ± 10 per cent.

3.2.9 Correction for dead time and background

The use of a high efficiency detector also complicates the correction for dead time (Hayward 1961). If two low efficiency detectors are used, the dead time losses can be considered as part of the overall efficiency and no correction is required to the basic coincidence equation. However a correction must be made if one of the detectors has a high efficiency. Campion has shown that for equal dead times T in the β and γ channels:

$$\frac{N_\beta N_\gamma}{N_c} = N \left[1 - NTE_\beta E_\gamma \right] \ , \qquad \ldots (12)$$

provided the source disintegration rate is such that $(N\ T)^2$ is much less than $N\ T$ and that the dead time associated with the coincidence channel is not greater than T.

If E_β is greater than 0.85, E_γ is less than 0.1, and N T is less than 0.05, Equation 12 can be simplified without introducing an error greater than 0.1 per cent. to the more familiar form:

$$N = \frac{N_\beta N_\gamma}{N_c(1-N_\gamma T)} \qquad \ldots (13)$$

If the dead time in the beta channel (T_β) is greater than the gamma dead time (T_γ), this equation becomes:

$$N = \frac{N_\beta N_\gamma}{N_c(1-N_\gamma T_\gamma)} \quad , \qquad \ldots (14)$$

and for T_γ greater than T_β:

$$N = \frac{N_\beta N_\gamma}{N_c(1-N_\gamma T_\beta)} \quad . \qquad \ldots (15)$$

It is useful to note that from Equation 15 it also follows that:

$$E_\beta = \frac{N_c(1-N_\gamma T_\beta)}{N_\gamma(1-N_\beta T_\beta)} \qquad \ldots (16)$$

and

$$E_\gamma = \frac{N_c}{N_\beta} \quad . \qquad \ldots (17)$$

In all the corrections given in the preceding sections, the observed count rates N_β, N_γ and N_c are count rates obtained in excess of the background counts.

3.3 Statistical Accuracy for the Coincidence Equation

The statistical accuracy for N in Equation 4 cannot be obtained directly from the standard deviations of the counting rates in the three channels because they are not independent.

It has been shown by Campion and Taylor (1961) that the fractional standard deviation for N is given by:

$$\sigma = \left[\frac{1}{N_c}\left(2E_\beta E_\gamma - E_\beta - E_\gamma + 1\right)\right]^{\frac{1}{2}} , \qquad \ldots (18)$$

where N_c = coincidence count accumulated in time t, and E_β and E_γ = overall beta and gamma efficiencies.

It follows from Equation 18 that for two low efficiency detectors, σ approaches the value $N_c^{-\frac{1}{2}}$, that is, the statistical accuracy is determined entirely by the accumulated coincidence count. When E_γ is small the coincident count rate is also small, so long counting times are required for good statistics. On the other hand, if E_β approaches the value 1.0, σ approaches $N_\beta^{-\frac{1}{2}}$, that is, the accuracy is determined entirely by the accumulated β count, and good statistical accuracy can be obtained with shorter counting times.

In Equation 18 Campion assumes contribution to the standard deviation from background and chance coincidence to be negligible. In practice this is not usually the case; the gamma background in the coincidence counter to be described here may double the value of the standard deviation for weak sources. A rigorous treatment of the background and chance coincidences leads to a cumbersome expression for σ but a useful approximation can be derived if it is assumed that the backgrounds in the three counting channels and the chance coincidence rate are independent.

Let β = fractional standard deviation in the net beta count, due to background only,

B_β = total background count in time t_b ,

N_β = net source count in time t_s .

Then:

Total count with source in counter = $N_\beta + \dfrac{B_\beta\, t_s}{t_b}$.

Therefore:

count rate with source in counter $= \dfrac{N_\beta}{t_s} + \dfrac{B_\beta}{t_b}$.

Standard deviation for total count rate (due to background only)

$$= \frac{1}{t_s}\left(\frac{B_\beta\, t_s}{t_b}\right)^{\frac{1}{2}} .$$

Standard deviation for background count rate is $\dfrac{(B_\beta)^{\frac{1}{2}}}{t_b}$.

Therefore:

$$\sigma_\beta = \left[\left\{\frac{1}{t_s}\left(\frac{B_\beta\, t_s}{t_b}\right)^{\frac{1}{2}}\right\}^2 + \left\{\frac{1}{t_b}\;(B_\beta)\right\}^2\right]^{\frac{1}{2}}$$

$$= \left(\frac{B_\beta}{t_b \, t_s} + \frac{B_\beta}{t_b^{\,2}} \right)^{\frac{1}{2}}$$

$$= \left[\frac{B_\beta \, r \, (r + 1)}{N_\beta} \right]^{\frac{1}{2}} , \qquad \ldots . \; (19)$$

where $r = \frac{t_s}{t_b}$.

Similarly the fractional standard deviation σ_γ in the net gamma count due to background only is given by:

$$\sigma_\gamma = \frac{\left[B_\gamma \, r \, (r + 1) \right]^{\frac{1}{2}}}{N_\gamma} . \qquad \ldots . \; (20)$$

For the coincidence channel, chance coincidence counts must be considered as well as background. This leads to the following equation for σ_c, the fractional standard deviation in the true coincidence count.

$$\sigma_c = \frac{\left[B_c r \, (r + 1) + 2A \right]^{\frac{1}{2}}}{N_c} , \qquad \ldots . \; (21)$$

where A = total coincidence count accumulated in time t_s.

The total fractional standard deviation in N is then obtained by combining Equations 18 to 21.

$$\sigma = \left[\frac{2E_\beta E_\gamma - E_\beta - E_\gamma + 1}{N_c} + \frac{B_\beta r(r+1)}{N_\beta^{\,2}} + \frac{B_\gamma r(r+1)}{N_\gamma^{\,2}} + \frac{B_c r(r+1) + 2A}{N_c^{\,2}} \right]^{\frac{1}{2}} . \qquad \ldots . \; (22)$$

When $E_\beta = 1$, the first term in Equation 22 reduces to N_β^{-1}. As the beta and coincidence backgrounds and the chance coincidence rates are small in practice, the 2nd and 4th terms may generally be neglected. This leads to the approximation:

$$\sigma = \left(\frac{1}{N_\beta} + \frac{B_\gamma r(r+1)}{N_\gamma^{\,2}} \right)^{\frac{1}{2}} . \qquad \ldots . \; (23)$$

3.4 Description of $4\pi\beta$-γ Coincidence Counter

The coincidence counter head is shown in Figure 1. The proportional beta counter is as described in Section 2.2.

The gamma detector consists of two scintillation heads, fitted with 1½ inch x 1 inch NaI(Tl) crystals, placed one on each side of the proportional counter. Lead shielding is used to reduce the gamma background.

Figure 3 is a block diagram of the high voltage supplies, amplifiers and coincidence circuit used. The outputs from the two scintillation heads are mixed in the pre-amplifier. The outputs from the beta and gamma amplifiers are applied to the first and second inputs of the coincidence unit and after passing through separate discriminator and dead time circuits are fed to separate inputs of an A.A.E.C. Type 2 Scaler operating as a twin scaler. The coincident output pulses are applied to the counting channel of a second Type 2 Scaler, whose timing channel controls all three counting channels.

If it is necessary to select a particular photo-peak from the scintillation head output a single channel analyser is connected to a parallel output from the gamma amplifier. The analyser output is then connected to the third input on the coincidence unit and the coincidence scaler is connected to the triple coincidence output. This coincidence arrangement enables a long resolving time to be used for triple coincidences, to allow for delays in the analyser, without increasing the chance coincidence count.

4. ADJUSTMENT OF THE $4\pi\beta$ COUNTER

The following conditions must be satisfied to obtain acceptable performance from the proportional counter.

(a) The gas multiplication and amplifier gain must be sufficient to ensure that virtually all betas entering the counting gas volume result in pulses at the amplifier output of greater amplitude than the discriminator bias voltage.

(b) The voltage applied to the counter must not exceed the point which marks the onset of multiple discharging produced by excitation of the gas molecules.

(c) The noise level at the amplifier output must not exceed the discriminator bias level.

(d) A stable operating condition is required so that the count rate is insensitive to small changes in counter voltage, amplifier gain, or discriminator bias level.

The following procedure has been found useful for quickly finding the best operating point:

(i) set the discriminator bias to about 5 volts.

(ii) with no voltage on the counter increase the gain until amplifier noise pulses just begin to trigger the scaler. Then reduce the amplifier gain by a factor of about 2.

(iii) using a thin, small area ($\not<$ 1 cm dia.) source of the isotope to be standardised, centrally placed in the counter, plot the E.H.T. versus count rate curve and then set the E.H.T. to the centre of the plateau. A plateau with a slope of better than 0.5 per cent. per 100 volts and a length of 200 to 350 volts should be obtained.

(iv) the background count rate should be less than 2 counts per sec.

When a counter fitted with loop anodes is used for the first time or after replacing a loop, it is desirable to check that the loops are reasonably well matched by plotting a plateau curve for each half separately.

Some typical plateau curves for several counting gases and beta energies are shown in Figure 2.

5. ADJUSTMENT OF THE $4\pi\beta$-γ COINCIDENCE COUNTER

For satisfactory operation, the following conditions must be satisfied:

(a) The beta efficiency need not be known but should be as close as possible to 100 per cent., certainly greater than 85 per cent.

(b) Every coincidence between recorded beta and gamma events, must be counted.

(c) The average delay time in the beta and gamma channels should be equal, so that a minimum coincidence resolving time can be used.

5.1 The Beta Channel

Adjusted as described in Section 4.

5.2 The Gamma Channel

The following procedure has been found useful for setting up and matching the two scintillation heads.

First, a rough adjustment of gain and E.H.T. is made so that the maximum energy gammas (from the source to be standardised) produce pulses which just overload the amplifier. This can best be found by connecting a C.R.O. to the amplifier output.

Secondly, a single channel analyser and a single gamma source such as Cs-137 are used to make fine adjustments of the E.H.T. to one head so that the same photo-peak from each head is centred on one analyser channel.

5.3 The Coincidence Channel

Correct adjustment of the coincidence unit is of great importance as the principle of the coincidence method assumes 100 per cent. coincidence efficiency.

The coincidence resolving time is first set to about 0.3 μsec and a curve showing coincidence count rate versus delay times is plotted as described in the operating manual for the coincidence unit (A.E.R.E. Type 1036C). The delays are set to the peak of this curve.

Then a curve is plotted showing coincidence rate (corrected for accidental coincidences) versus resolving time. The resolving time is set at a point on the flat plateau region of this curve just above the knee. The accidental coincidence correction is obtained from the equation:

$$N_A = 2N_\beta N_\gamma T_r \quad , \qquad \ldots. (24)$$

where N_A = accidental coincidence rate,

T_r = coincidence resolving time, and

N_β and N_γ are the average count rates in the beta and gamma channels.

The coincidence delay and resolving times may require re-adjustment if any substantial change is made in the E.H.T., gain, or bias level in either the beta or gamma channels.

The coincidence resolving time is used in the calculation of standardisation results and should be measured to an accuracy of ± 10 per cent. or better. This may be done by measuring the chance coincidence rate for independent beta and gamma sources and solving Equation 24 for T_r.

5.4 Paralysis Times

In the calculation of standardisation results, a significant correction is required for dead-time losses. To enable these corrections to be made accurately the following conditions must be satisfied:

(a) The paralysis time setting of the discriminator should be larger than the maximum possible value of any variable dead time occurring in the pulse amplifiers or scalers.

(b) The paralysis time should be as short as possible.

(c) The paralysis time should be non-extending.

(d) The gamma dead time should be longer than the coincidence dead time, which is about 15 μsec.

The paralysis circuits in the A.E R.E. Type 1036C coincidence unit have been found to be satisfactory. The actual dead time should be measured as accurately as possible by the two source method described by Price (1958) and others.

6. SOURCE PREPARATION

The successful standardisation of a radioisotope depends very much on the way in which the source is prepared for counting. The most important requirements are:

(a) The source counted should be an accurately measured (usually by weight) representative sample of the source to be standardised.

(b) The activity of the source counted should be high enough to obtain good counting statistics without introducing large dead time corrections.

(c) The source self-absorption should be minimised.

(d) Source area should be small compared with dimensions of the counter.

(e) Absorption in the source support should be as low as possible. For $4\pi\beta$ counting it is necessary to measure the absorption and correct for this.

(f) Source support should have low surface electrical resistivity.

(g) Good mechanical and chemical stability of the source deposit is necessary.

(h) Care should be taken to ensure freedom from radioactive contamination.

The procedure adopted for source preparation is summarised in Section 6.1 and aspects requiring more detailed description are discussed in Sections 6.2 to 6.4.

6.1 Summary of Procedure Used for Source Preparation

(a) If the source to be standardised is not already in solution it is weighed (to an accuracy of better than 0.1 per cent.) and dissolved completely in a suitable solvent.

(b) An aliquot of the primary solution is accurately diluted in order to obtain a specific activity which will result in a suitable count rate.

(c) The thickness of the source support foil is measured and the foil is pre-treated with a suitable spreading agent. The foil is then placed on the balance and weighed. The required amount of source solution (estimated volumetrically) is then placed on the foil and the time noted. The mass is measured three or four times at intervals of about 15 seconds and if any loss in weight due to evaporation is observed, the initial weight is found by extrapolation back to zero time.

(d) If required, the active material is then precipitated by adding a suitable salt to the drop of solution.

(e) The source is dried slowly under dust free conditions. If any part of the source has been found to have spread more than 1.5 cm from the centre of the foil, the source is rejected.

(f) A second foil is placed over the source. Silver paint is used to achieve good electrical contact between the foil support ring and the conductive surface of the foils.

(g) The source is placed in the counter, the counter thoroughly flushed and an E.H.T. plateau curve plotted. If a good plateau is not obtained the source is rejected.

(h) The source is counted, and the specific activity of the primary solution calculated.

(i) The purity of the source is checked, usually by gamma spectrometry.

6.2 Preparation of Foils

In recent years many source mount materials have been tried. Aluminium foils down to about 200 $\mu g/cm^2$ thickness can be obtained, but are fragile and subject to corrosion from acids in the source solution. The best of the plastic materials is a polyvinyl-chloride acetate copolymer known commercially as VYNS resin. Strong, large area foils can be made with thickness less than 5 $\mu g/cm^2$. This material is resistant to acids, alkalis, and most organic solvents. The films do not readily absorb moisture and do not therefore change weight appreciably when stored for long periods. The use of VYNS for this purpose was first reported by Pate and Yaffe (1955).

VYNS resin has a high surface tension and does not spread readily on water. Two of the methods reported for spreading films have been tried. We were able to prepare very large area foils by the method of Pate and Yaffe but had difficulty in obtaining uniform thickness. A new simple method adopted here is similar to the method described by Green (1961).

A stainless steel sink or similar tank is filled with clean water to within about 1/4 inch of the top. About 1 ml of VYNS solution is then pipetted slowly onto the rounded edge of the sink and allowed to run down the side onto the water. The slow stream of resin solution falling on the water surface produces enough pressure to overcome the surface tension, and the VYNS spreads across the water in a band usually about 3 inches wide. Three or four films can then be picked up directly on the standard support rings used in the counter. The thickness of the foils can be controlled to some extent by adjusting the strength of the VYNS solution.

The foils produced in this way have been uniform. To pick the foils from the water surface the rings are floated on the top of the foil, the film is torn away from around each ring and the rings lifted from the water with a slow rolling motion.

To prepare the VYNS solution a stock solution is first made by dissolving one volume of the resin (supplied in the form of a white powder) in about two volumes of cyclohexanone. This takes about two days at room temperature, but about two hours if the mixture is maintained at about 50-60°C. Small quantities of stock solution are then diluted by further addition of cyclohexanone until the films of the required thickness are obtained. Films of 5 to 10 $\mu g/cm^2$ can be obtained by adding 2 to 3 parts of cyclohexanone to 1 part of the stock solution. Thicker foils are made by laminations of 5 $\mu g/cm^2$ foils. Laminated foils have been found to be stronger than single foils of the same thickness.

Since the foil forms part of the "cathode" of the $4\pi\beta$ counter the foil surface must be made conducting by evaporating gold onto the under side of the foils used to support the source and onto the upper surface of the foils used to cover the source.

It has been found that 15 to 20 μg/cm^2 of gold on each surface is necessary for satisfactory operation of the counter. The relationship between gold thickness and resistance has been measured experimentally and is shown in Figure 4. The resistivity is close to that of bulk gold at about 27 μg/cm^2 and rises steeply as the thickness decreases.

The foils are coated under vacuum of less than 1μ Hg. The gold is evaporated from a tungsten filament. Several foils held on a Perspex frame, equidistant from the filament, are coated at once. For a filament of 22 gauge wire about 1½ inches long, a filament to foil distance of about 6 inches has been found satisfactory.

The amount of gold deposited is controlled by manual adjustment of the filament current, using a simple resistance gauge as an indicator. This gauge gives only a rough indication of gold thickness, a more exact measurement (for foil absorption correction) is made later by means of an optical densitemeter.

6.3 Measurement of Foil Thickness

Accurate measurement of the thickness of VYNS foils is difficult. Pate and Yaffe (1955) reported the use of a beta gauge and a spectrophotometer calibrated gravimetrically. For the sources standardised so far we have found that the thickness can be estimated visually with sufficient accuracy (± 20 per cent.), by the characteristic interference colours as listed in Table 1.

The gold thickness is measured by means of an optical densitometer which was calibrated gravimetrically. The calibration curve showing the relation between optical density and gold thickness (determined by weighing a large number of foils) is shown in Figure 5.

6.4 Source Deposition

Thin, uniform source deposits are required in order to obtain minimum self absorption. The source absorption for a given activity will generally be minimum for the following conditions:

(a) <u>Minimum ratio of inactive to active material in the source.</u> One normally has little control over this factor - high specific activity material is to be preferred.

(b) <u>Source area sufficiently large to allow adequate spacing of particles of source material.</u> The maximum source area is limited by the counter size. The effect on detector efficiency of the displacement of a point source away from the centre of the supporting foil (in the plane of the foil) was determined experimentally for Co-60 betas. The results (Figure 6) show that sources up to 2 cm in diameter can be used, if located centrally on the foils.

When depositing

When depositing small drops of source solution onto the foils it is necessary to pre-treat the foils with insulin or a similar wetting agent in order to spread the source over a large area, that is, as close as possible to the maximum of 2 cm diameter. The wetting agent is applied by pipetting a small drop of insulin onto the centre of the foil and quickly resucking the solution, leaving a thin film over the central portion of the foil.

(c) Uniform distribution of source material over the total source area. It is difficult to obtain uniform deposits from the evaporation of a drop of solution. After drying, the bulk of the source material is usually found to be concentrated around the perimeter of the original drop with sparse distribution in the centre. Aggregation of source particles also occurs. This is particularly undesirable as self-absorption in individual aggregates may be much greater than would be expected from the average, superficial density of the source - the calculation of self-absorption is then difficult. Some workers have reported more even evaporation from aluminium foils than for plastic film such as VYNS, and for this reason some, for example, Martinssen (1958) tended to use comparatively thick aluminium foils rather than VYNS, in spite of the large foil absorption correction required.

Other methods of source deposition, such as electrospraying (Carswell and Milsted 1957) and vacuum distillation (Pate and Yaffe 1956) give very thin uniform deposits but are not quantitative. Although useful when experimenting with self-absorption problems, they are not useful at present for routine standardisation.

(d) Minimum particle size. Some workers have reported that the addition of traces of colloidal silica to the source drop produces large numbers of nuclei for the initiation of crystallisation and this results in the formation of a large number of particles of small size. Merritt, Taylor, and Campion (1959) report reductions in self-absorption of the order of 50-100 per cent. for many isotopes treated with colloidal silica.

For some isotopes it is possible to precipitate the active material, after placing the drop on the foil, by the addition of small amounts of a suitable salt. This also tends to produce small particles and more uniform distribution.

Precipitation of the active material is particularly important for volatile elements such as iodine and bromine; some of the active material may be lost during evaporation of the solvent, and it is therefore necessary to precipitate the active material as soon as the drop is placed on the foil.

6.5 Optimum Activity of Source Deposits

The amount of material to be deposited is adjusted to the highest count rate that can be measured accurately in the beta channel. For the apparatus used here this is about 0.1 μcuries or 3,700 counts/sec. However, if for some reason higher counting rates are necessary, no serious errors are introduced if sources up to 0.2 μcuries are used. The minimum useful source activity depends on the γ decay scheme of the particular isotope, the efficiency of the γ channel and the γ background count rate.

To obtain the desired count rates, it is frequently necessary to make a quantitative dilution of the original solution. This again is best done by weighing. It is important when making such dilutions to stabilise the solution by maintaining the same pH value as in the original. Further details of optimum source activity for particular isotopes are given in Section 7.

6.6 Calculation of Results

Calculation of the results for $4\pi\beta$-γ coincidence counting is done in the following order:

(a) All total counts are converted to "counts/sec" and averaged.

(b) Background count rates in the three channels are subtracted.

(c) Total coincidence rate is corrected for chance coincidences, using Equation 8.

(d) The coincidence equation, including dead time correction (Equation 12 for this counter) is used to evaluate "N".

(e) Any special decay scheme corrections are applied.

(f) The specific activity S_o of the original solution is calculated for some reference time t_o.

$$S_o = \frac{N \times F \times D}{W \times 3\cdot 7 \times 10^4} ,$$

where:

S_o = specific activity (μc/g) of original solution at time t_o,

N = corrected disintegration rate (disintegrations/sec) for the source counted at time t,

F = correction factor for decay during the interval $(t - t_o)$,

W = Mass (g) before drying, of the solution counted ,

D = dilution factor,

(g) The value of "N" is also calculated from the $4\pi\beta$ count rate (correcting for dead time).

(h) The beta efficiency E_β is calculated

$$E_\beta = \frac{(N)_\beta \times 100}{(N)_c} \quad \text{per cent.,}$$

where:

$(N)_\beta$= "N" calculated from the $4\pi\beta$ count, and

$(N)_o$= "N" calculated from the $4\pi\beta$-γ coincidence counts

If E_β is less than about 85 per cent. it may be necessary to make additional corrections to the coincidence equation (as discussed in Sections 3.2.1 to 3.2.7).

(i) The standard deviation due to random counting errors is calculated.

7. PROCEDURE ADOPTED AND RESULTS OBTAINED FOR THE STANDARDISATION OF SOME COMMONLY USED ISOTOPES

Standardised solutions of the β emitting isotopes Tl-204, P-32, and the β-γ emitters, Co-60, Au-198, I-131, Sb-124 have been obtained from overseas laboratories. Measurements on these solutions have been made using the counters described. The results have generally been within the certified limits of accuracy of the standards. Several other shorter-lived isotopes (Y-90, Na-24, K-42) have also been supplied by the Commission's Isotope Division, and some experience has been gained in standardising them. The procedure adopted and results for each isotope are given below. The decay schemes and radiation data shown in Table 2 were taken from Strominger, Hollander, and Seaborg (1958) and Allen, Smith, and Hiscott (1961).

7.1 Pure Beta Emitters

For the beta emitters, foil absorption corrections were made by using curves published by Pate and Yaffe (1955c) and source self-absorption corrections were made by reference to the curves of Merritt, Taylor, and Campion (1959).

7.1.1 Thallium 204

Thallium 204 was standardised when participating in an international intercomparison involving 17 other standardising laboratories. The National Physical Laboratory distributed two ampoules, each containing approximately 1 g of solution of specific activity about 100 µc/g, to each laboratory.

The low beta energy (see Table 2) introduces a significant self-absorption correction (up to 5 per cent.) which is difficult to assess. This factor is mainly responsible for the large spread in the results obtained. The specific activity obtained for the two ampoules received by this laboratory is shown in Table 3.

An estimate of the efficiency of the $4\pi\beta$ counter for the X-rays associated with the electron capture decay, showed this to be very low. A 2 per cent. correction for electron capture was therefore made.

The average value for the two ampoules was 101.9 uc/g. The mean value for all 18 countries was 101.1 uc/g with a standard deviation of 0.9 per cent. and a range of 14 per cent.

7.1.2 Phosphorus 32

Measurements were made on a standard phosphorus 32 source obtained from the Radiochemical Centre, Amersham, U.K. The decay scheme is shown in Table 2 and the results in Table 3. No correction was made for self absorption because of the high beta energy. A long-lived beta impurity (thought to be S-35) was found in the sources counted and a 5 per cent. correction for this was made; this was determined by repeated measurements over a period of 155 days, and extrapolation back to the reference time.

7.1.3 Yttrium-90

The yttrium-90 source measured was a non-standard one supplied by the Commission's Isotope Division. No corrections were applied other than for background and counter dead time. The results (Table 3) show the small spread in measurements obtained from a proportional beta counter when there is no appreciable self-absorption.

7.2 Beta-Gamma Emitters

The radiation data for the beta-gamma emitters standardised is shown in Table 2. Details of the sources standardised, and the results obtained are given in Table 4.

7.2.1 Cobalt-60

Because of the high beta efficiency (>90 per cent.) obtained for cobalt-60 and the simple decay scheme (see Table 2) no corrections were made apart from the usual dead time, background, and chance coincidence corrections. The only other correction which could be significant for this isotope is that due to the detection of γ-rays in the β-detector and γ-γ coincidences. To determine the order of magnitude of this correction, a Co-60 source was sandwiched between two gold coated Perspex plates, thick enough to absorb the 0.31 MeV betas. The β and γ,γ coincidence count rates for this source (which had been previously standardised in the usual way) were found. The value determined for the γ efficiency of the β detector was approximately 0.023 and the probability of γ-γ coincidences, 0.00093. Evaluating Equation 8 by putting $E_\beta = 0.90$, $(E_\beta)_\gamma = 0.023$, $E_c = 0.00093\ (1-E_\beta) = 0.000093$ and ${}^{\beta}E_\gamma = 0.04$, the correction factor becomes 1.0012.

Although a thorough investigation of source preparation techniques has not been made for Co-60, four methods have been tried. Pre-treatment of the foils with insulin or the addition of colloidal silica generally gave less self-absorption than that obtained with untreated sources. In general, the best results were obtained by precipitation. This was done in the way described by Merritt, Taylor, and Campion (1959). A drop of distilled water is added to a source which has been previously weighed and dried. The source is then re-dried in an atmosphere of ammonia provided by rinsing a beaker with concentrated ammonium hydroxide solution and inverting it over the source.

Good counting accuracy can be obtained for source activities between 0.02 and 0.2µc, the optimum activity being about 0.1µc.

7.2.2 Gold-198

Beta efficiency of about 95 per cent. has been obtained for gold-198 so all corrections which depend on the value of $(1-E_\beta)$ are small. The two greatest corrections of this type are due to multiple branching (see Table 2) and internal conversion; the magnitude of these can be estimated as follows:

Neglecting the minor branch (1.37 MeV), we can use the correction given in Section 3.2.7 for two branches (Equation 10).

The gamma efficiency for the scintillation counter has been found to vary from 2.4 for gold-198 (0.41 MeV) to about 2.1 per cent. per γ ray for cobalt-60 (1.2 MeV); the values of $E_{\gamma a}$ and $E_{\gamma b}$ can therefore be considered to be about 2.5 per cent. and 1.5 per cent. at worst (that is, to give the worst value for $E_{\gamma a} - E_{\gamma b}$). $E_{\beta a}$ will be close to the observed overall efficiency, that is, 0.95, and $E_{\beta b}$, in view of the observed efficiency of 90 per cent. for Co-60, is probably greater than 0.85. The worst value for the correction can then be found by putting a = 0.99, b = 0.01, $E_{\beta a} = 0.95$, $E_{\beta b} = 0.85$, $E_{\gamma a} = 0.025$ and $E_{\gamma b} = 0.015$, giving a value of 0.9995 for the correction factor.

The order of effect of internal conversion can be estimated by considering the "a" branch only and applying the correction given in Section 3.2.4 (Equation 7).

The efficiency for the monoenergetic conversion electrons will be very close to 100 per cent, so,

$E_{ce} = 1.0$, $E_{\beta} = 0.97$ and $\alpha = 0.044$, giving a value of 1.0013 for the correction factor. This is in the opposite direction to the decay scheme correction and both can be ignored if errors less than 0.1 per cent. are not significant.

In general no significant difference in self-absorption was found between untreated sources and sources treated with insulin or silica. However, untreated sources occasionally show high self-absorption and it is generally advisable to use insulin-treated foils. For good counting statistics, sources used were in the range 0.04μc to 0.2μc, the optimum value being about 0.1μc.

7.2.3 Iodine-131

An estimate of the order of the decay scheme correction for iodine-131 can be obtained in the following way:

The overall β and γ efficiencies observed with this counter were 93 per cent. and 2.3 per cent. respectively. We can approximate to the I-131 decay scheme (see Table 2) by ignoring branch d and considering branches b and c together as one branch, (b'). Both β and γ energies in these two branches are similar, so no great error will be introduced by this simplification. From the observed overall efficiencies we can say that at worst $E_{\beta a} = 0.93$, $E_{\beta b'} = 0.90$, $E_{\gamma a} = 0.025$, and $E_{\gamma b'} = 0.015$; also a = 0.87 and b' = 0.12.

When these values are inserted into the expression for double branching (Equation 10), the correction factor becomes 0.9986.

The order of the internal conversion correction can be estimated by considering the main branch only and applying the correction given in Section 3.2.4 (Equation 7).

Here the worst effective value of $\alpha = \frac{5.3}{87} = 0.061$, and $E_c = 1.0$ and $E_\beta = 0.93$, which gives a value for the correction factor of 1.0043. This is in the opposite direction to the decay scheme so the overall correction will at worst be less than 0.3 per cent.

A third correction for I-131 is necessary to account for the highly converted γ from Xe-131^{m2}. However, this correction can be evaluated fairly accurately. The monoenergetic conversion electron will be detected with nearly 100 per cent. efficiency and is not coincident with the I-131 betas.

Consequently:

$$N_\beta = N\,(E_\beta + 0.007)$$

$$N_\gamma = N\,E_\gamma$$

$$N_c = N\,E_\beta E_\gamma \quad .$$

Therefore:

$$\frac{N_\beta N_\gamma}{N_c} = N\left(1 + \frac{0.007}{E_\beta}\right) \quad . \qquad \ldots. \ (24)$$

Here $E_\beta = 0.93$ so the correction factor becomes 1.0075.

The main problem in depositing iodine is loss of active material by evaporation. It has been found that up to 0 per cent. can be lost from untreated sources in this way. However, precipitation of the iodine as silver iodide has been found to be almost 100 per cent. successful. A trace of silver-nitrate solution is added to the source drop on the foil as soon as it has been weighed. The minimum quantity of silver nitrate required appears to be about 0.2 mg $AgNO_3$ per g of source solution for the strength of iodine solutions usually measured. However, more careful investigations may show that smaller amounts of $AgNO_3$ may be effective.

The most useful range of source activity for I-131 is about 0.04μc to 0.2μc, the optimum being about 0.1μc.

7.2.4 Antimony-124

Antimony-124 has a complicated decay scheme and consequently no attempt has been made in Table 2 to identify the gammas with any particular branch. There is a 0.6 MeV cascade gamma, for example, coincident with nearly all betas.

The decay scheme correction may be estimated roughly by making the following assumptions:

(a) E_β for the 0.22 MeV branch ($E_{\beta a}$) = 0.85 or more.

(b) E_β for all other branches together ($E_{\beta b}$) = 0.98 (observed overall efficiency for all branches being 0.97).

(c) E_γ for the 0.22 MeV β branch ($E_{\gamma a}$) = 0.02. The reasons for assumption (c) are as follows: The decay scheme shows the following gammas associated with this branch: 2.09 MeV (6 per cent.) followed by a 0.6 MeV γ in cascade; also 1.37 MeV (5 per cent.) followed mainly by two cascades, 0.72 MeV and 0.6 MeV. The mean energy is therefore about 1.08 MeV. The γ efficiency for the counter used has been found to vary from about 2.4 per cent. for gold-198 gammas (0.41 MeV) to about 2.1 per cent. (per gamma ray) for cobalt-60 (1.2 MeV). The lowest efficiency likely at 1.08 MeV is therefore about 2.1 per cent.

(d) E_γ for all other branches together ($E_{\gamma b}$) which is predominantly about 0.6 MeV, will be not more than 0.024.

The double branch correction factor, Equation 10, can therefore be evaluated, making a = 0.11, b = 0.89, $E_{\beta c}$ = 0.98, $E_{\gamma a}$ = 0.021 and $E_{\gamma b}$ = 0.024, yielding a value of 0.9983.

It should be possible to reduce the magnitude of this error by using a single channel analyser to discriminate against all gammas except the 0.60 MeV one. This gamma is coincident with about 99 per cent. of the betas and so the gamma efficiency becomes virtually the same for all branches. Since none of these gammas are internally converted, the term ($E_{\gamma a}$ - $E_{\gamma b}$) in Equation 10 becomes very nearly zero.

With the low efficiency gamma detector at present in use the gamma count rate becomes very small when restricted to a narrow channel so the method has not been used. However, it is proposed to install larger NaI crystals to make the single channel analyser very useful for "simplifying" complicated gamma decay schemes such as antimony-124.

No appreciable difference was observed between treated and untreated sources, but, as before, treatment with insulin or colloidal silica was used. Source activities in the range 0.02 to 0.2μc can be counted, the optimum value being about 0.1 μc.

7.2.5 Sodium-24

Owing to the simple decay scheme and high β efficiency obtained (~ 99%) for sodium-24, no special corrections need be considered.

7.2.6 Potassium-42

Owing to the high β energies for potassium-42, 100 per cent. efficiency should be obtained, so all corrections dependent on β efficiency (including the effect of the significant amount of bremsstrahlung from the 3.6 MeV β) are negligible.

Owing to the low γ intensity, a source activity of about 0.2μc is required for accurate counting.

This was the first isotope standardised and the sources were prepared on single VYNS foils gold coated on one side only. Subsequent tests using Co-60 showed nearly 4 per cent. increase in the efficiency of the 4πβ counter when both sides of the foils were gold coated. This probably accounts for the low efficiency (~ 97.3 per cent.) shown for K-42 in Table 4.

8. CONCLUSIONS

The instruments and methods described were found satisfactory for the standardisation of many beta and beta-gamma emitting isotopes to an accuracy of ± 2 per cent. or better.

A detector head with higher gamma efficiency is desirable to increase the range of source activities which can be accurately counted, and to permit the use of a pulse amplitude analyser for the selection of particular photo-peaks in isotopes with complex decay schemes. A detector head consisting of two 3 inch x 1 inch NaI(Tl) crystals and a re-designed 4π proportional counter should give a gamma efficiency of about 28 per cent.

Low energy pure beta emitters and electron capture isotopes cannot be standardised satisfactorily at present. The main reason for this is the uncertainty in correcting for the source self absorption of low energy betas and X-rays. Improved techniques for source deposition or for the measurement of self-absorption are required. The electro-spraying method of Carswell and Milstead (1957), for obtaining thin sources, and the tracer technique of Campion, Taylor, and Merritt (1960) for the measurement of self absorption seem to be the most promising of the techniques described in the literature.

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TABLE 1

INTERFERENCE COLOURS FOR VYNS FILMS(1)

Superficial density ($\mu g/cm^2$)	Film thickness ($m\,\mu$)	Calculated(2) wavelength(λ) for reinforcement ($m\,\mu$)	Calculated(2) wavelength(λ) for destruction ($m\,\mu$)	Colour observed
1	7	14	21	Dark grey
5	36	72	108	Light grey
10	70	140	210	White
20	140	280	420	Light yellow
25	180	360	540	Yellow brown
30	210	420	630	Purple)
35	250	500	750	Blue)n=1
40	290	580	870	Yellow)
45	320	640	960	Red)
> 50	> 360	–	–	Colours of diminishing intensity ($n \geqslant 2$)

(1) Due to Pate and Yaffe (1955c).

(2) For reinforcement, $(n + \frac{1}{2})\lambda = 2\mu d \cos \theta$,

for destruction, $n\lambda = 2\mu d \cos \theta$,

where μ = refractive index (1.5 for VYNS)

d = film thickness

θ = angle of reflection

n = integer

TABLE 2 ISOTOPE RADIATION DATA

Isotope and Half-life	Radiation and Energy (MeV)				Production Process	Production Impurities
	Branch	Beta	Gamma	I.C.		
Tl-204 3.9y	a	0.77 (98%) E.C. (2%)	-	-	$Tl203(n,\gamma)Tl^{204}$	None
P-32 14.2d	a	1.71 (100%)	-	-	$P^{31}(n,\gamma)P^{32}$	None
Y-90 64.2h	a	2.25 (100%)	-	-	$Y^{89}(n,\gamma)Y^{90}$	None
Co-60 5.27y	a	0.31 (100%)	1.17 (100%) 1.33 (100%)	-	$Co^{59}(n,\gamma)Co^{60}$	Co^{60m}
	b	1.48 (0.01%)	-	-		
Au-198 2.70d	a	0.96 (~ 99%)	0.412 (95.6%)	4.2%	$Au^{197}(n,\gamma)Au^{198}$	Au^{199}
	b	0.29 (1%)	0.68 (1.1%) 1.09 (0.26%)	-		
	c	1.37 (0.025%)	-	-		
I-131 8.04d	a	0.61 (87%)	0.36 (80%) 0.28 (5%) 0.08 (2%)	1.0% 0.3% 4.0%	$U(n,f)Te^{131}$ $Te^{131} \xrightarrow{\beta} I^{131}$ (24.8m)	None
	b	0.33 (9%)	0.64 (9%)	-		
	c	0.25 (3%)	0.72 (3%)	-		
	d	0.81 (1%)	0.163 (0%) Via 12 day Xe^{131m}	0.7%		

(continued)

TABLE 2 - (continued)

Isotope and Half-life	Radiation and Energy (MeV)				Production Process	Production Impurities
	Branch	Beta	Gamma	I.C.		
Sb-124 60 d		0.22 (11%) 0.61 (51%) 0.95 (6%) 1.59 (7%) 1.66 (2%) 2.31 (23%)	0.60 (99%) 0.65 (8%) 0.72 (14%) 0.97 (3%) 1.05 (2%) 1.33 (1%) 1.37 (5%) 1.45 (2%) 1.70 (50%) 2.09 (6%)	-	$Sb^{123}(n,\gamma)Sb^{124}$	Sb^{122m} Sb^{122} Sb^{124m}
Na-24 15.0h	a	1.39 (100%)	1.37 (100%) 2.75 (100%)	-	$Na^{23}(n,\gamma)Na^{24}$	K^{42}
K-42 12.45h	a	3.6 (82%)	-	-	$K^{41}(n,\gamma)K^{42}$	Na^{24}
	b	2.0 (18%)	1.52 (18%) 0.32 (weak)	-		

TABLE 3

RESULTS OF TRIAL STANDARDISATIONS BY 4πβ PROPORTIONAL COUNTER

Details of Sources:							
Isotope	Tl-204			Tl-204			
Supplier	N.P.L.			N.P.L.			
Source number	Ampoule 53			Ampoule 55			
Chemical form	Tl_2SO_4			Tl_2SO_4			
Solvent	$N/10\ HNO_3$			$N/10\ HNO_3$			
Mass	1.0381 g			1.0g			
Certified activity	101.1 μc/g (1)			101.1 μc/g (1)			
Accuracy	0.9% (2)			0.9% (2)			
Results Obtained:							
Dilution factor	972.4	471.9	287.3	1021.4	293.8	220.3	82.21
Foils counted	4	5	2	4	2	4	2
Activity (μc/g)	104.2	103.1	104.9	98.8	101.7	98.7	99.5
Mean activity(μc/g)	104.1 μc/g			99.7 μc/g			
σ_{mean} (3) (%)	0.44%			0.48%			
Max. counting errors	0.1%			0.1%			
Corrections Applied:							
Background (%)	0.07 to 2.0						
Paralysis (%)	0.2 to 5.0						
Foil absorption (%)	0.1 to 0.2						
Self absorption (%)	2.5						
Electron capture (%)	2.0						
Decay (%)	0.001 to 0.6						

(1) Mean of results from 18 laboratories.

(2) Standard deviation of the mean of the 18 results.

(3) σ_{mean} = Standard deviation of mean = $\frac{100}{x}\sqrt{\frac{\Sigma(x-\bar{x})^2}{n(n-1)}}$

TABLE 3 (Continued)

Details of Sources:		
Isotope	P-32	Y-90
Supplier	Radiochem. Centre	Isotope Division, A.A.E.C.
Source number	PO304	Order No. 473
Chemical form	NaH_2PO_4	-
Solvent	H_2O + 0.1% Formalin	-
Mass	1.012g	10.1198g
Certified activity	1650 µc/g	-
Accuracy	± 2%(4)	-
Results Obtained:		
Dilution factor	49.79	487.8
Foils counted	2	3
Mean activity (µc/g)	1690	229.6(5)
σ (mean) (%)	0.61%	0.14%
Max. counting error %	0.1%	0.1%
Corrections Applied:		
Background (%)	0.1	0.2
Paralysis (%)	3	5
Foil absorption (%)	nil	nil
Self absorption (%)	nil	nil
Impurity (%)	5	nil
Decay (%)	350	20 to 90

(4) Quoted as "effective error". Maximum certified error was $^{+6\%}_{-4\%}$.

(5) Further measurements, repeated over 5 half-lives showed a spread of less than 0.25% for each foil.

TABLE 4

RESULTS OF TRIAL STANDARDISATIONS BY $4\pi\beta$-γ COINCIDENCE COUNTER

Details of Sources:			
Isotope	Co-60	**Au-198**	I-131
Supplier	Radiochem. Centre	I.A.E.A.	I.A.E.A.
Source No.	P0309	Amp. No. 3	Amp. No. 24
Chemical form	$Co(NO_3)_2$	$Au\ Cl_3$	NaI
Solvent	N/100 HNO_3	See Note (2)	See note (3)
Mass (g)	1.118	3.999	4.025
Cert. activity(μc/g)	93.20	11.25	13.53
Accuracy	±1%	±1%	±1%
Results Obtained:			
Dilution factor	93.82	1.0	1.0
Foils counted	5	4	2
Mean activity(μc/g)	93.7	11.40	13.59
$\sigma_{(mean)}$ (%)	0.12	0.18	0.95
Max. counting errors (%)	0.15 to 0.4	0.25	0.13
Beta efficiency E_β(%)	92	95	93
Gamma eff. E_γ (%)	4.3	2.3	2.3
Corrections Applied:			
Background (%)	3 to 13	6 to 16	5 to 12
Paralysis (%)	0.1 to 0.5	0.05 to 0.15	0.2
Chance coincid. (%)	0.4 to 2.0	0.05 to 1.2	1 to 2
Conversion (%)	nil	nil	0.75
Decay (%)	1.0	100 to 1700	70 to 1400

(1) "Effective error" ± 1%, maximum error ± 3%.

(2) 122 μg KCN/g in H_2O.

(3) 20 mg NaOH + 20 $Na_2S_2O_3$ per litre of H_2O.

TABLE 4 (Continued)

Details of Sources:			
Isotope	Sb-124	Na-24	K-42
Supplier	Radiochem. Centre.	A.A.E.C.	A.A.E.C.
Source No.	P0315	Order No.133	Order No.133
Chemical form	$SbCl_3$	Na_2CO_3	K_2CO_3
Solvent	2.5N HCl	H_2O	H_2O
Mass (g)	1.101	∼10	∼10
Cert.activity(μc/g)	140	-	-
Accuracy	± 2%(4)		
Results Obtained:			
Dilution factor	95.45	100.45	96.28
Foils counted	3	4	3
Mean activity(μc/g)	143(5)	60.67(6)	12160
σ (mean) (%)	0.77	0.18	0.17
Max. counting errors (%)	0.06 to 0.38	0.1 to 0.35	0.35 to 0.91
Beta efficiency E_β (%)	96	98	97.3(7)0
Gamma eff. E_γ (%)	4.3	4.4	0.61
Corrections Applied:			
Background (%)	4 to 12	9 to 19	20 to 30
Paralysis (%)	0.05 to 0.15	0.05 to 0.14	0.02
Chance coincid.(%)	0.4 to 1.0	0.3 to 0.6	0.3 to 2.6
Decay (%)	24 to 71	41 to 54	0-300

(4) Maximum certified error ± 6%.

(5) The decay rate indicated the presence of a long-lived impurity. This was not identified and no correction was applied.

(6) This result was used to calibrate a substandard $4\pi\gamma$ ion chamber which has subsequently been found accurate to ± 2%.

(7) Single foils coated with gold on one side only were used.

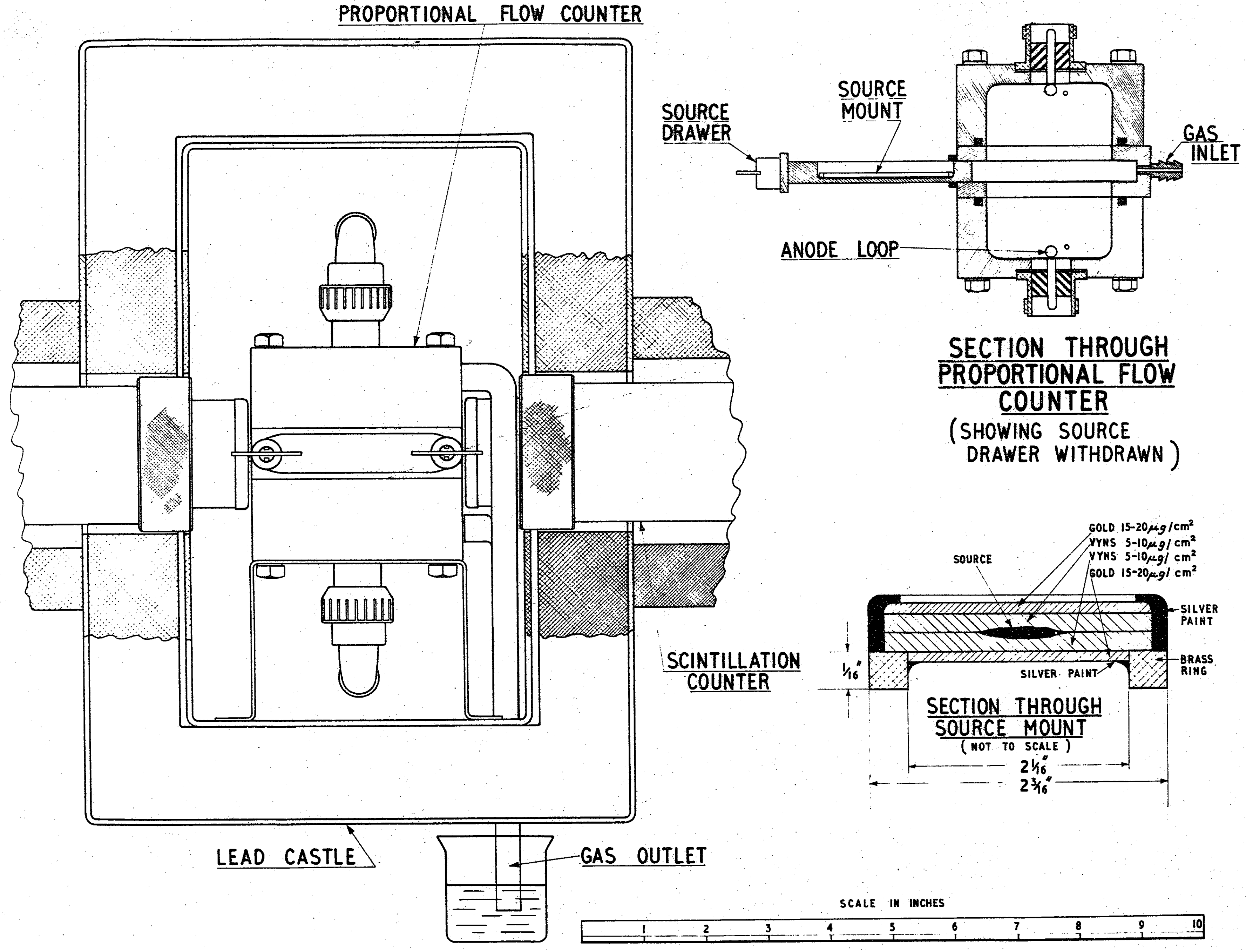

FIGURE I. $4\pi\beta$-γ COINCIDENCE COUNTING HEAD.

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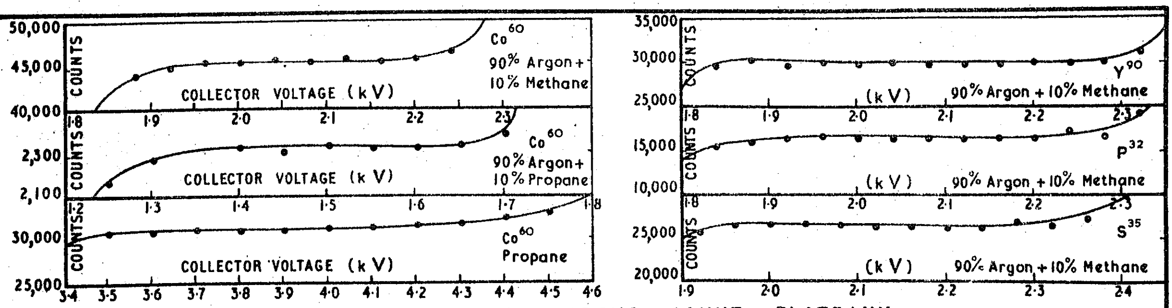


FIGURE 2a E.H.T. VERSUS COUNT — PLATEAUX

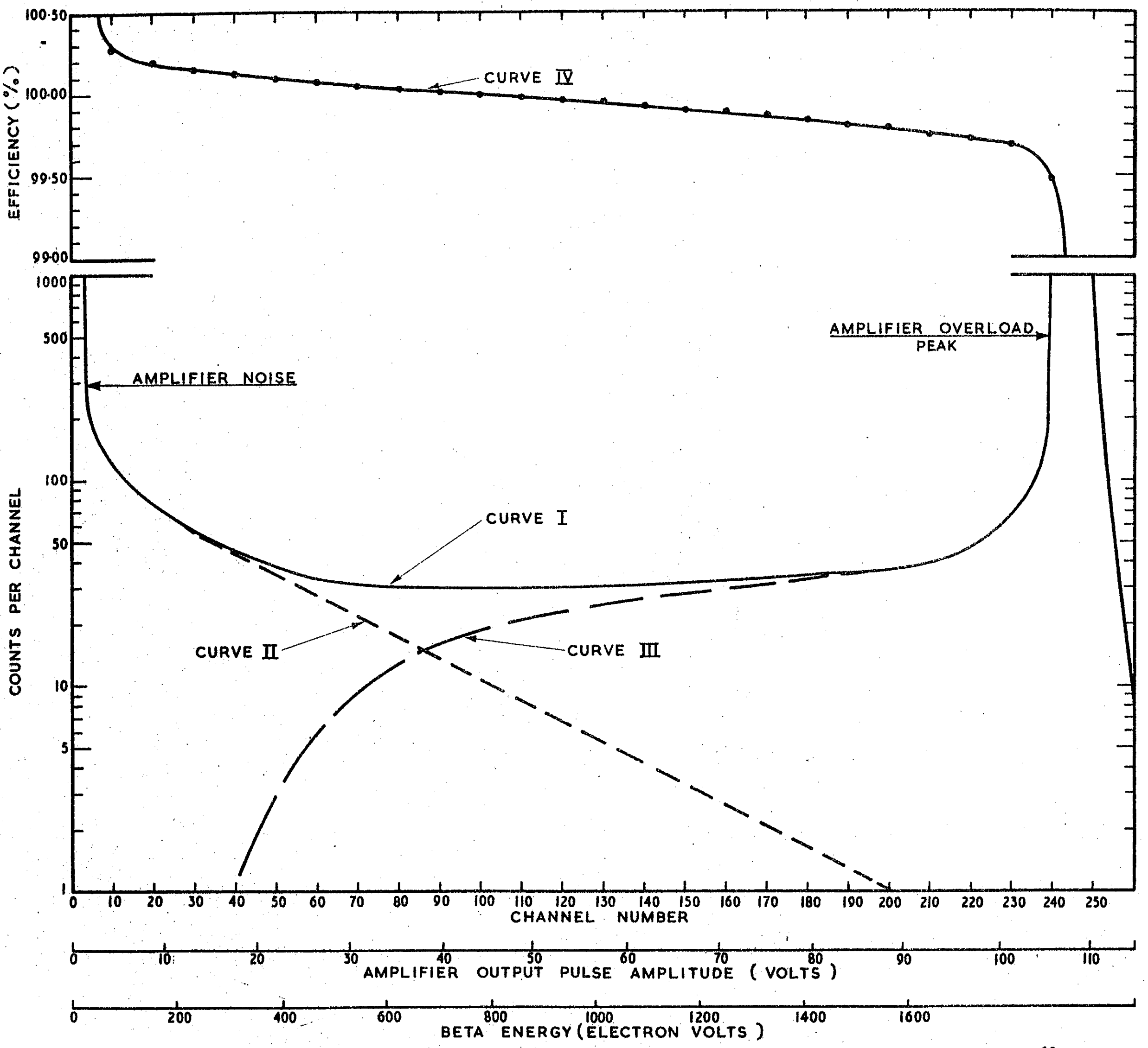


FIGURE 2b. DIFFERENTIAL AND INTEGRAL DISCRIMINATOR BIAS CURVES FOR Co^{60} BETAS IN ARGON-METHANE GAS FOR AN AMPLIFIER GAIN OF 10,000 AND AN E.H.T OF 1920 VOLTS.

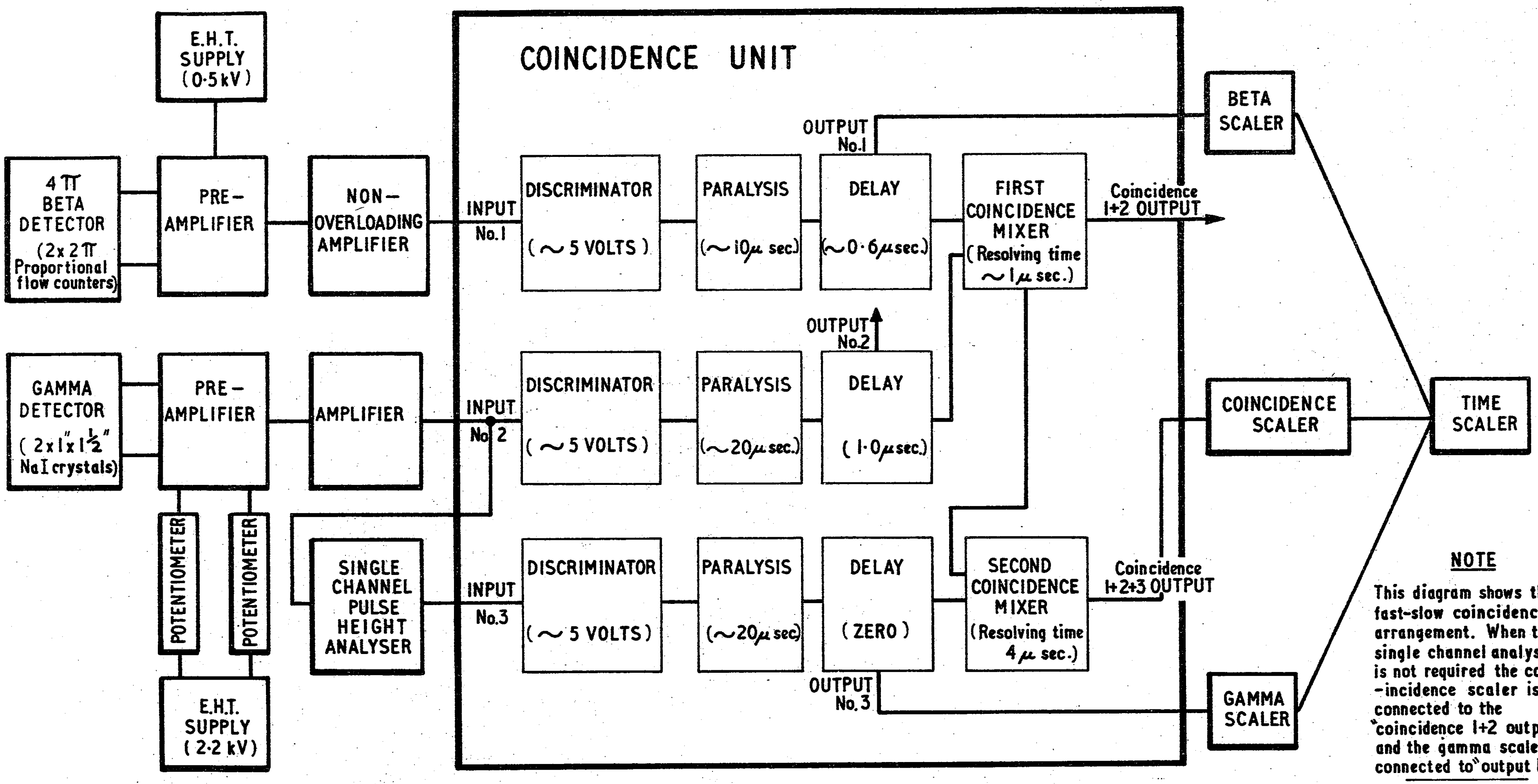
CURVE I. OBSERVED PULSE HEIGHT DISTRIBUTION AT THE OUTPUT OF THE NON-OVERLOADING AMPLIFIER.

CURVE II. ESTIMATED DISTRIBUTION OF "AFTER PULSES", BY EXTRAPOLATION.

CURVE III. CURVE II SUBTRACTED FROM CURVE I. AREA UNDER THIS CURVE APPROXIMATES THE NUMBER OF BETAS ENTERING THE COUNTER

CURVE IV. AREA UNDER CURVE I (above any channel number) DIVIDED BY THE TOTAL AREA UNDER CURVE III. THIS CURVE SHOWS EXPECTED COUNTER EFFICIENCY AS A FUNCTION OF DISCRIMINATOR BIAS LEVEL.

FIGURE 2. CHARACTERISTICS OF 1364A 4πβ PROPORTIONAL COUNTER



NOTE

This diagram shows the fast-slow coincidence arrangement. When the single channel analyser is not required the co-incidence scaler is connected to the "coincidence 1+2 output" and the gamma scaler is connected to "output No.2"

FIGURE 3. BLOCK DIAGRAM OF 4π β-γ COINCIDENCE COUNTER

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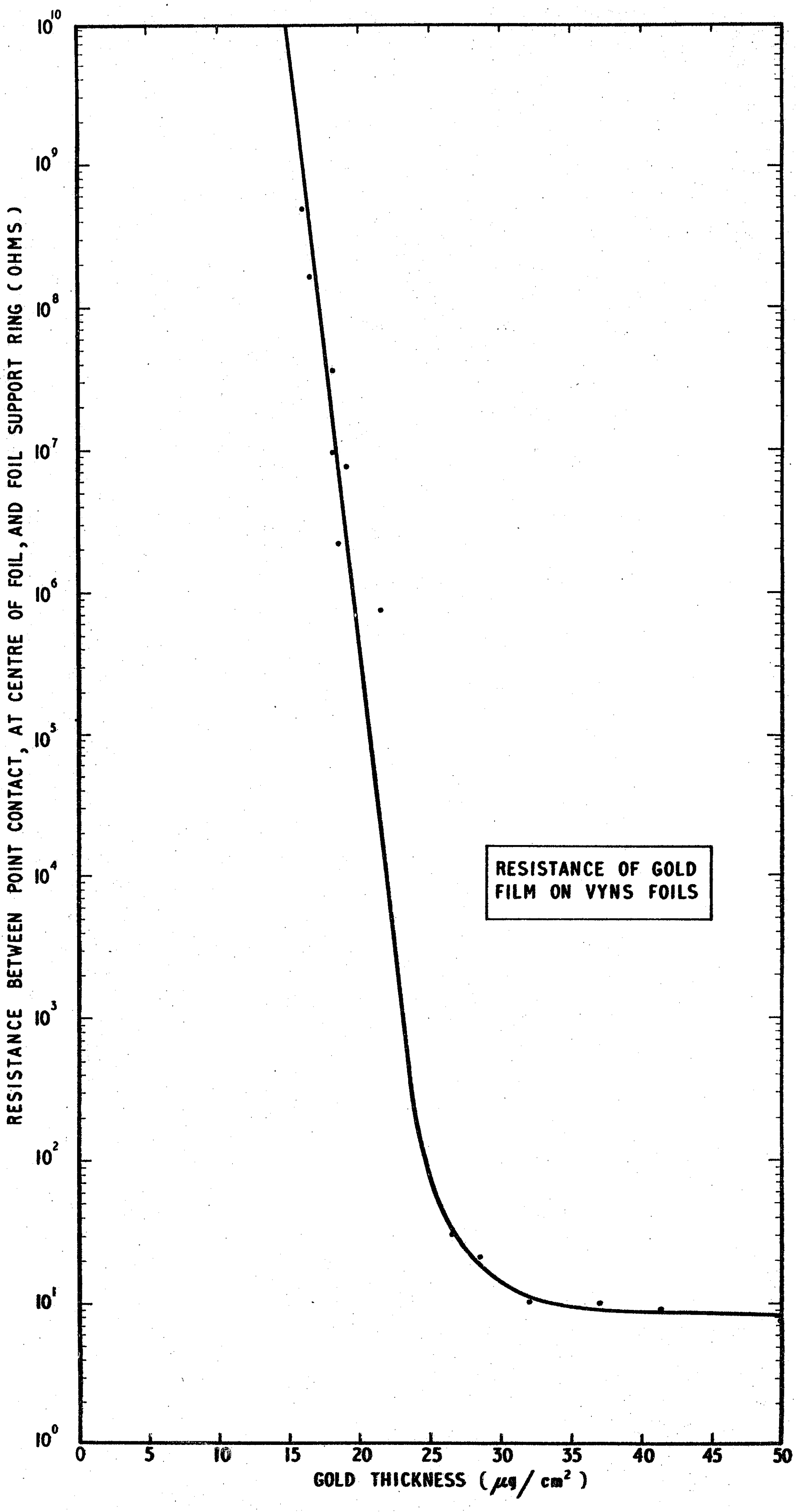

FIGURE 4. RESISTANCE OF GOLD FILM ON VYNS FOILS

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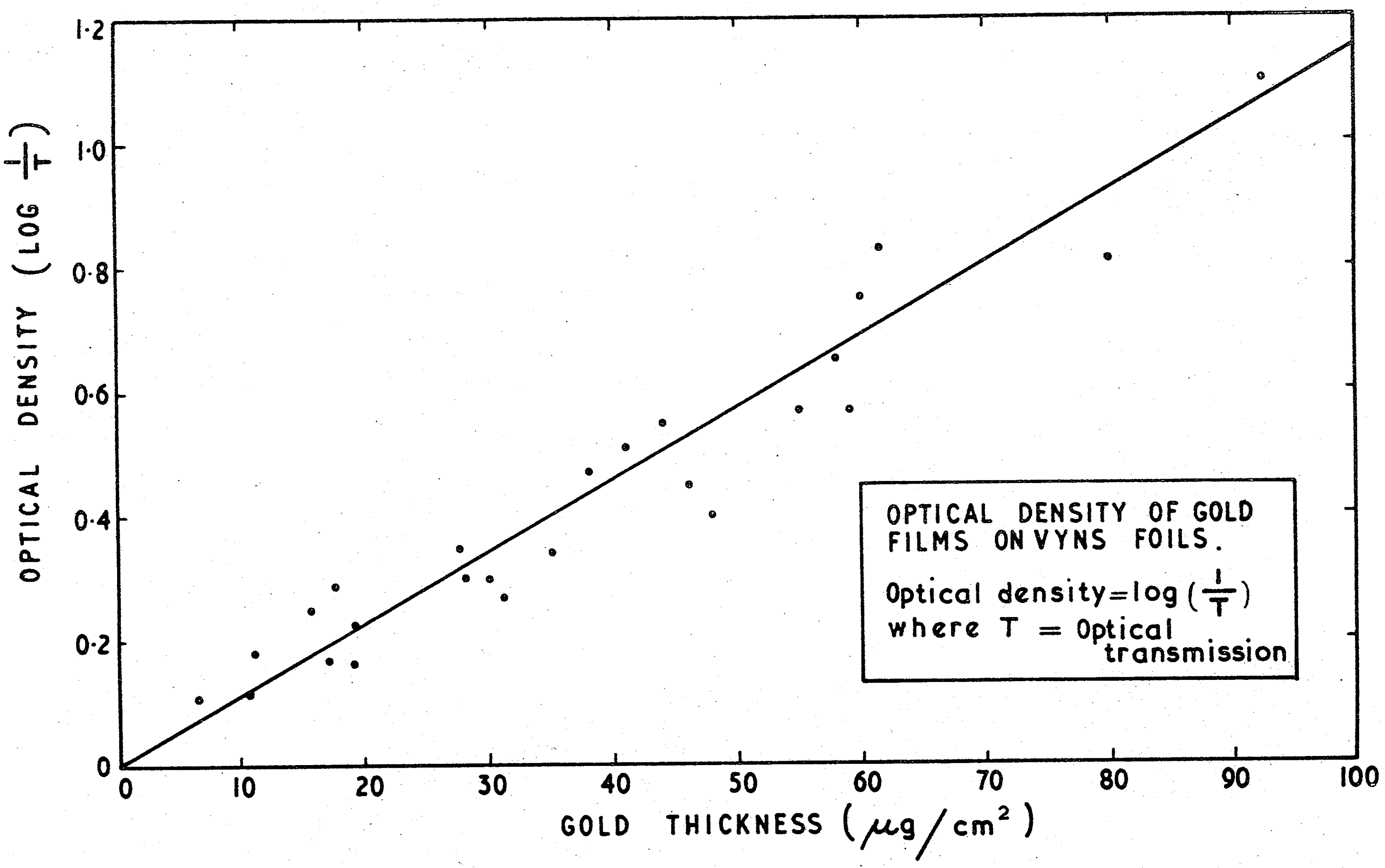

FIGURE 5. OPTICAL DENSITY OF GOLD FILMS ON VYNS FOILS

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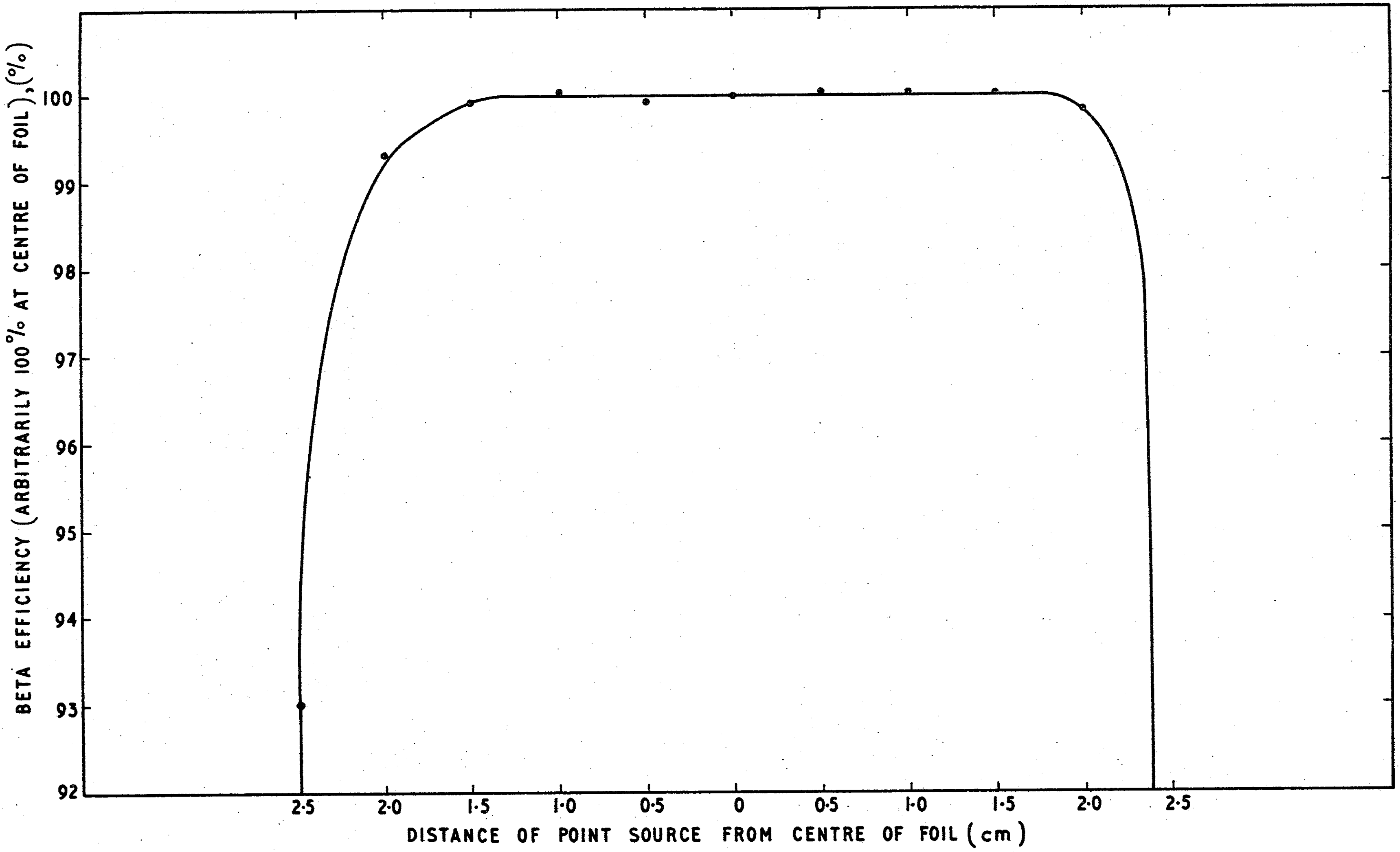

FIGURE 6. EFFECT OF SOURCE POSITION ON THE EFFICIENCY OF THE 1364 A COUNTER
(FOR POINT SOURCE OF Au^{198})

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