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Measurement of the Absolute Activity of ^{235}U by the Generalized Coincidence Method

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The absolute activity of ^{235}U contained in a sample was measured utilizing a sum-coincidence circuit which selects only the α-particles emitted simultaneously with the 143 and 186 keV gamma radiations from the ^{231}Th (product nucleus).

The α-particles were detected by means of a new type of a gas scintillating chamber, in which the light emitted by excitation of the gas atoms, due to the passage of a charged incoming particle, has its intensity increased by the action of an applied electric field. The γ-radiations were detected by means of a $1'' \times 1^{1/2''}$ NaI(Tl) scintillation detector.

The value obtained for the half-life of ^{235}U, $(7.04 \pm 0.01) \times 10^8$ y, was compared with the data available from various observers which used different experimental techniques. It is shown that our results are in excellent agreement with the best data available on this subject.

Introduction

The absolute measurement of the activity of α–γ emitters whose decay schemes are simple is frequently measured by the method of coincidences whose advantages were emphasized by several observers (Campion, 1959; Gandy, 1962; Roost *et al.*, 1968). However, for radionuclides which show a complex disintegration scheme, the measurement of their absolute activity becomes very complicated owing to the necessity of correcting for several factors as well as to the inexistence of a generalized formula of this method for any radionuclide.

In this case, special care must be taken with the design of the detection system of the α- and γ-radiations in order that the obtained data can be used with success.

With this purpose in mind, an α-detector was built whose characteristics are more suitable for such measurements. This detector is constituted essentially by a gaseous scintillator, built in such a way that the number of photons produced by the α-particle along its track is multiplied by the action of an electric field applied to its anode, operating either in the proportional region or in the region of the limited proportionality.

These photons are channeled through a light pipe to the window of a photo multiplier tube in order to obtain an electric pulse whose amplitude is proportional to the energy loss of the particle in the gaseous scintillator.

In our arrangement, the γ-radiation due to the ^{235}U (enriched at 19.99% in a sample of U_3O_8), was detected by means of a $1'' \times 1^{1/2''}$ NaI(Tl) of scintillator.

The precision obtained with this technique was checked comparing the measured ^{235}U half-life with the values obtained by various authors (Nier, 1939; Sayag, 1951; Fleming *et al.*, 1952; Würger and Meyer, 1957; Deruytter *et al.*, 1965; White *et al.*, 1965; Jaffey *et al.*, 1971; Deruytter and Penning, 1974) using different techniques; it is shown that our measurements agree, within the experimental error with the best measurement found in the literature.

The Generalized Coincidence Method

The absolute measurement of radionuclides activities can be made by the coincidence method (Campion *et al.*, 1960; Baerg, 1966). It's principle lies in the fact that whenever a radionuclide emits an ionizing particle the product nucleus usually is left in an excited state and, in the great majority of cases, its desexcitation is followed by emission of electromagnetic radiation. As such emission occurs in an extremely short time, usually between 10^{-9} to 10^{-12} s after the particle emission, those γ-rays can be considered coincident with the particle. In the ideal case

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this method has its application limited to the following conditions:

(a) simple disintegration scheme, the two radiations of different nature are simultaneous;
(b) the detectors used must be selective to each type of radiation measured and should show a long term stability;
(c) radioactive point source.

When those conditions are satisfied, it is shown that the measured activity is independent of the detectors efficiencies, of the geometry factors and of the disintegration scheme. In those conditions lies the great advantage of this method in relation to others, since the great majority of systematic errors are excluded and the activity is expressed only as a function of the observed data. However, when the coincidence method is applied to a nuclide which decays through a complex disintegration scheme, it is necessary to develop a generalized formula in order to calculate the absolute measurement of activity.

Let us consider a disintegration scheme with any α-groups, and let a_r be the branching ratio of the r-th group and ϵ_{α_r} the efficiency of the α-detector for that group. Consider also that at least one of the α-groups is followed by one or more γ-radiations γ_r, which are detected in the γ-counter with efficiency ϵ_{γ_r}. If N_0 is the number of α disintegrations per second, the observed counting rate in the α-channel (N_α), of the α-channel (N_γ) and the α–γ ($N_{\alpha\gamma}$) coincidences will be given by the following expressions:

$$N_\alpha = N_0 \cdot \sum_{r=1}^{n} a_r \cdot \epsilon_{\alpha_r} \tag{1}$$

$$N_\gamma = N_0 \cdot \sum_{r=1}^{n} a_r \cdot \epsilon_{\gamma_r} \tag{2}$$

$$N_{\alpha\gamma} = N_0 \cdot \sum_{r=1}^{n} a_r \cdot \epsilon_{\alpha_r} \cdot \epsilon_{\gamma_r}. \tag{3}$$

One should observe that for disintegration scheme in which one of the α-groups leaves the product nucleus in its fundamental state, or when the γ-radiations associated to one of the α-groups are selected, those groups are considered as being followed by γ-rays detected with zero efficiency. Under those hypothesis the activity of the source, N_0, will be:

$$N_0 = \frac{N_\alpha \cdot N_\gamma}{N_{\alpha\gamma}} \cdot \frac{\sum_{r=1}^{n} a_r \cdot \epsilon_{\alpha_r} \cdot \epsilon_{\gamma_r}}{\sum_{r=1}^{n} a_r \cdot \epsilon_{\alpha_r} \sum_{r=1}^{n} a_r \cdot \epsilon_{\gamma_r}}. \tag{4}$$

This formula shows that the correction factor can be reduced to unity if ϵ_{α_r} or ϵ_{γ_r} were constants, independent of r. In our measurements the above condition is satisfied by the proportional scintillator (ϵ_{α_r} is a constant independent of r).

Experimental

In our measurements a source of uranium, enriched at $(19.99 \pm 0.01)\%$ in ^{235}U, as U_3O_8 was used; the source was obtained by the evaporation technique in high vacuum and deposited on a stainless steel disk of 20 mm dia.

The detection of α- and γ-radiations was made using a gaseous proportional scintillator counter (Policarpo *et al.*, 1974; Khoury, 1981; Campos, 1984) and a $1 \times 1^{1/2''}$ NaI(Tl) scintillation counter, respectively. The U_3O_8 source was placed on this crystal in order to have the largest detection solid angle (Fig. 1). The system, constituted by the source and the detector was shielded from the background radiation by blocks of lead 5 cm thick.

The α-detector and its light guide, and the NaI(Tl) crystal were attached to RCA6342 photo-multiplier tubes with silicone grease in order to increase the light transmission. The photo multiplier tubes were directly connected to preamplifiers based on the AD509 operational amplifiers, in order to avoid the use of coaxial cables and to decrease the stray capacity of the anode in relation to ground and get the best signal to noise ratio. The pulses from the preamplifier were connected to the linear amplifiers and the γ-ray pulses were sent to a single channel analyzer, whose purpose was to select the γ-rays from ^{235}U and reject the pulses due to ^{238}U, ^{234}Th, etc from the U_3O_8 source. By

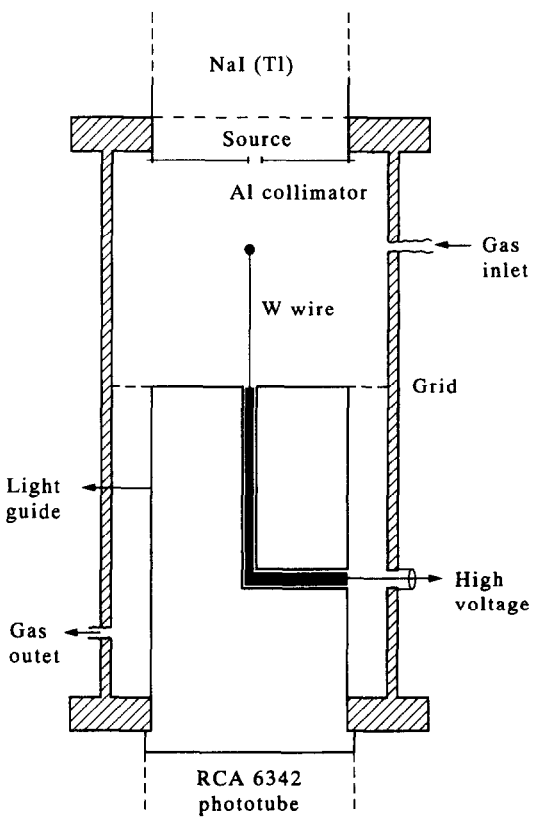

Fig. 1. Gas scintillation proportional counter (GSPC).

means of this procedure a selection of the γ-rays of interest from ^{235}U were selected and used in the coincidence line with the alpha pulses.

In order to obtain net pulses in the input coincidence analyzer, the pulses from both lines were normalized in order to obtain rectangular pulses of constant width and well defined amplitudes (we use different amplitudes for the α- and γ-lines, for reasons which will be described later). Those pulses were sent to the base of a BC337 transistor used as emitter-follower, used as a sum-coincidence amplifier, in order to obtain pulses from the collector with final amplitude equal to the sum of the amplitudes of the α- and γ-lines. Whenever the α- and γ-pulses are separated by a time interval larger than the resolving time of the system, one obtains in the output independent pulses whose amplitudes are smaller than the sum of the two pulses and have an amplitude which is defined by the monostable CD4047 (which is used to normalize the pulses from α- and γ-lines). The difference of amplitude of the three pulses are observed as three different lines, corresponding to three different channels numbers observed in the multichannel analyzer (ORTEC model 6240). In this way, individual counts of the α, γ and coincidences are simultaneously registered on the multi-channel analyzer. The block diagram is shown in Fig. 2.

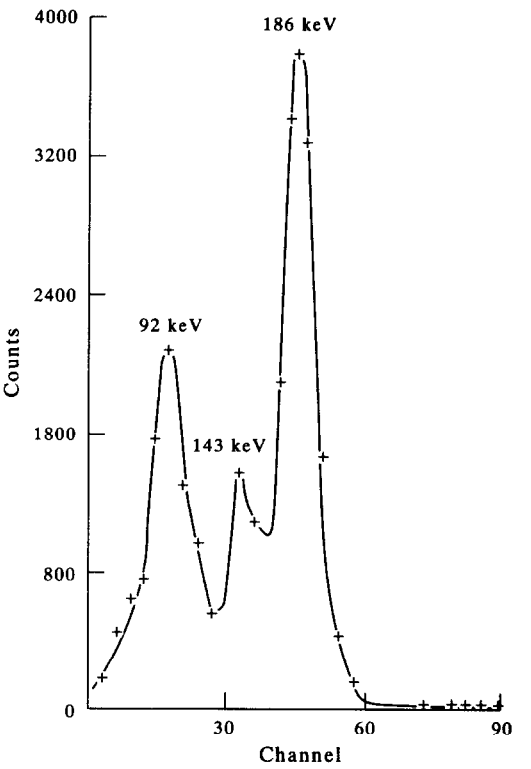

Fig. 3. Energy spectrum of the γ-radiation from the U_3O_8 source obtained with the NaI(Tl) detector.

Results

The energy spectrum of the γ-radiation from the U_3O_8 source obtained with a NaI(Tl) crystal is presented in Fig. 3. The peak at 92 keV is due to either ^{231}Th or ^{234}Th. The second and third peaks are due to γ rays of 143 and 186 keV, respectively, from ^{231}Th. In the coincidence measurements we have used only the γ-rays from ^{231}Th in the energy range between 143 and 186 keV.

The energy spectrum of the α-radiation from the U_3O_8 source obtained with the gaseous proportional scintillator is shown in Fig. 4. In this spectrum the α-particles due ^{234}U, ^{235}U, ^{236}U and ^{238}U are shown: the energy of the α-particles (Schmorak, 1982, 1983) from those isotopes are very close to each other and therefore indistinguishable by the gaseous proportional scintillator. It follows that in the α-line of the coincidence circuit the contributions from all the α-particles of the above groups are present and a correcting factor for the alpha radiation of ^{235}U of interest, through a measurement of its radiation must be introduced.

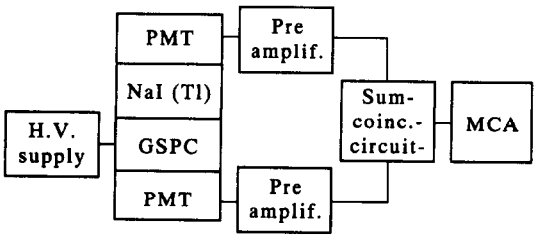

Fig. 2. Block diagram of the equipment used in this experiment.

Once those conditions were adjusted for the operation of both the α- and γ-detectors, a measurement of the coincidences were taken using the system presented in Fig. 5. One should notice that it is necessary to provide a selection of the pulse amplitudes corresponding to the energies between 143 and 186 keV through the adjustment of the discriminator levels V_1 and V_2; in a similar way the discrimination level of the α-particles should be adjusted V_0 which corresponds to the discrimination between true α-particles and the noise. The choice of V_0, V_1 and V_2 was made through a measurement of the energy spectrum of γ- and α-radiations from U_3O_8 source which are represented in Figs 3 and 4, respectively.

The observation of these spectra shows that the channel which corresponds to the beginning of the α-pulses, corresponding to V_0, is the channel 32 and the γ-line the levels V_1 and V_2 are associated to the channels 26 and 60, respectively. It can be easily shown that with such adjustment a 10 V level corresponds to the 1024 channel.

The final results of the α–γ coincidences from the U_3O_8 sample are shown in Fig. 6. The three observed lines are due to pulses from the α, γ and α–γ coincidences: the individual α and γ pulses are shown in the lines corresponding to 2.0 V and 4.8 V, respectively. When a coincidence occurs, a pulse of 6.8 V

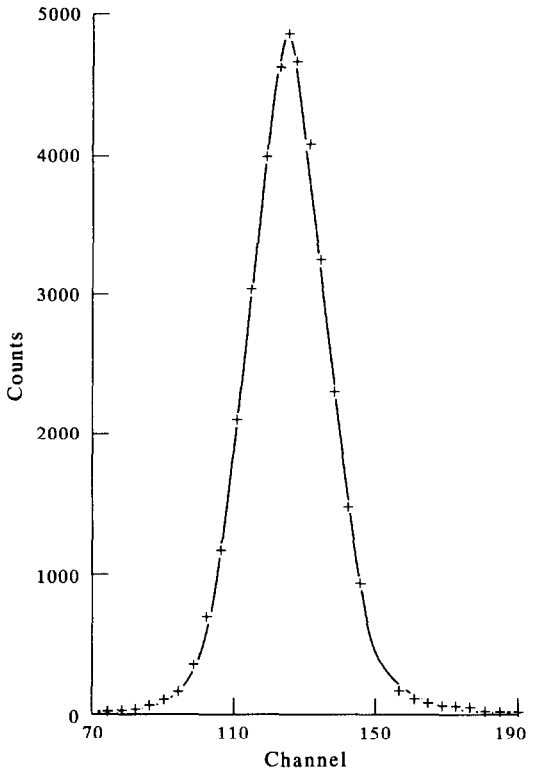

Fig. 4. Energy spectrum of the α-radiation from the U_3O_8 source obtained with the GSPC.

appears as a third line in the multi channel analyzer screen.

The obtained counting rates for the α (N_α), γ (N_γ) and the genuine coincidences were:

$$N_\alpha = (174.79 \pm 0.02)$$

$$N_\gamma = (9.030 \pm 0.005)$$

$$N_{\alpha\text{-}\gamma} = (2.421 \pm 0.002).$$

Prior to the calculation of the absolute activity of the sample, a careful analysis was made of the influence of the error inherent to the method of coincidences, as well as of the possible systematic errors of the measurements and are described in the following chapter.

Correction Factors and Evaluation of the Systematic Errors

1. Contribution of the α-activities of ^{234}U and ^{238}U in the measurements

In order to evaluate the contribution of the α-activities due to ^{234}U and ^{238}U in our measurements, the α-spectrum of a very thin U_3U_8 sample (from the same material used in our measurements) was made with a silicon surface barrier detector (ORTEC, model CA24-300-300). Figure 7 presents this spectrum, where three groups of α-particles, due to ^{238}U, ^{235}U and ^{234}U are clearly shown.

One can notice that the activity due to the ^{234}U is larger than that of the other isotopes, showing that its relative abundance increases during the process of uranium enrichment—as one would expect—and its anomalous abundance could be explained by assuming that the uranium was obtained from recycled fuel elements. An isotopic analysis of the sample was carried out at IPEN (Instituto de Pesquisas Energéticas e Nucleares of the Brazilian National Nuclear Energy Commission), which showed the presence of ^{236}U in the sample, as well. Since this isotope emits α-particles of an energy very close to those of the ^{235}U they were not observed by the surface barrier detector and its contribution was masked by that of the ^{235}U. This problem, introduced by the source, was solved through a calculation of the ^{236}U in our source, taking into account the mass of the source, the isotopic abundance and the disintegration constant of the ^{236}U (this contribution was found to be 1% of the total

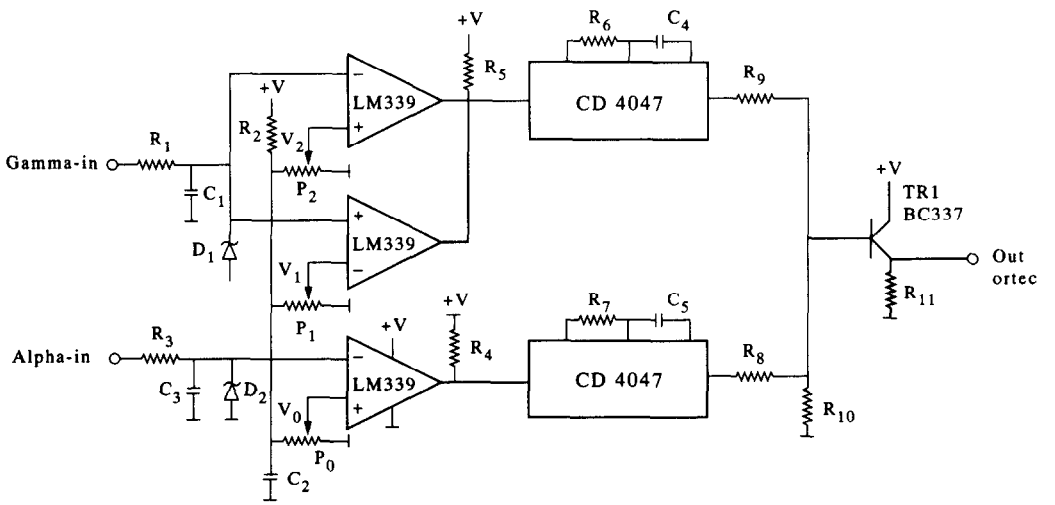

Fig. 5. Sum-coincidence circuit.

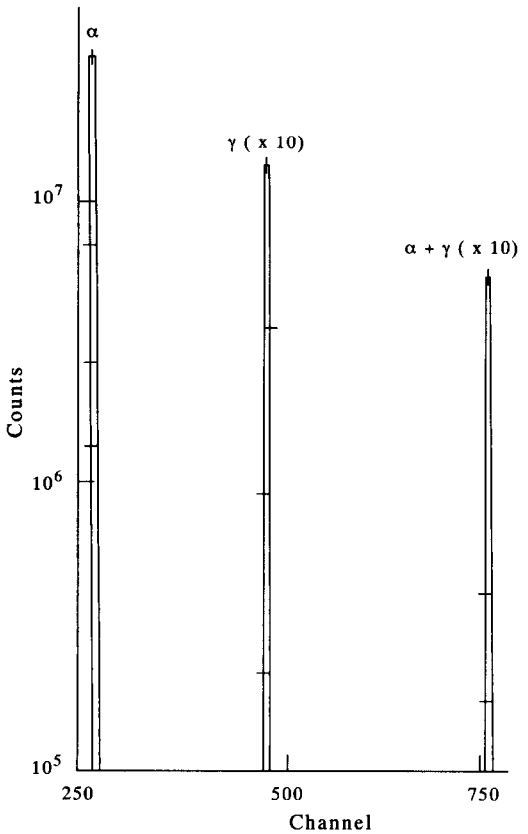

Fig. 6. Alpha–gamma sum-coincidence spectrum.

α-particles from the enriched U_3O_8 source). Through a fitting of the three peaks of the α-particles, corresponding to the ^{234}U, ^{235}U, ^{236}U and ^{238}U, the contribution of the α-activity due to the ^{235}U present in the sample was found to be $(5.20 \pm 0.01)\%$.

2. The sum of the γ-rays in the NaI(Tl) detector

The analysis of the disintegration scheme of the ^{235}U shows the presence of γ-rays whose energy are either smaller or greater than those connected with the coincidence measurements. It is necessary, therefore, to calculate the probability that a sum of γ-rays, of an energy smaller than those chosen, might lay in the chosen detection range and might contribute to the counting rate; on the other hand, the sum of γ-rays situated in the range of interest, would lead to pulses laying outside of the selected region of interest and would give rise to a decrease of the true counting rate.

These corrections were evaluated considering the angular distribution described by a normalized function and taking into account the intrinsic efficiency of the NaI(Tl) crystal for the γ-rays of interest and their disintegration constants. The obtained result for the sum of the γ-rays of smaller energy which lay within the region of interest for the detection increasing its counting rate, was found to be:

$$I_{sum} = (0.146 \pm 0.014)\%$$

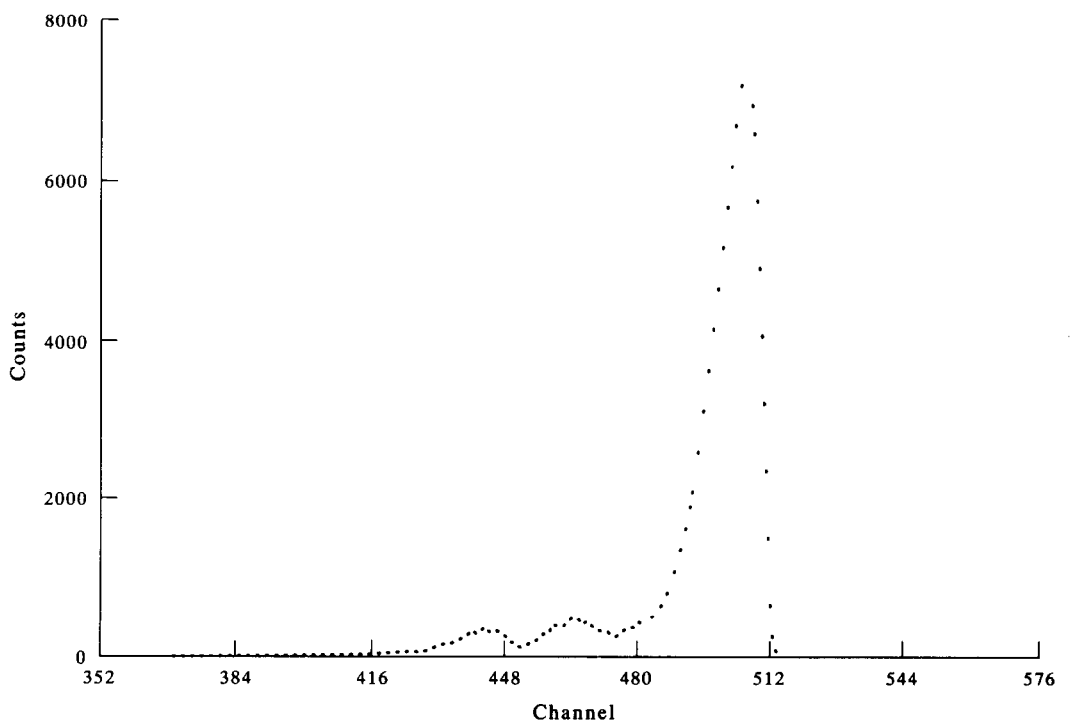

Fig. 7. Energy spectrum of the α-radiation from the U_3O_8 source obtained with a silicon surface barrier detector.

and the intensity of the sum of the γ-rays whose energy lies within the region of interest, which were not counted, was

$$I_{sum} = (1.61 \pm 0.16)\%.$$

Those results show the necessity for a correction of the individual γ counting rate, as well as for the total branching ratio of the γ-rays selected for the coincidences with the α-rays.

3. Compton scattering

Two different conditions were analyzed: the first deals with γ scattering in the brass cylinder and in the lead shield used in the counting system; the second problem concerns the continuous distribution of the γ-rays due to the Compton scattering, whose energies, lying in the region of interest of the coincidences, might give rise to unwanted coincidences.

In order to verify its influence, the γ-ray spectrum was measured with both the alpha and γ-ray counters facing each other (with the same geometry used in the measurements) under the lead shield. Another measurement was made without the lead shield and keeping the counters separated from each other, in order to avoid the eventual contribution of the scattering from the α-particles counter and its lead shield. In both cases, after correcting for the background, the same results were obtained showing that such an effect has a negligible influence on the measurements.

On the other hand, an analysis of the Compton scattering due to γ-rays whose energy is larger than those of the interest region has a negligible influence in the coincidence measurements. This can be explained by the low probability of deexcitation of the ^{231}Th nucleus through the emission of γ-rays of an energy larger than 205 keV (0.86% of the total intensity), as can be observed by its disintegration scheme (Schmorak, 1983).

4. Efficiency of the scintillators for the detection of radiations different from those for which they were built

We have pointed out that one of the conditions for a successful use of the coincidence method lays on the use of adequate and selective detectors for the radiations involved. In our work, the γ detector utilized, a NaI(Tl) scintillator was found to be insensitive to the α-radiation from the U_3O_8 source—as one would expect from the thickness of the aluminum layer used to shield it from light, as well as to the added absorption of such particles from the source due to its stainless steel backing.

The insensitivity of the gaseous scintillator for γ- and β-rays was checked by interposing a 63.0 mg/cm^2 aluminum absorbing layer for the total absorption of the α-rays. No pulses of a significant amplitude (above the background noise) were observed.

5. Random coincidences

The resolving time of the coincidence circuit $(0.931 \pm 0.002)\ \mu$s was determined experimentally by the usual method based on the use of two independent radioactive sources (^{137}Cs and ^{241}Am). The random coincidences per second (N_c) were found to be $N_c = (2.121 \pm 0.005) \times 10^{-3}$. This value corresponds to 0.1% of the number of genuine coincidences and can be considered negligible in our measurements, whose precision is about 0.2%.

6. Measurement of the source mass and self-absorption problems

In order to optimize the ratio between the number of the genuine and random coincidences, a source of low activity was used. Its thickness, smaller than 1 mg/cm^2, is sufficiently low to assure negligible self-absorption. On the other hand, since the source is spread on a small area, it is necessary that the efficiency of the radiation detection (α or γ) be the same in all its points. Such a condition is fulfilled for the γ-radiation since its self-absorption can be neglected.

7. Stability of the measuring system

The mains and the DC power supplies were stabilized: variations of 10.0% of the AC line produced no measurable effect on the counting rates. In the preamplifiers and in the main amplifiers the voltage stabilization circuits assured that their stability was better than 0.01% per volt of fluctuation of the mains supply. The high voltage supply was provided with a stabilizer that assured a stability better than 0.0007% of the applied voltage. Since the data was registered in a ORTEC multi channel analyzer, any interruption of the mains supply would interrupt automatically the accumulation of data without a loss of the pre-recorded information which was accumulated in its magnetic memory.

An additional check of the stability of the α- and γ-lines, that is, the stability of whole system was obtained through an analysis of the energy spectrum from these radiations during long periods. The reproduction of the characteristics, energy resolution and channel number, corresponding to the maximum counting rate of the spectra obtained in long periods constitutes an assurance of the excellent stability of the whole counting system during our measurements.

Calculation of the Absolute Activity of the ^{235}U

The absolute activity due to the ^{235}U, contained in the U_3O_8 sample is given by:

$$-\frac{dN}{dt} = \frac{N_\alpha N_\gamma}{N_{\alpha-\gamma}}. \tag{5}$$

In our measurements the window of the γ-channel was adjusted to measure the γ-rays from the ^{231}Th in

coincidence with the α-particles from ^{235}U; the corresponding branching ratio was 80.78% (Schmorak, 1983). In this way only 80.78% of the α-particles from the ^{235}U are in genuine coincidence with the selected γ-rays. However there are γ-rays (1.61%) whose energies lay within the selected γ-energy window which can give rise to sum pulses which are not counted because those pulses are on the outside of the selected window. In this way the correction factor for the α-particles which are in coincidence will be (80.78–1.61)%.

On the other hand, sum of the detected γ-pulses (0.146%) are due to sums of γ-rays of smaller energies which when added could lie within the counting window giving rise to extra pulses. The correction factor for those pulses will be (100–0.146)%.

It follows unambiguously that the activity of the source is given by:

$$-\frac{dN}{dt} = \frac{0.99854 \cdot (9.030 \pm 0.005) \cdot 0.052 \cdot 0.7917 \cdot (174.79 \pm 0.02)}{(2.421 \pm 0.002)} - \frac{dN}{dt} = (26.80 \pm 0.04)\ \text{Bq} \qquad (6)$$

which represents the acitivity of the ^{235}U.

In order to check the precision of our measurements, the half-life of ^{235}U was calculated, taking into account the number of atoms of the ^{235}U present in the source of U_3O_8 (2.000 $\pm$ 0.004) mg. The obtained result was

$$T_{1/2} = (7.04 \pm 0.01) \times 10^8\ \text{y}.$$

This value is better than that obtained by several previous measurements (see Table 1) and agrees, within the experimental error, with the most accurate measurement of the ^{235}U half-life $(7.04 \pm 0.005) \times 10^8$ y due to Jaffey *et al.* (1971).

Conclusion

(1) The measurement made with the sum-coincidence technique can be used for the measurement of the half-life of the ^{235}U with a precision comparable with the best data available in the literature.

(2) The excellent agreement with the data shows that the scintillation proportional counter presents a high level of long term stability, comparable to the most sophisticated equipment used in the standardization of radionuclide absolute activities; its use is recommendend due to the simplicity of its construction and operation.

(3) The sum-coincidence circuit, designed for our experiments allows a simultaneous determination of all parameters involved in the measurements; it presents definite advantages for measurements with a high degree of accuracy for providing an independent check of all the essential systems whose data is used for the final computation of the results.

(4) The enriched uranium sample used in our experiments was obtained from recycled fuel-elements (with 0.121% of ^{236}U). If the experiment were carried out with a sample of enriched natural uranium, the ^{236}U isotope would not be present and the measurement would be much easier.

(5) Due to the high accuracy of the obtained results, one can conclude that the experimental system used can be used with advantage for the determination of the half-lives of α–γ emitters, due to the excellent stability conditions of the counters and of the electronic circuits used, which were, in its great majority, designed and built in our laboratory.

Table 1. Summary of the experimental determination of the ^{235}U half-lives

Method	$t_{1/2}(10^8$ y)	Reference
Mass spectrometry	7.13 ± 0.15	Nier (1939)
Ionization chamber	7.07 ± 0.33	Sayag (1951)
Specific activity	7.13 ± 0.17	Fleming *et al.* (1952)
Ionization chamber	6.84 ± 0.11	Würger and Meyer (1957)
Si detector	6.92 ± 0.09	Deruytter *et al.* (1965)
Specific activity	7.13 ± 0.09	White *et al.* (1965)
Specific activity	7.04 ± 0.005	Jaffey *et al.* (1971)
Surface barrier det.	6.85 ± 0.09	Deruytter *et al.* (1965)
Absolute activity	7.04 ± 0.01	This work

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