STANDARDIZATION OF RADIOACTIVE SOURCES[1,2]

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INTRODUCTION

Primary standards of radioactivity are in some ways, analogous to fixed points in the temperature scale in that each can be used for comparison with "unknowns" not too far removed from the standard. Over a thousand radioactive species are known to exist, but only a comparatively few satisfy such basic requirements for primary standards as: known decay scheme; appropriate half life and disintegration energy; radiochemical and radioisotopic purity; and ease of preparation and availability.

Whereas it had originally been considered that a relatively few standards covering the range of β- and γ-energies of interest would suffice, the thought has been recently expressed that standards may be required for many additional radionuclides. For example, discrepancies in measurements using end-window counters may arise in extrapolating to "zero total absorber" because of the difference in the energy distribution of the β-ray spectra of two isotopes that have approximately the same maximum β-energy. Similarly, although a given β-γ-standard may be suitable for determining quantitatively the activity of a second radionuclide by comparison of respective beta activities, it may be totally inapplicable, for example, for use in corresponding measurements involving comparative γ-ray counting.

Where chemical methods are involved in the assay, interference has been found to occur from analogous compounds of the same element (e.g., $Na_2HP^{32}O_4$ and $Na_2HP^{32}O_3$), as well as from different radio-species of the same element in a given compound (e.g., $Na_2HP^{32}O_4$ and $Na_2HP^{33}O_4$).

Radium was formerly unique in being the only radionuclide for which the establishment of radiochemical purity was equivalent to primary standardization. The curie was officially redefined, however, as reported by Evans (1b) and Paneth (2) at the July, 1950, meeting of the Joint Commission on Standards, Units, and Constants of Radioactivity as "the quantity of any radioactive nuclide in which the number of disintegrations per second is 3.700×10^{10}." This new definition divorces the curie unit from a special association with radon and radium. The use of the term "rutherford" to denote 10^6 disintegrations per second was abandoned in 1950.

The progress in the acceptance of definitions for radioactivity units and standards has been reported by Evans (1a, 1b). The present survey of the problems involved in the preparation and certification of primary standards

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[1] The survey of the literature pertaining to this review was concluded on March 1, 1954.

[2] The following abbreviations are used: MIT for Massachusetts Institute of Technology; NBS for National Bureau of Standards; ORNL for Oak Ridge National Laboratory; AECL for Atomic Energy of Canada, Limited.

of radioactivity indicates major progress in the determination of the absolute disintegration rates of β- and of β-γ-emitters. The situation is much improved over the period 1947–49, and the availability of standards within the United States such as C^{14}, Co^{60}, I^{131}, P^{32}, Ra^{226}, Pb^{210} (Radium D-E), Sr^{90} and Tl^{204} is one measure of the advances made.

Assembly by the National Bureau of Standards (3a) of published information on the decay scheme, half life and energy distribution of many radionuclides has served as a spur for additional research in these fields. Prompt publication of such summaries is now a routine matter (3b), and some of the information is available on punched cards (4).

ADVANCE IN TECHNIQUES OF MEASUREMENTS

Determination of the Half Life of Radionuclides

The determination of the half life of a nuclide by any method at constant, but unknown, efficiency is essentially a straightforward procedure. If R_1 and R_2 are the observed counting rates obtained with a given detector during a time interval, t, the half life, $T_{\frac{1}{2}}$ can be calculated:

$$T_{\frac{1}{2}} = 0.693t/[2.3 \log_{10} (R_1/R_2)] \qquad 1.$$

The method is best adapted to those nuclides for which the initial and final measurements can be obtained in a reasonable length of time.

Half lives have been measured to four significant figures by use of a differential ionization chamber and a vibrating reed amplifier with feedback (5, 6). With this device, it has been possible to obtain significant measurements over a period of 12 half lives with some short-lived radionuclides. An automatic recording electroscope has been constructed (7) to determine half lives of the order of a few minutes with an accuracy of ±0.02 min. If a nuclide has a long life (such as C^{14}), the half lives can be calulated from the expression

$$T_{\frac{1}{2}} = 0.693N/(-dN/dt), \qquad 2.$$

where N is the total number of radioactive atoms present (determined by mass-spectrographic analysis and the weight of the sample present) and $-dN/dt$ is the measured rate of disintegration. Calorimetric methods have been used to determine directly the half life of radionuclides such as P^{32} (8).

Determination of the Rate of Disintegration

Measurements of the rate of disintegration can be made by: (*a*) coincidence counting (either β-γ or γ-γ); (*b*) Geiger-Mueller (G-M) or other type counters[3] under conditions of known geometry; and (*c*) calorimetry. Coincidence counting (11 to 15) involves the use of two counters: one for detecting

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[3] The development of scintillation counters and the advantages thereto have been reported in several symposia (9) as well as in the present volume, page 111. Halogen-quenched G-M counters have been developed (10) which are said to have a very long operating life and which use a visibly transparent, nonmetallic electrically conducting film as a cathode.

the β- (or γ-) radiation, and the other for detecting the (second) γ-radiation. Certain conditions must be fulfilled and various small corrections to the observed counting rates must be applied. If N_1 and N_2 are the counting rates of the β (or γ) and of the γ-counters, respectively, and N_{12} is the coincidence rate, the absolute rate of disintegration, $-dN/dt$, of the sample is given by

$$-dN/dt = N_1N_2/N_{12}. \qquad 3.$$

This method can be applied to the determination of the activity of long-lived as well as moderately short-lived nuclides.

The technique of "known geometry" with end-window counting, for example, involves measuring the rate of decay of a sample under conditions in which the counter "sees" a known solid angle at a known distance from the source. In principle the method is simple but corrections must be applied (16) for resolution or dead time losses; for backscattering from the support; for sidescattering from the walls of the shield enclosing the sample, the support and the counter; for absorption by the window of the counter, etc.

The method of end-window counting is applicable preferably to β-emitters with moderate or long half lives. A variation of the method of "known geometry" is that in which the sample to be measured is completely inside the counter or may even be part of the filling gas. This type of counter has been successfully used in measurements involving C^{14} and H^3.

The rate of charge loss has been measured in isolated systems containing a radionuclide by using a Lindemann electrometer and a compensating condenser (17). The method has been applied to P^{32} and Na^{22}.

Internal counters with 4π geometry suggested in 1947 by Borkowski (18) have been refined and used extensively (19a, 19b) for absolute β-measurements. Proportional 4π counters have been widely used in Canada (20), England (21, 22, 23) and in Germany (24, 25) for standardization of both high and low-energy β-emitters.

Panel discussions (16) on the techniques of absolute β-counting by the known geometry method have brought out a number of small, hitherto unsuspected sources of error regarding the effects of backscattering from supports, self-absorption, etc. New measurements have been made of the backscattering of positrons and electrons from various kinds of supporting materials (26, 27). The method of Marinelli (28) can be used for standardization if the decay scheme has been established. The rate of disintegration of a radionuclide can also be determined (29) if the average energy per disintegration, $\overline{E}$, is known.

Calorimetric methods for the determination of activity involve measurement of the rate of heat evolved, dQ/dt, by absorption of the energy emitted during the decay of the radionuclide. The method involves the usual thermal and other corrections applicable to calorimetry and is used preferably for β-emitters or for other isotopes where the radiation to be measured can be completely absorbed in the calorimeter. From the expression

$$dQ/dt = \overline{E}_\beta \, dN/dt \qquad 4.$$

either $-dN/dt$ or $\overline{E}_\beta$, the average energy per β-disintegration, can be computed if one of them is known.

The availability of intense, high specific-activity sources and the development of suitable calorimeters (8, 30) operating at liquid air temperatures has enabled estimation of the half lives of isotopes such as C^{14}.

The consensus presently seems to be (31c) that more reliable measurements can be made by internal 4π and by β-γ (or γ-γ) coincidence counting methods rather than by the method of known geometry. The method using the rate of charge loss (17) probably warrants further investigation.

DECAY SCHEMES

A clearer understanding of the techniques involved and of the errors inherent in coincidence counting (11, 12), the method of "delayed coincidence" counting (32), the development (33, 34) of amplifiers with very short resolving times (10^{-9} sec.), and the virtual elimination of spurious coincidences have permitted closer examination of many complex decay schemes.

A β-β (conversion electron) coincidence spectrometer has been devised (35) in which a Geiger-Mueller counter is placed at the "exit" end of the spectrometer and a scintillation counter is placed near the source. Coincidences are measured between the two counters corresponding to a selected electron "line" of known energy and all other β-radiation. The arrangement is said to be satisfactory for establishing decay schemes by β-β (conversion electron) coincidences.

A new method has been devised (36) for the absolute determination of β-ray energies in which a magnetic electron lens, working at unit modification, uses the image-rotation property to produce two lines by reversal of the current through the lens winding. The specific momentum, $H\rho$, of the focused electrons depends, among other factors, on the angle between the two lines.

Internal conversion coefficients have been measured by γ-x-ray coincidence counting (37, 38) as well as by the double-coil, thin lens, magnetic β-ray spectrometer (39). A revised table of K-shell internal-conversion coefficients is available (40).

SPECTROMETRY

Deutsch (41) has made a critical survey of the techniques of β- and γ-spectroscopy. One paragraph in particular is worth quoting:

> The aim of very high resolution spectrometry in the establishment of decay schemes is twofold: it permits the detection of lines which could otherwise not be resolved either from the continuum or from other neighbouring lines. In some of the more complicated decays the number of identified lines is almost proportional to the resolution used. In addition the use of precision energy measurements permits the application of the combination principle to the location of levels. For example in the decay of I^{131} there are six known γ-ray energies. If these are determined with an uncertainty of 1.5 per cent there appear seven combinations which might be interpreted as alternate decays involving the same initial and final states. With an accuracy of 0.5 per cent four of these remain. In fact, coincidence measurements have shown that only one of these is real, the other accidental.

An automatic count and control mechanism has been devised (42) which stepwise sweeps the β-spectrum automatically and counts the number of impulses received as a function of the focusing magnetic current. The device is said to duplicate with precision the manual control of a β-ray spectrometer.

Crystal spectrometers (43) have been developed for the determination of γ-ray energies to four or more significant figures. Scintillation spectrometers and counters for the measurement of the energy distribution and the half life (1 to 30 sec.) of β-ray emitters have been described (44).

A new type of scintillation spectrometer has been devised (45) in which incident photons are detected in a primary detecting NaI(Tl) crystal; the radiations not completely absorbed in the primary crystal are absorbed in a surrounding cluster of NaI(Tl) crystals which act as an anticoincidence circuit. The arrangement is said to yield the γ-ray spectrum directly without any interpretation of results which might become laborious, particularly if the incident γ-radiation has a complex spectrum. The apparatus has been used to study Cs^{137}, Na^{22}, and other radionuclides.

Pair spectrometers have been constructed (46, 47) in which rotation of the image in a short magnetic lens is used to separate negatrons and positrons by putting the pair converter a little to one side of the optical axis of the lens, from which it is possible to obtain coincidence "lines." With this apparatus the 2.62 Mev γ-ray of ThC″ (Tl^{208}) and the 2.76 Mev γ-ray of Na^{24} have been studied. A recent study has been made of the shapes of β-spectra (48), and tables for the analysis of β-spectra are available (49).

Mass Absorption Coefficients

New measurements have been reported for γ-ray absorption coefficients (50 to 53) over a range of energies from 0.1 to 6 Mev (Co^{60}, I^{131}, and others) in absorbers of atomic number from 13 to 82. The calculated values are in good agreement with those measured experimentally. This information is useful in correcting for the attenuation of the γ-radiation passing through the walls of the G-M counter or ion chamber.

Status of Individual Radionuclides

For the purpose of this presentation, the status of individual radionuclides is divided into three categories: (*a*) nuclides for which standards in the United States are available or are in preparation; (*b*) nuclides for which no standards are available, but for which considerable information on nuclear properties exists; and (*c*) nuclides for which standards would be desirable but for which considerably more information of various kinds is needed.

In each category, the information listed includes (when available): the method of production; method of obtaining radiochemical purity; the half life; the decay scheme; intercomparison of the activity between different laboratories; and the availability of standard samples. The nuclides are listed alphabetically within each category.

AVAILABLE STANDARDS

Summaries are presented for the following radionuclides: C^{14}, Co^{60}, I^{131}, P^{32}, Ra^{226}, Pb-Bi^{210} (Radium D-E), Sr^{90}, and Tl^{204}.

Carbon-14—Most of the C^{14} produced in the United States is now obtained by neutron irradiation of beryllium nitride, Be_3N_2. The C^{14} is separated as active CO_2 and precipitated as $BaCO_3$. Specific activities of 250 to 1500 mc./gm. of $BaCO_3$ are routinely available (54).

Labeled organic compounds are not suitable as standards for C^{14}. Skraba (55) has found that $C^{14}H_3OH$, for example, decomposes under self-irradiation to methane, ethylene glycol, glycerol, and other compounds to an extent sufficient to render it useless for primary standardization.

Full agreement has not yet been reached for the value of the half life of C^{14}. Because of the long half life of this radionuclide a direct measurement is not possible, and values ranging from a high of 7200 to a low of 4700 years have been obtained by indirect methods. Two general methods have been used: (*a*) end-window counting of solid barium carbonate; and (*b*) counting of gaseous C^{14} either as carbon dioxide or as methane in which the radioactive carbon forms a portion of the filling gas of the counter. Generally speaking, gas-phase counting has proven to be more reliable than the counting of solid barium carbonate.

Anderson (56) reviewed the techniques for detecting and measuring C^{14}, and Broda and co-workers (57) claim to have found it possible to measure as little as 2×10^{-12} curies (4 disintegrations/min.) of C^{14} using CO_2-CS_2 filled counters.

Hawkings *et al.* (58) reported a half life of 6360 ±200 years using a procedure involving a mass-spectrographic analysis of the active CO_2 and a pair of compensated CO_2-CS_2 filled (59, 60) internal gas counters of varying diameters. Jones (61), using alcohol-argon as a filling gas in an internal G-M counter, obtained 5589 ±75 yr. for the half life of C^{14}. Libby and co-workers (62, 63) also used alcohol-argon as the filling gas and employed counters of various ratios of length to diameter. The value for the half life first reported, 5720 ±20 years (62), was lowered to 5580 ±45 years (63) after further corrections were made for the end- and the wall-effects of the counters. It was assumed that the counting efficiency was 100 per cent.

In 1949 the National Bureau of Standards undertook a cooperative venture to determine the half life of C^{14} and to prepare standard samples of C^{14} for distribution. A sample of specially purified $BaCO_3$ treated to remove silica and other inert impurities was used as the starting point. Details of the NBS measurements have been reported by Manov & Curtiss (64).

The CO_2 from a portion of this active barium carbonate was generated by heating an intimate mixture of $BaCO_3$ with $PbCl_2$ at first to 500° C. and then to 1100° C. The evolved CO_2 was mixed thoroughly by repeated condensation with liquid air and evaporation into a small, closed flask and allowed to stand for several hours. Samples of this CO_2 were sent to several cooperating laboratories for mass spectrographic analysis.

An accurately weighed portion of the same active barium carbonate was mixed with a known weight of inert barium carbonate, the CO_2 evolved with perchloric acid and absorbed in an excess of sodium-hydroxide—sodium carbonate solution. The solution was diluted to a known volume, and samples were distributed to the participating laboratories for counting.

It is to be noted that the problem of determining the half life of C^{14} had been divided into two independent parts: (*a*) a collaborative determination of the isotopic ratio from which N in equation 2 could be calculated; and (*b*) a collaborative determination of $-dN/dt$. The results were reported in February, 1950 (31a, 31b) and later (65) in more detail.

In each case, gas-phase counting was performed with a compensated pair of CO_2-CS_2 filled counters. It is known (59, 60) that the resolving time of the CO_2-CS_2 counters is of the order of 1000 μsec., and at counting rates sufficiently high (50 to 100 counts/sec.) to obtain results of statistical significance in a reasonable length of time, the resolution-time losses are of the order of several per cent. (64)

The counting rates obtained by Chalk River and by MIT[2] were, at the time, provisional values. As an interim value pending further work, it was therefore suggested (31a) that the average isotopic ratio of the active barium carbonate used in the cooperative investigation be considered 3.88 per cent, the activity of the ampoules be tentatively assigned 1280 disintegrations/sec./ml., and the half life of C^{14} be estimated at 5400 years, with an uncertainty of perhaps ± 200 years.

Subsequent to this suggestion, two additional values for the half life using other samples of $BaCO_3$ have been reported by Bernstein & Ballentine (66a) in which it appears that gas-phase counting in the proportional region and in the Geiger-Mueller (G-M) region yield 5513 and 6360 years respectively. This discrepancy appears to have been partially resolved in a later paper (66b) in which the hypothesis was advanced that "over-quenching" of the G-M counter may have occurred which led to a significant loss (13.9 per cent) in the counting rate. Provided this observation and the explanation are correct, it may be appropriate to reduce the value for the half life obtained (66a) in the G-M region to 6360/1.139 or 5580 years. Gas-phase counting would therefore seem to lead to estimates of 5513 and 5580 years for the two sets of measurements. The agreement, however, may be fortuitous.

Evidently the last word on the half life of C^{14} has not yet been said and more experimental work needs be done. There seem to be one set of measurements in the range 5400 to 5600 years and another 6000 to 6200 years.

The maximum energy for the β-spectrum of C^{14} is 155 ±1 kev (67), and the decay scheme involves only a simple β-emission. Standard samples of C^{14} are available from the National Bureau of Standards.

Cobalt-60.—Co^{60} is generally prepared by neutron absorption in Co^{59}; sample preparation consists of solution in hydrochloric or other acids (with or without added cobalt carrier) and dilution to the appropriate activity.

The value for the half life of Co^{60} is approaching agreement. Recent determinations yield 5.26 ±0.17, 5.27 ±0.07, 5.25 ±0.21, and 5.21 ±0.04 years (6, 68, 69, 70), with an average of 5.25 years.[4]

The decay scheme involves a simple β-decay with $E_{\max}{}^{98} = 0.306 \pm 0.005$ Mev (72), followed by two γ-rays in cascade whose energies (43) are 1.1715 ±0.0010 and 1.331 ±0.0010 Mev, respectively.

The Bureau of Standards has prepared a set of ampoules containing Co^{60} which are dilute with respect to total solids (i.e., approximately 1 μg./ml.) and has measured the activity of these ampoules by β-γ-coincidence, 4π, and other counting methods. Preliminary results were reported by Mann & Seliger (27) and by Putnam (21). The former (27) studied two variations of the 4π β-counter (conducting film and mirror image) and two coincidence-counting methods (β-γ and γ-γ). On the same sample of Co^{60}, the relative results obtained (assigning arbitrarily a value of unity to the first method) were 1.000, 0.996, 1.003, and 1.006.

Iodine-131.—I^{131} is presently separated from fission products by oxidation to elemental iodine and volatilization from an aqueous solution. The radionuclide is distributed in the form of a weakly basic iodide solution containing a trace of $NaHSO_3$ (54).

The half life has been reported by Sreb (73) to be 8.1409 ±0.0062 days; 8.04 ±0.04 days by Sinclair & Halloway (69); 8.06 ±0.02 days by Lockett & Thomas (71); 8.075 ±0.022 days (measured over five half lives), by Seliger, Cavallo & Culpepper (74); and 8.05 ±0.01 days by Hawkings and his co-workers (75). The average value, neglecting (73) is 8.06 days.

Decay schemes for I^{131} have recently been reported by Bell, Cassidy & Kelley (76) and Cork and co-workers (77). Both schemes recognize the existence of the 12 day metastable state of Xe^{131} reported by Zeldes, Brosi & Ketelle (78), but associate it with different β-energies. Additional complexities in the decay scheme have since been discovered. Verster (79), Ketelle (80), Cavanagh (81), and Bell (82) and Kurbatov (83) have confirmed the existence of two additional β-energies, 250 kev (2 per cent), and 807 kev (1 per cent). Rose (84) has measured internal conversion electrons but apparently did not observe the 250 kev β-energy. Bell (85) has recently found a previously unknown γ-energy of about 0.5 Mev that does not fit into the presently accepted decay schemes. Caswell (29) has found the average energy of the β-radiation to be 0.189 ±0.008 Mev.

A number of methods are in use for the standardization of I^{131}; known geometry, coincidence counting, 4π counting, and ionization methods prevail (16). The status of the intercomparison of iodine standards is much more promising than it was six years ago.

The National Bureau of Standards since 1950 has been furnishing on a regular schedule 3-ml. ampoules of I^{131} (and of P^{32}) with a known activity (±3 per cent) in terms of disintegrations per second per milliliter of solution. The activity is of the order of 100,000 disintegrations/sec./ml., with higher

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[4] It is understood that the value of 4.95 ±0.04 years (71) has been withdrawn (31c).

activities occasionally available by special arrangement. Provision is made for pH control, the samples are low in total solids (approximately 10 μg./ml.), and instructions for use accompany each sample. 4π proportional counting is used for standardization (19a). The presence of the metastable, 12 day Xe^{131} introduces an error which can be neglected in end-window β-counting or in ionization chamber measurements where an overall accuracy of ±5 per cent is sufficient. Approximately 200 ampoules are distributed at six-month intervals to users within the United States, Australia, Canada and Great Britain, and frequent inter-checks between NBS,[2] ORNL,[2] and other standardizing laboratories serve to maintain the requisite accuracy in standardization.

An international comparison tabulated by Seliger (19b) for samples distributed by the NBS shows the following relative values (the NBS value is arbitrarily designated as unity): NBS, United States, $4\pi\beta$ (1.000); AECL,[2] Canada, 4π (1.022); and two values from Hammersmith Hospital, England, absolute γ-ion chamber (0.997) and 4π G-M (1.030).

Samples of I^{131} are being exchanged in an attempt to establish an international standard. The present agreement appears to be of the order of ±2 per cent.

Phosphorus-32.—Neutron irradiation of sulfur, followed by chemical oxidation and separation, is used to obtain P^{32} (54). If sulfur-containing compounds are irradiated to form phosphorus, the chemistry of separation is rather involved because of the different radiochemical forms in which the phosphorus may be found. For example, if calcium sulfate is irradiated, metaphosphates (PO_3^-), ortho-phosphates (PO_4^{---}), pyro-phosphates ($P_2O_7^{----}$) and phosphites (HPO_3^{--}) are formed. Failure to recognize the chemistry involved may lead to abnormal tracer behavior (69, 86).

New measurements have been reported for the half life of P^{32}. Using a calorimetric method, Bayly (8) obtained 14.30 ± 0.05 days; Lockett & Thomas (71) used a quartz fibre electroscope and obtained 14.50 ± 0.04 days over a period of five half lives. Neither worker, however, was aware of the possible presence of P^{33} (see below). The maximum β-energy for P^{32} has been found to be 1.704 ± 0.008 Mev (87a).

The existence of P^{33} in reactor-produced P^{32} has been reported by Jensen (87a, 88) and confirmed by Sheline (89) and others. The half life is 2.44 ± 0.2 days, and the maximum β-energy is 0.249 ± 0.002 (87b).

Collaborative determinations of the absolute strength of P^{32} samples have been carried out for a number of years between several standardizing laboratories, and the results have recently been summarized by Seliger (19b). The laboratories reporting, the methods used and the relative values obtained (19b) are as follows: United States NBS, 4π β-counter (1.000); ORNL, defined solid angle (0.986); Columbia University, parallel plate counter (1.006); AECL-Canada, 4π β-counter (1.002); Hammersmith Hospital, England, absolute β-ion chamber (0.971).

Ampoules containing known amounts of P^{32} have been available since

1950 from the National Bureau of Standards. These ampoules contain approximately 100,000 disintegrations/sec./ml. of P^{32} with 0.001 M H_3PO_4 added as carrier. The effect of P^{33} on the measurements is considered to be negligible if the time interval between NBS standardization and use involves less than two half lives. 4π proportional counting is used (19b). The availability and the method of distribution[5] for P^{32} and I^{131} are identical.

Radium-226.—Radium standards have been available for many years, but minor uncertainties still persist in determining the activity of one gram of radium. Careful measurements of the specific activity of radium were made by Kohman (91) in 1947, yielding $(3.608 \pm 0.028) \times 10^{10}$ α-disintegrations per second per gram of radium, and a half life of 1620 ± 13 years. One curie (of radium or of any other nuclide) was redefined in 1950 to be equivalent to 3.700×10^{10} disintegrations per second (1b, 2).

Lead-Bismuth-210 (Ra D-E).—Pb^{210} (RaD) is obtained by chemical separation from the equilibrium radium mixture, and an organic reagent, dithizone, is useful (92) for this purpose. Electroplating under carefully controlled conditions (93) is also employed to effect the separation. Bi^{210} (RaE) is obtained as the daughter product of Pb^{210}, and values of 5.02 ± 0.02 and 4.989 ± 0.013 days have been reported (71, 94) for the half life.

The original method for preparing standards of RaE was the subject of discussion between the National Bureau of Standards and the National Research Council, and the method decided upon at the time, early in 1946, was a compromise between (*a*) accurate standards desirable for the future, and (*b*) the immediate need for a fairly large number of reproducible standards with a somewhat lesser degree of accuracy. The NBS standards are composed of a circular deposit of active and inert PbO_2 weighing approximately one milligram; the material is deposited electrolytically in the center of a palladium-faced silver disk approximately one inch in diameter and 1/16-inch in thickness. Calibration was initially made by assaying accurately a radium ore for activity, extracting the total lead therefrom, and then electrolytically depositing a portion of the lead as lead peroxide. Several hundred such standards have been prepared and distributed.

In 1950, Burtt (95) and others found that there was a forward scattering of the β-particles from RaE because of the finite thickness of the PbO_2 deposit. The scattering amounted to approximately 7 per cent under the particular conditions of geometry, backscatter, sidescatter, etc., used by him. Seliger (26) has since made additional measurements of the β-particle backscattering from various sources.

These refinements in measurement point up a fact that has long been

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[5] Ten microcurie quantities of radionuclides with a half life of less than 30 days and 1 μc. quantities with a half life over 30 days may be interchanged between laboratories without prior approval by the U. S. Atomic Energy Commission (90). This action has served to stimulate intercomparisons not only of materials proposed as standards, but of various intermediate preparations. It applies, however, only to transfers within the United States.

recognized; i.e., that measurements made by comparison of the β-activities between a standard of a given radionuclide and the "unknown" should be made under identical conditions of geometry, thickness of deposit, backscattering, and that the energies of the standard and of the "unknown" and the shapes of their β-spectra should be as nearly alike as possible.

An independent calibration of the strength of the RaE sources can be obtained by measuring the growth of the α-activity from RaF. Such measurements have been made by Novey (96) and indicate that if proper corrections are applied, the agreement between β- and α-counting is within 2 per cent. National Bureau of Standards Pb^{210}-Bi^{210} standards are available in activities approximating 100, 200, 500, and 1000 disintegrations./sec., and have been widely used in this country and abroad. The international standardization of Pb^{210}-Bi^{210} has not yet been attempted.

Strontium-90.—Sr^{90}, obtained by separation from fission products, is a pure β-ray emitter which decays to Y^{90} and then to stable Zr^{90}. A value of 19.97 ±0.3 years (97) has been reported by Powers and Voigt for the half life, measurements being made over a period of seven years. The heretofore accepted figure is 25 years.

The maximum β-energies of Sr^{90} and Y^{90}, according to Cohn (98), are 0.525 and 2.24 Mev respectively; for Y^{90}, Moreau & Corba (99) found 2.270 ±0.02 Mev. Similar measurements have been made by Shepherd (100) on Y^{90}. The average β-energy of Y^{90} is 0.895 ±0.035 Mev (29).

The National Bureau of Standards has available for distribution standard samples of Sr^{90} in equilibrium with Y^{90}. The activity, approximately 10,000 disintegrations/sec./ml. of solution, is standardized by 2π β-counting, with an accuracy of ±1.5 per cent.

Thallium-204.—Tl^{204} is prepared by neutron irradiation of Tl^{203} and is made available as an aqueous solution of the nitrate. Various difficulties have been encountered in the preparation of samples with reproducible counting rates. Tl^{204} has chemical oxidation states of +1 and +3 in aqueous solution, and it has been reported by Peacock (31b) that electroplating is not satisfactory as a method of separation from other electrolytes. Thallous nitrate solutions in dilute nitric acid have been found by Reynolds (101) to be more stable than neutral or alkaline solutions of thallic ion or halide solutions of thallous ion.

Values for the maximum β-energy of Tl^{204} have been reported to be 0.765 ±0.010 Mev by Lidofsky, Macklin & Wu (102) and 0.760 ±0.01 Mev by der Mateosian & Smith (103). The Kurie plot is linear down to 0.150 Mev (102).

A value of 4.0 ±0.1 years for the half life of Tl^{204} has been obtained by Harbottle (104), and recently Lockett & Thomas (71) found 2.71 ±0.05 years using a quartz fibre electrometer with measurements extending over a period of eight months. Two older values (3) of 2.7 and 3.5 years have also been reported.

Reynolds (105) has recently published the results of a limited intercomparison (six laboratories) of a solution of Tl^{204} whose activity using different counting methods, was approximately 1×10^{-3} μc./ml. The results appear promising, with an average deviation of ±10 per cent. Further work is under way.

Standard samples of Tl^{204} are available from the National Bureau of Standards in the form of 3-ml. ampoules with an activity of approximately 10,000 disintegrations/sec./ml. Standardization is performed by 2π β-counting with an accuracy between +4 per cent and −2 per cent.

DESIRABLE STANDARDS; SOME NUCLEAR INFORMATION AVAILABLE

Summaries are presented for the following radionuclides: Au^{198}, Fe^{55}, Fe^{59}, Na^{24}, and H^3.

Gold-198.—The half life of Au^{198} has been found to be: 2.69 ±0.01 (5), 2.73 ±0.01 (69), 2.697 ±0.003 (71) and 2.74 ±0.04 (72) days. The decay scheme has been studied recently by a number of workers (72, 106 to 109). The major portion (98 per cent) of the gold decays by a simple β-emission for which values of 0.97 ±0.01 and 0.958 Mev have been found (42, 72). The single γ-ray energy has been measured yielding a value of 0.411770 ±0.000036 and 0.41173 ±0.00007 Mev (110, 111). The minor portion decays with a β-energy of 0.290 Mev and a γ-energy of 1.09 Mev. The Auger spectrum indicates that K capture to Pt^{198} cannot occur in more than 0.5 per cent of the disintegrations (112). The work of Brosi (107) confirms that of Cavanagh (106). It has been determined (34) that the half life of excited (411 kev) Hg^{198} resulting from the β-decay of Au^{198} is less than 3×10^{-11} sec. Hill & Mihelich (109) observed two weak γ-rays of 159 and 209 kev which have been attributed to Au^{199} formed by the successive reactions: Au^{197} (n, γ) Au^{198}; Au^{198} (n, γ) Au^{199}. The half life for Au^{199} was observed to be 3.3 days, the maximum β-energy is 0.32 Mev (3) and the calculated thermal neutron cross section of Au^{198} is estimated to be about 3.5×10^4 barns. The relative proportions of Au^{198}/Au^{199} were estimated[6] to be 95/1 for a sample irradiated for eight days in a flux of 10^{12} n/cm.²/sec. (109).

Standards for Au^{198} are needed. Presently, measurements of the strength of these sources are generally made by comparison with a radium needle using an ionization chamber counter or electroscope. ORNL assays Au^{198} routinely by means of a high pressure ionization chamber calibrated periodically by 4π coincidence counting of the same sample. Because of its comparatively short half life, it is doubtful that standard samples of Au^{198} can be distributed in the usual sense.

From the practical point of view, two courses of action are open: (*a*) standardization of a Au^{198} source by coincidence counting and simultaneous comparison with radium in an ionization chamber under rigidly specified conditions (the gold might then be certified in terms of radium-equivalents); (*b*) search for an isotope of reasonably long half life that has either the same

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[6] A private communication from H. H. Seliger of NBS indicates that for an infinitely long irradiation time, the ratio of Au^{198}/Au^{199} should be approximately 1000/1.

β- or γ-energy as Au^{198} (Tm^{170} has been suggested). Secondary standards might then be distributed for comparison of "unknowns" with either the β- or the γ-radiation, as appropriate.

Iron-55.—Fe^{55} is of interest because the decay is thought to proceed almost entirely by K-capture. Scintillation counting (113) has shown that the continuous radiation has an upper limit of 0.205 Mev, and a later value (38) of 0.206 ±0.020 Mev has been reported. A new value for the half life, 3.0 ±0.15 years has been found (114) which is in agreement with an earlier one of 2.94 years (68). Preliminary results on intercomparison of samples of Fe^{55} reported by Reynolds (101) indicate that an agreement of the order of ±15 per cent is possible.

Iron-59.—Fe^{59} is generally produced by cyclotron bombardment. Three values for the half life have been reported: 45.1 ±0.2, 47.1 ±0.5, and 45.0 ±0.2 days (6, 114, 115). The decay scheme has been studied by Rose and co-workers (116) and by Metzger (117).

Sodium-24.—Na^{24} is produced by neutron irradiation of stable sodium. The half life of Na^{24} has been reported by Sreb (118) to be 15.060 ±0.039 hr., by Lockett and Thomas (71) as 14.97 ±0.02 hr. and an earlier value of Wilson & Bishop (119) recalculated by Sreb to yield 14.96 ±0.10 hr. The three values agree within the limits of error.

Considerable interest has been manifested in the decay scheme of Na^{24} Turner & Cavanagh (120) have found evidence for a 4.17 Mev β-group and estimate the branching ratio to be 0.003 per cent. Siegbahn & Johansson (46) have used a new type of pair spectrometer in which rotation of the image in a short magnetic lens is used to separate negatrons from positrons by putting the pair-converter a little aside from the optical axis. The simultaneous emission of three particles (a decay β-ray and an electron-positron pair) from Na^{24} has been examined (121) and the value of the pair-conversion coefficient for the 2.78 Mev γ-ray has been determined to be $(7.6 \pm 1.9) \times 10^{-4}$. A pair conversion coefficient of $(8.25 \pm 1.05) \times 10^{-4}$ was obtained for positrons stopped in Be. Hedgran and Lind (122) have measured the energy of the γ-rays from Na^{24} and report 1.3680 ±0.0010 and 2.7535 ±0.0010 Mev.

Absolute measurements of the activity of Na^{24} have been performed by using cavity-type ionization chambers (22) and have been compared with the results of coincidence counting (21). It will be difficult to set up standards for such a short-lived material.

Tritium.—Two recent values for the half life of tritium yield 12.46 ±0.1 and 12.41 ±0.20 years (123, 124). Recent measurements of the maximum β-energy by Langer & Moffatt (125) yield 17.95 ±0.10 kev, while the average energy (123) is 5.69 ±0.04 kev. A bibliography of literature on tritium has been compiled (126).

The low-energy of the β-radiation from tritium makes quantitative measurements difficult or impractical by ordinary methods such as end-window counting and recourse must be had to gas-phase measurements (119, 127, 128). For samples which are essentially pure tritium, the method of gas-density (123) can be used and it is claimed that an accuracy of ±0.05 per cent can be attained within the range of 50–100 per cent T_2. A check on the composition of tritium-deuterium-hydrogen mixtures is obtained by mass-spectrographic analysis. (The tritium can be stored on spongy Zr or Pd, and released by gentle heating.)

For lower concentrations of tritium, internal gas-counting can be used. The counters are of brass with flat end-plates similar to those used by Libby (62) with alcohol-argon in the ratio of 1 to 9 at a total pressure of 10 cm. as the filling gas. The tritium is quantitatively diluted with ordinary hydrogen to give a reasonable counting rate in the G-M tube. A counting efficiency of 97 per cent is assumed, and the length of the plateau is approximately 200 volts with a 1000 volt threshold and a slope of approximately 3 per cent per 100 volts. Backgrounds of approximately 200 c/min. on a counter of $1\frac{1}{2}'' \times 12''$ are obtained. Unfortunately, some memory effects are associated with this type of counter, and the background has been found to increase approximately 200 counts/min. for each filling. The counters can, however, be cleaned thoroughly by rinsing with dilute aqueous ammonia and washing with distilled water. Robinson (127) has used a mixture of tritium and methane as a filling gas. The advantage claimed is that the resolving time is very short, that counting can proceed at a high rate of speed without undue coincidence losses, and that the memory effects are smaller.

It should also be possible to use a vibrating-reed electrometer and an ion chamber for routine measurements. Such a chamber could be calibrated at high concentrations of tritium by means of the gas-density balance and at low concentration by G-M gas-phase counting.

It was recently proposed (31b) that standard samples of tritium be prepared in the form of tritiated water with an approximate activity of 10^6 to 10^7 disintegrations/sec./ml. This work is now under way.

Desirable Standards; Little Nuclear Information Available

Summaries are presented for the following radionuclides: Cr^{51} and Zn^{65}.

Chromium-51.—A value of 27.75 ±0.3 days for the half life has been reported recently by Lyon (129) and the decay scheme which is 90 to 92 per cent K capture has been examined more thoroughly (129, 130). Reynolds (101) has measured the disintegration rate of the same sample of Cr^{51} (a) by x-γ-coincidence counting and (b) by x-ray counting with a proportional counter standardized by means of a source of Mn^{54} of known disintegration rate. Results by the two methods agreed within 3 per cent.

Zinc-65.—A new measurement (115) of the half life has yielded 245.0 ±0.8 days. A small percentage of the disintegrations are by positron emission, the main decay process being by K-capture. Furberg (131) has determined the branching ratio $K_2/(K_1+K_2)$ to be 0.44 ±0.03, where K_1 = number of capture processes leading to the ground state, and K_2 = number leading to the excited state. Major (132) has found 0.45 for the branching ratio, and Griffiths (133) by coincidence measurements found that one positron was emitted for every 65 ±5 γ-rays.

Mann and co-workers (134) measured the β-spectrum by means of a thin lens type magnetic spectrometer and found 0.325 ±0.002 Mev for the positron and 1.38 ±0.03 Mev for the γ-radiations.

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