Designation: D 3648 – ~~95~~03

Standard Practices for the Measurement of Radioactivity[1]

This standard is issued under the fixed designation D 3648; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These practices cover a review of the accepted counting practices currently used in radiochemical analyses. The practices are divided into four sections:

	Section
~~General Information~~	~~5 to 10~~
General Information	6 to 11
~~Alpha Counting~~	~~11 to 21~~
Alpha Counting	12 to 22
~~Beta Counting~~	~~22 to 32~~
Beta Counting	23 to 33
~~Gamma Counting~~	~~33 to 40~~
Gamma Counting	34 to 41

1.2 The general information sections contain information applicable to all types of radioactive measurements, while each of the other sections is specific for a particular type of radiation.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

~~[1] Precision and Bias These~~

[1] These practices are under the jurisdiction of ASTM Committee D-19 on Water and are the direct responsibility of D19.04 on Methods of Radiochemical Analysis .

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D 1066 Practice for Sampling Steam [2]
D 1129 Terminology Relating to Water [2]
D 1943 Test Method for Alpha Particle Radioactivity of Water [3]
D 2459 Test Method for Gamma Spectrometry of Water [4]
D 3084 Practice for Alpha Spectrometry of Water [3]
D 3085 Practice for Measurement of Low-Level Activity in Water [3]
D 3370 Practices for Sampling Water from Closed Conduits [2]
D 3649 Test Method for High-Resolution Gamma-Ray Spectrometry of Water [3]
E 380 Practice for Use of the International System of Units (SI) (the Modernized Metric System) [5]

2.2 *ANSI/ISO Standards:*[6]

ANSI N42.14 Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides
ISO Guide to the Expression of Uncertainty in Measurement, 1993

3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of terms used in these practices, refer to Terminology D 1129. For an explanation of the metric system, including units, symbols, and conversion factors, see Practice E 380.

4. Summary of Practices

4.1 The practices are a compilation of the various counting techniques employed in the measurement of radioactivity. The important variables that affect the accuracy or precision of counting data are presented. Because a wide variety of instruments and techniques are available for radiochemical laboratories, the types of instruments and techniques to be selected will be determined by the information desired. In a simple tracer application using a single radioactive isotope having favorable properties of high purity, energy, and ample activity, a simple detector will probably be sufficient and techniques may offer no problems other than those related to reproducibility. The other extreme would be a laboratory requiring quantitative identification of a variety of radionuclides, preparation of standards, or studies of the characteristic radiation from radionuclides. For the latter, a variety of specialized instruments are required. Most radiochemical laboratories require a level of information between these two extremes.

4.2 A basic requirement for accurate measurements is the use of accurate standards for instrument calibration. With the present availability of good standards, only the highly diverse radiochemistry laboratories require instrumentation suitable for producing their own radioactive standards. However, it is advisable to compare each new standard received against the previous standard.

4.3 Thus, the typical laboratory may be equipped with proportional or Geiger-Mueller counters for beta counting, sodium iodide or germanium detectors, or both, in conjunction with multichannel analyzers for gamma spectrometry, and scintillation counters suitable for alpha- or beta-emitting radionuclides.

5. Significance and Use

5.1 This practice was developed for the purpose of summarizing the various generic radiometric techniques, equipment, and practices that are used for the measurement of radioactivity.

GENERAL INFORMATION

6. Experimental Design

6.1 In order to properly design valid experimental procedures, careful consideration must be given to the following;
6.1.1 radionuclide to be determined,
6.1.2 relative activity levels of interferences,
6.1.3 type and energy of the radiation,
6.1.4 original sample matrix, and
6.1.5 required accuracy.
6.2 Having considered 6.1.1-6.1.5, it is now possible to make the following decisions:
6.2.1 chemical or physical form that the sample must be in for radioassay,
6.2.2 chemical purification steps,
6.2.3 type of detector required,

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.02.

[4] Discontinued—See 1987 *Annual Book of ASTM Standards* , Vol 12.01.

[5] *Annual Book of ASTM Standards*, Vol 14.02 (Excerpts in Vol 11.02).

~~[6] The boldface numbers in parentheses refer to the list of references appended to these practices.~~

[6] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

6.2.4 energy spectrometry, if required,

6.2.5 length of time the sample must be counted in order to obtain statistically valid data,

6.2.6 isotopic composition, if it must be determined, and

6.2.7 size of sample required.

6.3 For example, gamma-ray measurements can usually be performed with little or no sample preparation, whereas both alpha and beta counting will almost always require chemical processing. If low levels of radiation are to be determined, very large samples and complex counting equipment may be necessary.

6.3.1 More detailed discussions of the problems and interferences are included in the sections for each particular type of radiation to be measured.

7. Apparatus

7.1 *Location Requirements*:

7.1.1 The apparatus required for the measurement of radioactivity consists, in general, of the detector and associated electronic equipment. The latter usually includes a stable power supply, preamplifiers, a device to store or display the electrical pulses generated by the detector, or both, and one or more devices to record information.

7.1.2 Some detectors and high-gain amplifiers are temperature sensitive; therefore, changes in pulse amplitude can occur as room temperature varies. For this reason, it is necessary to provide temperature-controlled air conditioning in the counting room.

7.1.3 Instrumentation should never be located in a chemical laboratory where corrosive vapors will cause rapid deterioration and failure.

7.2 *Instrument Electrical Power Supply*—Detector and electronic responses are a function of the applied voltage; therefore, it is essential that only a very stable, low-noise electrical supply be used or that suitable stabilization be included in the system.

7.3 *Shielding*:

7.3.1 The purpose of shielding is to reduce the background count rate of a measurement system. Shielding reduces background by absorbing some of the components of cosmic radiation and some of the radiations emitted from material in the surroundings. Ideally, the material used for shielding should itself be free of any radioactive material that might contribute to the background. In practice, this is difficult to achieve as most construction materials contain at least some naturally radioactive ~~species~~ isotopes (such ~~as potassium-40,~~ as ^{40}K, members of the uranium and thorium series, etc.). The thickness of the shielding material should be such that it will absorb most of the soft components of cosmic radiation. This will reduce cosmic-ray background by approximately 25 %. Shielding of beta- or gamma-ray detectors with anticoincidence systems can further reduce the cosmic-ray or Compton scattering background for very low-level counting.

7.3.2 Detectors have a certain background counting rate from naturally occurring radionuclides and cosmic radiation from the surroundings; and from the radioactivity in the detector itself. The background counting rate will depend on the amounts of these types of radiation and on the sensitivity of the detector to the radiations.

7.3.3 In alpha counting, low backgrounds are readily achieved since the short range of alpha particles in most materials makes effective shielding easy. Furthermore, alpha detectors are quite insensitive to the electromagnetic components of cosmic and other environmental radiation.

7.4 *Care of Instruments*:

7.4.1 The requirements for and advantages of operating all counting equipment under conditions as constant and reproducible as possible have been pointed out earlier in this section. The same philosophy suggests the desirability of leaving all counting equipment constantly powered. This implies leaving the line voltage on the electrical components at all times. The advantage to be gained by this practice is the elimination of the start-up surge voltage, which causes rapid aging, and the instability that occurs during the time the instrument is coming up to normal temperature.

7.4.2 A regularly scheduled and implemented program of maintenance is helpful in obtaining satisfactory results. The maintenance program should include not only checking the necessary operating conditions and characteristics of the components, but also regular cleaning of the equipment.

7.5 *Sample and Detector Holders* —In order to quantify counting data, it is necessary that all samples be presented to the detector in the same "geometry." This means that the samples and standards should be prepared for counting in the same way so that the distance between the source and the detector remains as constant as possible. In practice, this usually means that the detector and the sample are in a fixed position. Another configuration often used is to have the detector in a fixed position within the shield, and beneath it a shelf-like arrangement for the reproducible positioning of the sample at several distances from the detector.

7.6 *Special Instrumentation*—This section covers some radiation detection instruments and auxiliary equipment that may be required for special application in the measurement of radioactivity in water.

7.6.1 4-π *Counter:*

7.6.1.1 The 4-π counter is a detector designed for the measurement of the absolute disintegration rate of a radioactive source by counting the source under conditions that approach a geometry of 4-π steradians. Its most prevalent use is for the absolute

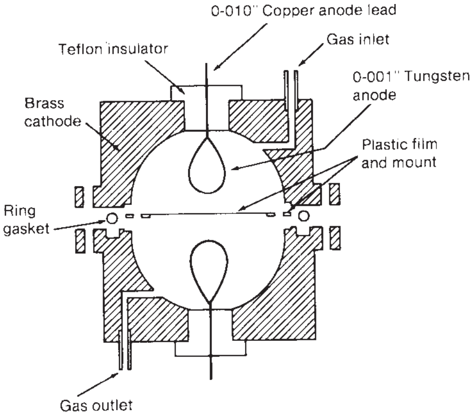

FIG. 1 The 4π-Counting Chamber

measurement of beta emitters **(1, 2)**.[7] For this purpose, a gas-flow proportional counter similar to that in Fig. 1 is common. It consists of two hemispherical or cylindrical chambers whose walls form the cathode, and a looped wire anode in each chamber. The source is mounted on a thin supporting film between the two halves, and the counts recorded in each half are summed. A 10 % methane-90 % argon ~~(90 %)-methane (10 %)~~ gas mixture can be used; however, pure methane gives flatter and longer plateaus and is preferred for the most accurate work. The disadvantage is that considerably higher voltages, about 3000 V, rather than the 2000 V suitable for ~~argon-methane,~~ methane-argon, are necessary. As with all gas-filled proportional counters, very pure gas is necessary for very high detector efficiency. The absence of electronegative gases that attach electrons is particularly important since the negative pulse due to electrons is counted in this detector. Commercial chemically pure (cp) gases are ordinarily satisfactory, but they should be dried for best results. A high-voltage power supply for the detector, an amplifier, discriminator, and a scaler complete the system.

7.6.1.2 To convert counting rate to disintegration rate, the principal corrections required are for self-absorption in the source and for absorption in the support film. The support film should be as thin as practicable to minimize absorption of beta particles emitted in the downward direction. Polyester film with a thickness of about 0.9 mg/cm^2 is readily available and easily handled. However, it is too thick for accurate work with the lower energy beta emitters. For this purpose, thin films (≃5 to 10 µg/cm^2) are prepared by spreading a solution of a polymer in an organic solvent on water. VYNS **(1)**, Formvar **(2)** , and Tygon **(3)** plastics have been used for this purpose.

7.6.1.3 The films must be made electrically conducting (since they are a part of the chamber cathode) by covering them with a thin layer (2 to 5 µg/cm^2) of gold or palladium by vacuum evaporation. The absorption loss of beta particles in the film must be known. Published values can be used, if necessary, but for accurate work an absorption curve using very thin absorbers should be taken **(1)** . The "sandwich" method, in which the film absorption is calculated from the decrease in counting rate that occurs when the source surface is covered with a film of the same thickness as the backing film, is suitable for the higher beta energies.

7.6.1.4 The source itself must be very thin and deposited uniformly on the support to obtain negligible self-absorption. Various techniques have been used for spreading the source; for example, the evaporation of ^{63}Ni-dimethylglyoxime onto the support film **(1)**, the addition of a TFE-fluorocarbon suspension **(3)**, collodial silica, or insulin to the film as spreading agents, and hydrolysis **(2)** . Self-absorption in the source or mount can be measured by 4-π beta-gamma coincidence counting **(4, 5)**. The 4-π beta counter is placed next to a ~~sodium iodide scintillation crystal,~~ NaI(Tl) detector, or a portion of the chamber wall is replaced by a ~~sodium iodide crystal,~~ NaI(Tl) detector, and the absolute disintegration rate is evaluated by coincidence counting **(6, 7)**. By adding a suitable beta-gamma tracer, the method has been used for pure beta as well as beta-gamma emitters **(8)**. Accurate standardization of pure low-energy beta emitters (for example, ^{63}Ni) is difficult, and the original literature should be consulted by those inexperienced with this technique.

7.6.1.5 Photon (gamma and strong X-ray) scintillation counters with geometries approaching 4-π steradians can be constructed from NaI(Tl) crystals in either of two ways. A well crystal (that is, a cylindrical crystal with a small axial hole covered with a second crystal) will provide nearly 4-π geometry for small sources, as will two solid crystals placed very close together with a small source between them. The counts from both crystals are summed as in the gas-flow counter. The deviation for 4-π geometry can be calculated from the physical dimensions. For absolute gamma-ray counting, the efficiency of the crystal for the gamma energy being measured and the absorption in the crystal cover must be taken into account. Additional information on scintillation counting is given in 7.6.4. The liquid scintillation counter is also essentially a 4-π counter for beta particles, since nearly all the radiations are emitted into and interact with the detecting medium.

7.6.2 *Low-Geometry Counters*—This type of instrument is particularly useful for the absolute counting of alpha particles. The alpha emitter, in the form of a very thin solid source, is placed at a distance from the detector such that only a small fraction (<1 %)

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[7] The boldface numbers in parentheses refer to the list of references appended to these practices.

of the alpha particles are emitted in a direction to enter the counter. This solid angle is obtained from the physical measurements of the instrument. The space between the source and the detector is evacuated to eliminate the loss of alpha particles by absorption in air. The detector can be any counter that is 100 % efficient for all alpha particles that enter the sensitive volume—a gas-flow proportional counter with a window that is thin (less than approximately 1 mg/cm^2) compared to the range of the alpha particles or the semiconductor alpha detector with a 1-mg/cm^2 covering. The advantages of this instrument for absolute alpha counting are that: (*1*) the effect of absorption of alpha particles in the source itself is kept to a minimum since only particles that travel the minimum distance in the source enter the detector (particles that have longer paths in the source are emitted at the wrong angle, and (*2*) backscattered alpha particles (those that are emitted into the source backing and are reflected back up through the source) lose sufficient energy so that they cannot enter the detector. One such instrument is described in Reference **(9)** .

7.6.3 *Internal Gas Counters*:

7.6.3.1 The internal gas counter is so named because the radioactive material, in the gaseous state, is placed inside a counting chamber and thus becomes part of the counting gas itself. It is useful for high-efficiency counting of weak beta- and X-ray emitters. The radiations do not have to penetrate a counter window or solid source before entering the sensitive volume of a detector. The counter may be an ionization chamber, or it may be operated in the Geiger-Mueller or proportional mode. Most present-day instruments are of the latter type, and they generally take the form of a metal or metal-coated glass cylinder as a cathode with a thin anode wire running coaxially through it and insulated from the cylinder ends. A wire through the wall makes electrical contact to the cathode. The counter has a tube opening through which it may be connected to a gas-handling system for filling. The purity of the gas is important for efficient and reproducible counting, particularly in the proportional mode.

7.6.3.2 In a modification of the internal gas counter, scintillation counting has been used in place of gas-ionization counting. The inner walls of the chamber are coated with a scintillation material and the radioactive gas introduced. An optical window is made a part of the chamber, and the counting is done by placing this window on a multiplier phototube to detect the scintillations. This system is particularly useful for counting radon gas with zinc sulfide as the scintillator. Additional details on internal gas counting may be found in Reference **(10)** .

7.6.4 *Spectrometers and Energy-Dependent Detectors*:

7.6.4.1 The availability of energy-dependent detectors (detectors whose output signal is proportional to the energy of the radiation detected) that are easy to operate and maintain and have good resolution makes it possible to measure not only the total activity of a radioactive sample but the energy spectrum of the nuclear radiations emitted. Nuclear spectrometry is most useful for alpha particles, electromagnetic radiation (gamma and X rays), and conversion electrons, since these radiations are emitted with discrete energies. Beta spectra have more limited use since beta particles are emitted from a nucleus with a continuous energy distribution up to a characteristic maximum (E-max), making a spectrum containing several different beta emitters difficult to resolve into its components. The advantages of spectrometric over total activity measurements of radioactive sources are increased selectivity, sensitivity, and accuracy because nuclide identification is more certain, interference from other radioactive nuclides in the sample is diminished or eliminated, and counter backgrounds are reduced since only a small portion of the total energy region is used for each radiation. The detectors for alpha spectra are gridded ion-chambers and silicon semiconductor detectors. These are described in Practice D 3084. ~~Gridded ion-chambers are no longer available commercially and must be constructed by the user.~~ A variety of semiconductors can be purchased, and these detectors have essentially replaced ion-chambers for alpha spectrometry, although the chambers have the advantages of high efficiency (nearly 50 %) for large-area sources.

7.6.4.2 The principal detectors used for gamma-ray spectrometry are high purity germanium semiconductors (HPGe) and thallium-activated sodium iodide scintillation ~~crystals, NaI(Tl), and lithium-drifted germanium semiconductors, Ge(Li).~~ crystals (NaI(Tl)). For X rays and very low energy gamma rays, ~~lithium-drifted silicon semiconductor Si(Li), intrinsic Ge,~~ HPGe and gas-filled thin (approximately 1 mg/cm^2) window proportional counters are used. Sodium iodide is hygroscopic, so the crystal must be hermetically sealed, and the entire crystal-phototube package must be light-tight. The complete spectrometer also requires a high-voltage power supply for the phototube (usually operated at 800 to 1000 V), a preamplifier, linear amplifier, pulse-height analyzer, and output recorder. The crystal is packaged in aluminum or stainless steel. The portion of the cover through which gamma rays enter is normally thinner than the rest of the package in order to reduce low-energy photon attenuation. Sodium iodide crystals are available in a large range of sizes and shapes, from 25 by 25-mm cylinders to hemispheres and cylinders at least 305 mm in diameter. Information on the types of crystal packages and mountings that can be used is available from the manufacturers.

7.6.4.3 Germanium ~~and silicon~~ detectors are junction-type semiconductor devices in which a large sensitive region has been produced by ~~drifting metallic lithium into germanium or silicon under~~ the ~~influence~~ refinement of ~~an electric field at an elevated temperature (100~~ germanium to ~~400°C).~~ extremely low impurity levels. The crystal functions as a "solid ion chamber" when a high voltage is applied. ~~Because of~~ To provide the ~~high mobility of the small lithium atom, the Ge(Li) detector must be kept cold to prevent the diffusion of lithium out of the crystal. In addition,~~ semiconductor function, and in order to obtain ~~high~~ optimum resolution, the detector must be operated at low temperatures to reduce thermal noise. At room temperature, sufficient free electrons will be present in the crystal to obscure the measurement of gamma and X rays (but not of alpha particles). Consequently, the HPGe detectors are operated and kept ~~at liquid nitrogen temperatures~~ in the range of 80 to 100 degrees Kelvin by a cryostat consisting of a metallic cold-finger immersed in a ~~Dewar flask~~ dewar containing liquid ~~nitrogen. If the Ge(Li) detector is allowed to warm to room temperature for a short time, its resolution will deteriorate and in an hour~~ nitrogen or ~~so it will lose sufficient lithium so that it cannot function as a detector without redrifting.~~ by thermoelectric cooling. The detector is kept hermetically sealed in a vacuum to prevent impurities from condensing on the surface and lowering its resistance and is thermally and electrically isolated, usually by vacuum, to reduce heat transfer from the room to the crystal, and to maintain the high voltage required for the crystal to be a diode. A low atomic number material, such as aluminum, is the usual covering, and a molecular sieve pump is incorporated into the system to maintain the vacuum. The electronic components required to obtain spectra are similar to those for sodium iodide crystals, except that because smaller pulses must be measured, high-quality electronics are needed. The complete system includes a high-voltage bias supply for the detector (up to 5000 V for large depletion volumes), a ~~preamplifier, amplifier~~ preamplifier (usually charge-sensitive), amplifier, biased amplifier (if needed), pulse height analyzer, and recording device. Current technology provides for digital signal processing for the signal immediately after the signal leaves the preamplifier. The digital signal processing technology provides improved resolution and peak energy stability across a large range of ambient temperatures and signal processing rates. In more advanced products, the signal processing, bias supply, and multichannel pulse height analyzer are provided as a single component.

7.6.4.4 A gamma ray entering either a NaI(Tl) crystal or a semiconductor detector may lose all or part of its energy in the detector. ~~In~~ When all of the ~~former case,~~ energy is absorbed in the detector, through multiple Compton interactions or the photoelectric effect, a full energy peak is obtained. Otherwise, only part of the energy will be observed and a Compton continuum spectrum is seen. An alternative process for high-energy gamma rays (>1.02 MeV) is pair production, in which an electron-position pair is produced, and gamma-ray peaks are observed at 0.511-MeV intervals below the full energy peak. The two most important operating characteristics of gamma detectors are efficiency and resolution. The “peak-to-Compton” or “peak-to-valley” ratio is frequently given in the literature and is related to both efficiency and resolution. These parameters should be specified by the manufacturer and the conditions under which they were measured should be given, normally by reference to an international standard (for example, ANSI N42.14) describing the measurement terms and conditions.

7.6.4.5 The resolution of ~~sodium iodide crystals is usually~~ a gamma-ray detector may be specified in terms of the width of the full-energy gamma-ray peak at half its maximum height—the “full width at half maximum” (FWHM)—or as the ratio of the FWHM (in units of energy) to the energy of the peak centroid. The later quantity is typically expressed as a percentage. This is shown graphically in the gamma-ray spectrum in Fig. 2. ~~The resolution improves~~ While the FWHM increases with increasing ~~energy and~~ energy, the percent resolution improves. The standard for comparison is usually percent resolution of the 0.662-MeV gamma ray emitted in the decay of ^{137}Cs. Good ~~sodium iodide crystals~~ NaI(Tl) detectors have resolutions in the range of 6.5 to 7 % for ^{137}Cs. Detection efficiency for the same geometry and window thickness is a function of several parameters and much published information on efficiencies for various energies, detector sizes, source-to-detector distances, and other variables is available **(11)**. The efficiency for gamma-ray detection may be expressed in various ways. Of primary interest in spectrometry is the full peak efficiency—the fraction of incident gamma rays that give a full-energy peak for a particular source-detector configuration. For a 102-mm thick NaI(Tl) ~~crystal,~~ detector, with the source on the surface (zero distance), this fraction is approximately 0.24 for the 0.662-MeV gamma-ray of ^{137}Cs and approximately 0.14 for the 1.33-MeV gamma-ray of ^{60}Co. The “peak-to-valley” or “peak-to-Compton” ratio is the ratio of counts at the maximum height of the full-energy peak to the counts at the minimum of the Compton continuum (Fig. 2). A high ratio indicates narrow peaks, that is, good resolution, for that particular efficiency. The Compton spectrum does not give useful information in gamma-ray spectrometry and can be considered as “noise.” The ratio varies with energy and is frequently given for the 1.33-MeV peak of ^{60}Co. It increases as the crystal size increases, and, after passing through a minimum, increases as the source-to-detector distance increases, since a larger fraction of the gamma rays pass through the full depth of the crystal. A peak-to-valley ratio of 12:1 for a crystal is very good. This ratio can be increased by

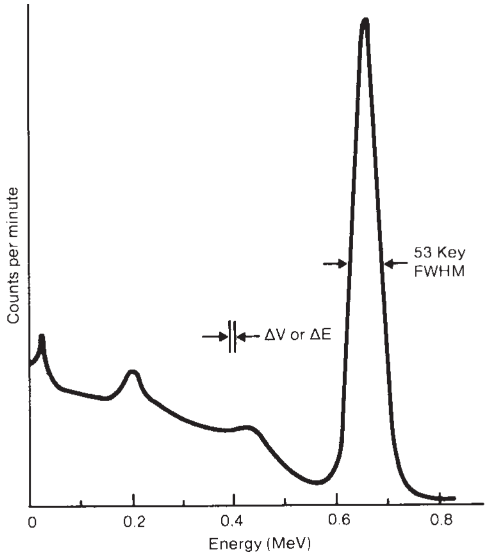


FIG. 2 Pulse Height or Energy Spectrum of Cesium-137

anti-coincidence shielding to cancel Compton events as described in 7.6.5. The efficiency of silicon for gamma-rays is considerably less than sodium iodide because of its lower atomic number (the efficiency for photoelectric absorption of gamma-rays is proportional to Z^5) and lower density (the density of NaI is 3.7 g/cm^3 and of silicon 2.4 g/cm^3).

7.6.4.6 For a 1-MeV gamma-ray, the total absorption coefficient is about 2 ~~mm^{-1}~~ mm^{-1} for sodium iodide, 1.5 ~~mm^{-1}~~ mm^{-1} for silicon, and 3 ~~mm^{-1}~~ mm^{-1} for germanium. ~~However, germanium detectors are not yet available~~ As defined in ~~sizes approaching that of sodium iodide. The~~ IEEE and IEC standards, the efficiency of ~~a Ge(Li)~~ an HPGe detector is generally expressed by comparison with that of a 76 by 76-mm cylindrical NaI(Tl) detector. Comparison is made between the full-energy peak efficiencies for the 1.33-MeV gamma ray of ^{60}Co when the source is 250 mm from the detector. A germanium detector with a volume of 35 cm^3 has an efficiency approximately 5 % that of a 76 by 76-mm NaI(Tl) crystal. Larger ~~Ge(Li)~~ HPGe detectors are now available with relative efficiencies of ~~25~~ greater than 200 %. The very large HPGe detectors provide signal to ~~30 %.~~ noise ratios and resolution far superior to that offered by NaI(Tl).

7.6.4.7 There are limitations in the efficiency of the light production and collection processes in the ~~sodium-iodide-photomultiplier~~ NaI(Tl) detector system that make its resolution inferior to that of semiconductor detectors. One important factor is that about 500 eV are required to produce an electron at the photocathode in a ~~sodium iodide~~ NaI(Tl) detector system, while the average energy to produce the analogous electron-hole pair in silicon is only 3.5 eV and in ~~germanium~~ HPGe 2.8 eV. The resolution of semiconductor detectors does not change greatly with energy. Presently available ~~germanium~~ HPGe detectors have resolutions of 1.5 to 2.8 keV at 1.33 MeV and are from ~~3~~ 10 to ~~3~~ 200 % efficient as compared to a 76 by 76-mm NaI(Tl) detector. ~~This~~ The greater resolution makes this detector the one of choice for gamma-ray ~~spectrometry and cancels~~ spectrometry; the ability to ~~some extent~~ produce very large high purity HPGe crystals also cancels the effect of the higher efficiency previously available only from ~~sodium iodide.~~ NaI(Tl). Since the pulses from a single photopeak are spread over a much smaller energy range in ~~germanium~~ HPGe than in ~~sodium iodide,~~ NaI(Tl), the background under the peak is much less. This means that for small sources of moderately energetic ~~gamma rays, germanium~~ gamma-rays, HPGe is more sensitive (that is, better detection capability) than ~~sodium iodide.~~ NaI(Tl). This is indicated in Table 1, where the efficiencies and backgrounds of 76 by 76-mm ~~sodium iodide crystal~~ NaI(Tl) detector and a 35-cm^3(5.5 % efficiency) ~~germanium~~ HPGe detector are compared.

7.6.4.8 Spectra of beta particles and conversion electrons can be obtained with sodium iodide and semiconductor detectors sufficiently thick (a few centimetres) to absorb the particles completely. One disadvantage of ~~sodium iodide~~ NaI(Tl) and ~~cooled lithium-drifted~~ cooled semiconductors is their relatively thick entrance windows. Other semiconductor detectors, particularly the silicon surface barrier type, have thin entrance windows and can be used for beta particles at room ~~temperature or better at 0°C.~~ temperature. The resolution of silicon surface barrier detectors is 5 to 10-keV ~~resolution~~ for 600-keV electrons ~~is better than the~~ and 12 to 30-keV resolution for 5-MeV alpha particles.

7.6.4.9 Good spectra of low-energy beta particles, conversion electrons, and X rays can be obtained with a gas-flow proportional counter provided that a linear preamplifier is used. The resolution is intermediate between NaI(Tl) and HPGe. To reduce backscattering, the chamber should be made of low *Z* material. A counter constructed of a cylinder of graphite-impregnated plastic, poly(methyl methacrylate) ends, and a thin coaxial center wire gives good spectra for such radiations **(11)**. A hole is cut into the outer wall and covered with aluminized polyester film to provide a thin entrance window. ~~Argon (90 %)-methane (10 %)~~ Methane (10 %)-argon (90 %) is a suitable counting gas.

7.6.4.10 Organic scintillators, such as anthracene and polystyrene polymerized with scintillating compounds, are also useful for beta spectrometry. They are packaged with a phototube in a manner similar to a sodium iodide crystal~~s~~. Liquid scintillation

TABLE 1 Comparative Performance of NaI(Tl) and HPGe~~(Li)~~ Gamma-Ray Detectors

Note—

NaI = 76 by 76-mm cylindrical detector.
Ge(Li) = 35-cm^3 active volume, 5.5 % efficiency.
A = small source placed on detector.
B = 57 by 57 by 57-mm thick source place on detector.
Counting efficiency = percent of photons emitted from the source that give a full-energy peak.
Shielding = 152 mm of iron, 3.2 mm of lead.
Counting time = one 30 000-s count for both source and background.
Detection limit = the number of photons emitted from the source whose net count equals twice the counting error, or

$$N + N_B + 2(N - N_B)^{1/2}$$

where *N* is the total number of counts recorded when the source is measured and N_B is the total number of counts recorded when the background is measured.

Photon Energy Detector	Background (cps) Under Peak	Counting Efficiency,%		Detection Limit, photons/s	
		A	B	A	B
0.14 MeV					
NaI	24	26	18	2.4	3.5
Ge	0.7	12	4	0.92	2.8
0.66 MeV					
NaI	20	14	9	4.1	6.3
Ge	0.11	1.3	0.68	3.6	6.8
1.33 MeV					
NaI	8	5.8	3.8	6.2	9.5
Ge	0.055	0.75	0.38	4.5	8.9

mixtures also give beta spectra, and the output of a commercial liquid scintillation counter can be fed into a multichannel pulse-height analyzer to obtain a spectrum **(2)** . A spectrum of ^{210}Pb ^{210}Bi ^{210}Po in Fig. 3 shows the resolution obtainable by liquid scintillation counting of aqueous samples in a dioxane-based solution. The ^{210}Bi curve is from a beta particle, and the ^{210}Po peak is from an alpha particle. Organic scintillators are preferable to ~~sodium iodide~~ NaI(Tl) for beta spectrometry because less backscattering occurs.

7.6.4.11 The output pulses of any energy-dependent detector, after linear amplification, must be sorted out according to energy to obtain the spectrum of incident radiation. The high resolution available in detectors requires analyzers with hundreds of channels to realize their full resolving power. The amplified pulse is digitized by an analog-to-digital converter (ADC), and the resulting number for a particular pulse is recorded in a pulse counter whose location is determined by digital circuitry. This makes it possible to use a digital computer to count and store in its memory the number of pulses in each channel. This conversion and storage is relatively slow, and the analyzer is blocked from processing a second pulse until the previous processing is completed. The time required to process a pulse increases with channel number. The instruments now available are sufficiently fast for almost all ~~water~~ environmental measurement purposes. Some loss of pulse information is acceptable, as the analyzers measure and record “live time” fairly accurately. Thus, the counting time recorded by the analyzer will be the actual time it was in a condition to receive detector pulses, and not the elapsed time. To maintain good accuracy, the activity of the sample should be adjusted to give live times of 90 % or more. A computer ~~may be permanently~~ is typically combined with the ADC ~~to operate only in the pulse-height analyzer mode (“hard wired”), or a separate~~ and ~~larger computer is “soft wired”~~ has a program to ~~the ADC and can also be programmed for~~ control detector operations ~~other than pulse height analysis, such as~~ and perform data reduction and ~~spectrum resolution. In either case, this type~~ analysis. The program may provide an output of ~~analyzer makes possible automatic digital readout on printers, paper, and magnetic tape, automatic~~ the spectrum ~~plotting, cathode-ray tube curve~~ and results to a monitor, a printer or a digital ~~presentation, and internal data reduction.~~ storage device.

7.6.4.12 All multichannel pulse-height analyzers currently available are ~~transistorized,~~ digital, and are fairly reliable instruments and relatively easy to operate. Their maintenance and repair is, however, a specialized skill similar to other computer repair. In comparing analyzers, some of the important specifications to consider are the number of channels, count capacity, stability, live-time accuracy, linearity, type of pulse input acceptable, and ADC speed. The minimum number of channels useful for NaI(Tl) gamma-spectrometry is 128; ~~Ge(Li)~~ HPGe detectors should be used with at least a 1000-channel analyzer and alpha and beta spectra can profitably use 100 to 400 channels, depending on the energy range to be covered. Analyzers with 4096 channels are fairly common, and larger analyzers are available for special purposes.

7.6.4.13 Semiconductor detectors require low-noise, charge-sensitive amplifiers. Because of their excellent resolution, semiconductor detectors are often used with a biased amplifier following the main amplifier to isolate a portion of the spectrum for analysis. This makes it possible to use smaller analyzers than would otherwise be necessary.

7.6.5 *Anti-Coincidence Counters*:

7.6.5.1 Substantial background reduction can be achieved in beta and gamma counters by surrounding or covering the sample detector with another detector also sensitive to beta or gamma radiation, and connecting them electronically so that any pulse

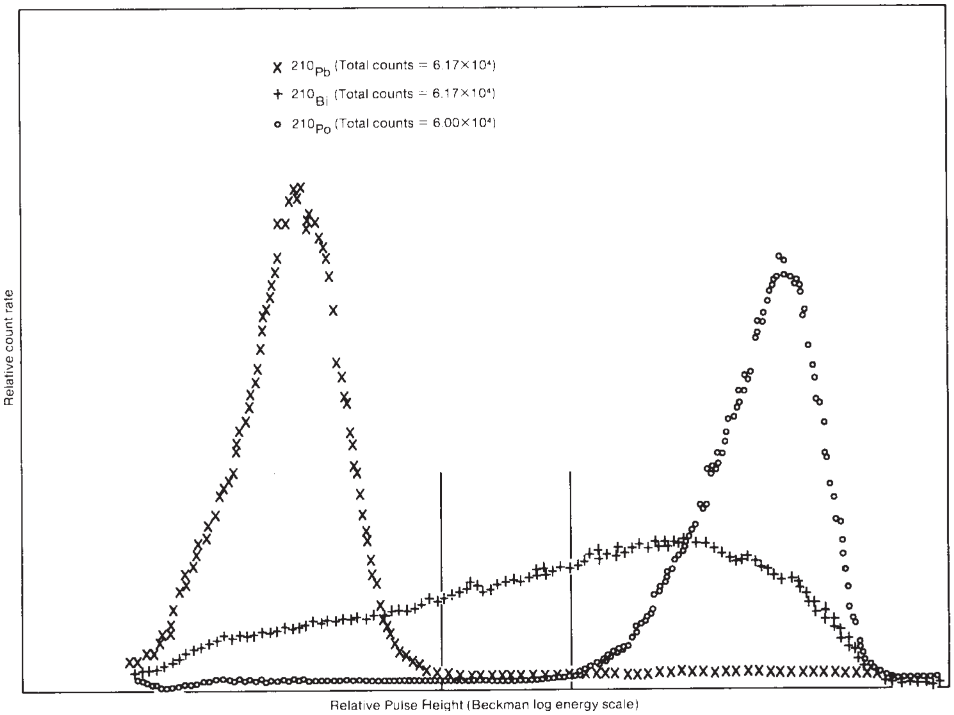


FIG. 3 Spectrum of ^{210}Pb ^{210}Bi ^{210}Po

appearing in both detectors is cancelled and not recorded as a count. This is usually referred to as anti-coincidence shielding, and is recommended for obtaining very low backgrounds. This type of counter was used for many years in directional studies of cosmic rays, and was first applied to reducing the background of beta counters by Libby in his study of natural ^{14}C. The thick metal shielding (lead, iron, or mercury) ordinarily used to reduce cosmic-ray and gamma-ray background must also be present, and is placed outside the anti-coincidence shielding. Gas-filled beta detectors are generally shielded by gas-filled detectors, and such anti-coincidence shielding is effective primarily against the particulate component of cosmic rays. The anti-coincidence shielding for beta counters may consist of a number of long Geiger-Mueller tubes ("cosmic-ray counters") surrounding the sample detector or a large (approximately 152 mm square) gas-flow detector, with several anode wires so the entire area of the counter is sensitive, placed just above the sample detector. For counting solid beta sources, the sample detector has a diameter of 25 to 51 mm. Surrounding these counters on all six sides there is frequently a layer of high-purity copper to absorb gamma rays emitted from the outermost shielding, and 102 to 152 mm of lead or iron on all six sides. This is the form usually taken by the commercially available anti-coincidence shielded beta counters. Plastic or inorganic scintillators could also be used as the anti-coincidence shielding.

7.6.5.2 Anti-coincidence shielding of gamma-ray detectors operates in a similar way, and is particularly useful in reducing the Compton continuum background of gamma rays **(12)** . Gamma rays that undergo Compton scattering and produce a pulse in both the detector and the anti-coincidence shield are cancelled electronically. Ideally, only those gamma rays that are completely absorbed in the sample detector itself produce a count that is recorded with the total energy of the gamma ray (full-energy peak). There are second-order effects that prevent complete elimination of Compton scattering, but the improvement is substantial. The anti-coincidence shield can be a large ~~sodium iodide~~ NaI(Tl) or plastic scintillator suitably attached to phototubes. They usually have a large annular hole into which the sample detector, a smaller ~~sodium iodide~~ NaI(Tl) detector, or ~~germanium iodide~~ HPGe detector is placed **(13, 14)**.

7.6.6 *Coincidence Counters*:

7.6.6.1 In coincidence counting, two or more radiation detectors are used together to measure the same sample, and only those nuclear events or counts that occur simultaneously in all detectors are recorded. The coincidence counting technique finds considerable application in studying radioactive decay schemes; but in the measurement of radioactivity, the principal uses are for the standardization of radioactive sources and for counter background reduction.

7.6.6.2 Coincidence counting is a very powerful method for absolute disintegration rate measurement **(6, 15)**. Both alpha and beta emitters can be standardized if their decay schemes are such that β-γ, γ-γ, β-β, α-β, or α-X-ray coincidence occur in their decay. Gamma-gamma coincidence counting with two ~~sodium iodide crystals,~~ NaI(Tl) detectors, and the source placed between them, is an excellent method of reducing the background from Compton scattered events. Its use is limited, of course, to counting nuclides that emit two photons in cascade (which are essentially simultaneous), either directly as in ^{60}Co, by annihilation of positrons as in ^{65}Zn, or by immediate emission of a gamma ray following electron capture decay. If the ~~crystals~~ detectors are operated with single-channel pulse-height analyzers to limit the events recorded from each ~~crystal~~ detector to one of the full-energy peaks of the photons being emitted, then essentially only those photons will be counted. Non-coincident pulses of any energy in either one of the ~~crystals~~ detectors will be cancelled, including cosmic-ray photons in the background and degraded or Compton scattered photons from higher energy gamma rays in the sample. Thus, the method reduces interference from other gamma emitters in the sample. If, instead of single-channel analyzers, two multichannel analyzers are used to record the complete spectrum from each ~~crystal,~~ detector, singly and in coincidence, then the complete coincident gamma-ray spectrum can be obtained with one measurement. The efficiency for coincidence counting is low since it is the product of the individual efficiencies in each ~~crystal,~~ detector, but the sensitivity is generally improved because of the large background reduction **(16)**. This technique is often referred to as two-parameter or multidimensional gamma-ray spectrometry.

7.6.6.3 Additional background improvement is obtained if the two ~~crystals~~ detectors are surrounded by a large annular ~~sodium iodide~~ NaI(Tl) or plastic scintillation ~~crystal~~ detector connected in anti-coincidence with the two inner ~~crystals.~~ detectors. In this case a gamma ray that gives a pulse, but is not completely absorbed in one of the two inner ~~crystals,~~ detectors, and also gives a pulse in the surrounding ~~crystal,~~ detector, is cancelled electronically **(13, 16)**. This provides additional reduction in the Compton scattering background. ~~Lithium-drifted germanium~~ HPGe detectors may be used in place of the inner ~~sodium iodide crystals~~ NaI(Tl) detectors for improved resolution and sensitivities **(14)**.

7.7 All of the equipment described in Section 7 is available commercially.

8. Sampling

8.1 Collect the sample in accordance with Practice D 1066 or Section 14.3 of Practices D 3370.

8.2 Sample an appropriate volume depending on the expected concentration of radioactivity in the water. For precise measurements without long counting times, it is advisable to count an aliquot that contains at least 40 ~~dps~~ Bq of radioactivity.

8.3 Chemical treatment of samples to prevent biological or algal growth is not recommended and should be avoided unless essential. When necessary, select the reagents used to avoid chemical interaction with the radioactive species in the sample. Analyze samples promptly.

8.4 Chemical treatment of samples to retain radioactive species in solution may be used but carefully select the specific treatment. The use of oxidizing acids such as HNO_3 is not recommended when iodide is present since it may be oxidized to iodine and lost or be absorbed into the plastic containers if they are used. In some cases, extreme chemical treatment may be used to keep a particular chemical species in solution; examples are strongly alkaline conditions to hold molybdenum and ruthenium in solution, or acid conditions with fluoride ion to keep zirconium in solution. The addition of an acid such as hydrochloric is generally desirable to reduce hydrolysis and the loss of activity on container walls. Frequently, samples will contain insoluble material. In such cases, treat the sample by one of the following methods:

8.4.1 Filter the insoluble material and analyze both the filtrate and insoluble matter on the filter. During filtration, some material may be sorbed onto the filter and assumed to be insoluble when in fact it is soluble.

8.4.2 Centrifuge the sample and analyze both phases. Wash the insoluble phase with distilled water to remove all soluble material without dissolving the insoluble fraction.

8.4.3 In either of the above separations when the total activity is required, the insoluble matter may be dissolved and recombined with the soluble fraction. When radioactivity is left on the walls of the sample container, desorb it and add it to the sample.

8.5 Composite samples may be made by mixing aliquots of successive samples collected by an automatic sampler. Analysis of such composite samples yields average results only and loses information on short-term effects.

9. Instrument Operation and Control

9.1 The following procedures ensure that counting equipment is functioning properly and remains in calibration.

9.2 *Establishing Counter Characteristics* :

9.2.1 The first step in instrument control is to establish the operating characteristics of the system. Carefully measure the efficiency for counting the nuclide of interest under the conditions to be employed. Select counting conditions, that is, optimize gain, discriminator setting, and voltage for the radionuclide of interest. Set the operating voltage so that any change in counting rate is minimized for a given voltage fluctuation. Adjust the discriminators to exclude noise and unwanted interferences from the nuclides being counted. Make adjustment to optimize the signal-to-noise ratio. When the counting conditions have been selected, monitor known interferences to determine such things as the effect of counting betas in an alpha counter or alpha pulses in a beta proportional counter, etc.

9.2.2 Make daily performance checks and maintain a log for each instrument. This log should include the count for a standard and a background. When the counting rate differs statistically from the expected performance, perform additional counting to determine if the counter is malfunctioning. High background can indicate either an instrumental problem or counter contamination.

9.2.3 Certified standards are available from several suppliers. Most solution standards have the pH controlled and carrier added to ensure that hydrolysis or sorption, or both, do not change the concentration of the solution. When dilutions are made it is important to maintain the stability of the solution by diluting with a proper matrix. Store the standard in a container that minimizes evaporation by loss either through the walls or out of the stopper.

9.2.4 If a planchet is prepared as a standard, place it in a suitable container for storage, which will prevent the surface containing the activity from being contacted. A recommended practice is the preparation of two standards, using one and storing the second for periodic checks to see that the working standard has not been altered.

9.3 *Counter Control and Tolerance Charts*:

9.3.1 Evaluate the daily standard counts made on any counter on a statistical or tolerance basis. The best way to do this is to maintain a control chart on each counter. A control chart is a graph showing the number of counts recorded in a fixed counting period against the day of the year. Select a radioactive source having a suitable emission rate to give several thousands of counts in a relatively short counting time. Each measurement should be at least 10 000 counts in a given measurement time period. Determine the initial entry, ~~$\bar{N}$~~ $\bar{C}$, from the average of at least ten measurements over a period of days. For a statistical control chart, enter the ~~error~~ control limit bands of ±2~~S_x~~ s_c *and* ±3~~S_x~~ s_c (~~S_x~~ s_c = $\sqrt{\bar{C}}$ ~~$\bar{N}$~~ *) and draw lines on the chart that allow for decay of the standard over the year. For a tolerance chart, select the tolerance band for* ~~$\bar{N}$~~ $\bar{C}$ *, based on needs, to which the counter will be held, for example, from ±1 % or ±3 % of the* ~~$\bar{N}$~~ $\bar{C}$ *, etc. The tolerance band is equivalent to the ±3*~~S_x~~ s_c *control limit band of a control chart. Draw control chart and tolerance band lines on the chart that allow for decay of the radioactive standard over the year. An example of a control chart is shown in Fig. 4.*

9.3.2 For a statistical control chart, enter the result of the standard count in the control chart and take the following action:

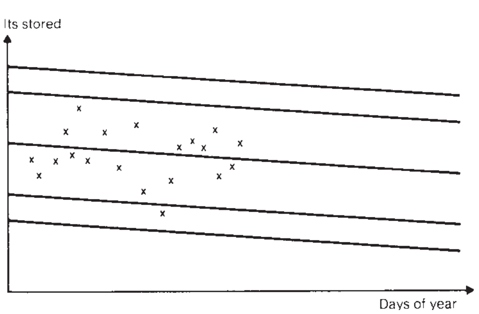


FIG. 4 Typical Counter Control Chart

9.3.2.1 If the result is inside the ±2 ~~S_x~~s_c *band, consider the counter to be in control.*

9.3.2.2 If the result lies outside the ±2 ~~S_x~~s_c *band, but inside the* ±3~~S_x~~s_c *band, consider the counter to be in control but flag this result.*

9.3.2.3 If the result lies outside the ±3 ~~S_x~~s_c *band, consider the counter out of control. Corrective action is needed if repeated counts remain outside the* ±3~~S_x~~s_c *band.*

9.3.3 For a tolerance chart, enter the result of the standard count in the tolerance chart and take the following action:

9.3.3.1 If the result lies outside the tolerance band, consider the counter out of control. Corrective action is needed if repeated counts remain outside the tolerance band.

9.3.4 For alpha and gamma spectrometry, it is also important to monitor for system resolution (see Fig. 5).

9.3.5 In addition to the control charts made on each counter, keep all pertinent information about the system in a log book or permanent file.

Counter Logs

α and β Counters	*γ Spectrometers*
standard counts	standard counts
background counts	standard resolution
system changes	background counts
control charts	control charts

10. Counting Statistics

10.1 Each nuclear disintegration **(6, 17, 18)** is a completely random and independent process. Established methods of statistical analysis are available to describe the random phenomenon of nuclear disintegration. The total number of particles counted in a time period can be shown to deviate from the average in accordance with the expression, ~~N~~$C \pm \sqrt{\cancel{N}C}$ where ~~N~~C is the number of counts in the counting period t. Similar behavior is found in the background count rate for any counter and the recorded background will deviate from the average in accordance with the expression ~~N~~ $C_{\cancel{B}b} \pm \sqrt{\cancel{N_B}C_b}$ *where* ~~N~~$C_{\cancel{B}b}$ *is the number of counts recorded in the counting period* $t_{\cancel{B}b}$. ~~*T*~~ *In the situation* ~~*f*~~ *where the net count is 0, the* $\sqrt{N}$ *expression may be replaced by* $\sqrt{N + 1}$*. Determine the net counting rate as follows:*

~~$N_x = \text{Net count rate} = \left(\frac{N}{t} - \frac{N_B}{t_B}\right)$~~ (1)

$$C_n = \text{Net count rate} = \left(\frac{C}{t} - \frac{C_b}{t_b}\right) \quad (1)$$

The standard deviation (uncertainty at the 68.3 % confidence level) of the ~~Net~~ net count rate, ~~N_x~~C_n, *is defined as follows:*

~~$S_x = \sqrt{(N/t^2) + (N_B/t_B^{\ 2})}$~~ (2)

$$s_n = \sqrt{(C/t^2) + (C_b/t_b^{\ 2})} \quad (2)$$

or for the net count rate with its standard deviation:

~~$\text{Net count rate} = \left(\frac{N}{t} - \frac{N_B}{t_B}\right) \pm \sqrt{\frac{N}{t^2} + \frac{N_B}{t_B^{\ 2}}}$~~ (3)

$$\text{Net count rate} = \left(\frac{C}{t} - \frac{C_b}{t_b}\right) \pm \sqrt{\frac{C}{t^2} + \frac{C_b}{t_b^{\ 2}}} \quad (3)$$

In practice, many counts will be made that deviate from the average by more or less than the standard deviation, since ~~S_x~~s_n *by definition would give those limits X −* ~~S_x~~s_n *and X +* ~~S_x~~s_n*, which would include approximately 68 % of all observed values. In terms of the population of observations between limits, one can express the chance* ~~*of*~~ *or probability of a result being between the limits X −* ~~KS_x~~ks_n *and X +* ~~KS_x~~ks_n *as "confidence levels," where* ~~*K*~~*k is simply a multiplier for* ~~S_x~~s_n*. It may also be described as the "coverage factor" as used in the GUM (see ISO Guide to the Expression of Uncertainty in Measurement). In evaluating the effect of* ~~*K*~~ *k for total counts (per measurement period) greater than* ∼*60, one can tabulate* ~~*K*~~*k and the "confidence level."*

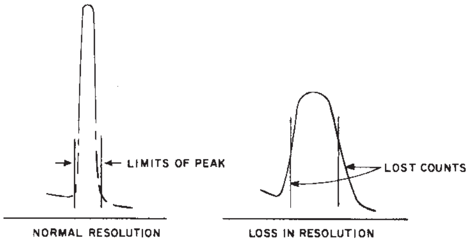


FIG. 5 System Resolution in Alpha and Gamma Spectrometry

~~Confidence Level, %~~ Confidence Level, %	~~K~~ k
68.3	1.0
90	1.65
95	1.96
99	2.58
99.7	3.0

10.2 The 95 % confidence limit is frequently used and has been generally accepted since ~~errors~~ counts outside this band are considered statistically significant. In practice, ~~K~~k *is often rounded to 2.0 and the term 2*~~S~~$s_{\bar{x}\ n}$, *or 2*σ, ~~*error*~~ *standard deviation is often used. In some cases the 3*~~S~~$s_{\bar{x}n}$, *or 3*σ, ~~*error*~~ *standard deviation is used and gives the 99.7 % confidence limits.*

10.3 *Precision*—There is a measure of the reproducibility of a measurement. There are a number of items that affect the precision of radioactivity measurements, the more important of these are as follows:

10.3.1 *Position*—For point sources the observed radioactivity is proportional to the reciprocal of the square of the distance of the source from the detector. Measure all sources being directly compared at the same source-to-detector distance.

10.3.2 *Radiation Scattering*—Changes in the sample support and backing material can seriously affect the precision of radiation measurements, particularly that of beta radiation. Standards and samples should have the same backing material. The use of a sample support rack constructed of a material with low atomic number will reduce the effect of side-scattering. Scattering from backing material can be minimized by the use of a thin, low atomic number material.

10.3.3 *Background*—Measure this radioactivity with no sample near the detector and subtract from all measurements of gross sample activity. This requirement becomes more critical as the net sample activity becomes small with respect to the background. Perform routine periodic measurements of the background to check for possible detector contamination or malfunction.

10.3.4 *Absorption*—Alpha and beta radiations are partly absorbed by the sample and by all substances separating the sample from the detector. This effect is usually small for gamma radiation but beta and alpha radiation are seriously affected. If sources of the same atomic number and mass are compared on the same counter in the same geometry, absorption will be constant under these conditions but should not be ignored. Since the sample mass may vary significantly, construct calibration curves to correct for changes in self-absorption.

10.3.5 *Quenching*—This is any process that reduces the photon output in a scintillation system; this in turn reduces the measured count rate. Quenching can be caused by such things as sample color and chemicals in the sample. The need to correct for this phenomenon can be avoided if samples of the same color and same chemical composition are compared. If this is not possible, most instrument manufacturers and texts describe methods for construction of calibration curves to correct for this phenomenon **(19, 20, 21)**.

10.4 *Overall Uncertainty in a Determination* —Report measurement results with the estimated overall measurement uncertainties shown. This overall measurement uncertainty may be expressed as the relative standard deviation (RSD). There are two approaches to this: (*1*) rigid propagation of uncertainties, which is not sound in practice since the individual components are not well characterized, and (*2*) a combination of known uncertainties. In combining uncertainties, use the following relationship:

~~$$Sx = \sqrt{S_1^{\ 2} + S_2^{\ 2}} \quad (4)$$~~

$$U = \sqrt{U_1^{\ 2} + U_2^{\ 2}} \quad (4)$$

U 1 U2

where:

~~S~~ U = overall uncertainty of the measurement (RSD),
~~S~~U_1 = random counting uncertainty (RSD), and
~~S~~U_2 = other random uncertainties (RSD).

The work can be divided into two main classes, as follows:

10.4.1 *Gamma Spectrometry*—Assuming that the integrity of the sample is known, the sampling and treatment effects are at a minimum and then only two significant terms are present. These are accuracy of calibration and precision of counting:

~~$$S_{\gamma spec} = \sqrt{S_c^{\ 2} + S_a^{\ 2}} \quad (5)$$~~

$$U_{\gamma spec} = \sqrt{U_c^{\ 2} + U_a^{\ 2}} \quad (5)$$

Uc Ua

where:

~~Sγ~~$U\gamma_{spec}$ = overall error for the measurement ~~(Relative Standard Deviation or RSD),~~ (RSD),
~~S~~U_c = calibration uncertainty (RSD), and
~~S~~U_a = counting uncertainty (RSD).

10.4.2 *Separative Work*—The sample treatment introduces uncertainty into the measurement. One estimate of great merit is as follows:

~~$Sx = \sqrt{S_M{}^2 + S_a{}^2 + S_c{}^2}$~~ (6)

$$U = \sqrt{U_m{}^2 + U_a{}^2 + U_c{}^2} \quad (6)$$

Um Ua Uc

where:

~~S_x~~U = overall uncertainty for the ~~measurement (RSD),~~ measurement,
~~S_M~~U_m = intrinsic precision of the ~~method (RSD),~~ method,
~~S~~U_a = counting ~~uncertainty (RSD),~~ uncertainty, and
~~S~~U_c = calibration ~~uncertainty (RSD).~~ uncertainty.

The intrinsic precision may be determined by doing a single-operator precision (SOP) test at three or four activity levels.

SOP Test Data

Level, dps	S_x(%RSD)	S_a(%RSD)	S_M(%RSD)		
0.2	8	S_{a1}	S_{M1}	}	Average $\bar{S}_M$
2	4	S_{a2}	S_{M2}		
200	2	S_{a3}	S_{M3}		

The use of methods that have been round-robin tested will provide a method where ~~S~~s~~$_x$~~$_n$ *is known.*

10.4.3 The overall uncertainty of a measurement may be used to make a statistical comparison between two duplicate or replicate results. This can be done using a relationship of following form **(39)**:

$$\frac{A_{a1} - A_{a2}}{\sqrt{S_{x1}^2 + S_{x2}^2}} \quad (7)$$

The resulting value of this relationship can be used to assess the statistical agreement between the two results.

10.5 *Minimum Detectable Activity* :

10.5.1 The minimum detectable activity (MDA) is a statistical measure of the sensitivity of a counting determination. In the analysis of environmental samples or discharge samples, the sensitivity obviously becomes an important and often critical item. To determine if a measured sample count rate is different than the instrument background, the measured sample count rate is evaluated against the decision (or critical) level as defined by Currie **(38)**. The decision level is defined as the“ quantity of analyte at or above which a decision is made that a positive quantity of analyte is present.” The industry standard has set the probability of erroneously reporting a detectable nuclide in an appropriate blank or sample at 0.05. Under this conversion, the decision level is mathematically defined as:

~~$\text{Decision Level Count Rate} = DLCR = 1.645 \times S_o$~~ (8)

$$\text{Decision Level Count Rate} = DLCR = 1.645 \times s_o \quad (8)$$

where ~~S~~s_o is defined as s~~$_B$~~$_b \times \sqrt{2}$ and ~~S_B~~s_b *is the standard deviation of the background count rate. The above DLCR equation assumes paired observations, for example, the sample and appropriate blank (background) are counted for the same length of time. The a priori minimum detectable activity* **(38)** *is defined as “The amount of a radionuclide, which, if present in a sample, would be detected with a 0.05 probability of non-detection while accepting a 0.05 probability of false detection (erroneously detecting that radionuclide in an appropriate blank sample).” The a priori minimum detectable activity, MDA, is mathematically defined as:*

~~$MDA = \frac{2.71}{T \times K} + 4.65 \frac{S_B}{K}$~~ (9)

$$MDA = \frac{2.71}{t \times K} + 4.65 \frac{s_b}{K} \quad (9)$$

where ~~S_B~~s_b is defined above, ~~T~~t is the sample counting time, and K includes the efficiency of detection of the counter as defined in the subsequent sections, the unit activity conversion factor, and the appropriate branching ratio of the emission under consideration. The MDA is expressed in units of Bq. Again this mathematical definition assumes paired observations. The *a priori* MDA is a “before the fact” calculation, or, more specifically, assumed to be a nominal MDA without prior knowledge of the activity level or nuclide interferences within the sample. For the *a priori* MDA to be truly representative of the radiation measurement system~~'~~'s detection capability for a given matrix, the use of the appropriate blank with interferences is required. However, since prior knowledge of the sample constituents and interferences is not usually available, the *a priori* MDA is typically a nominal value defining the system~~'~~'s capability for a given appropriate blank. It should also be noted that the mathematical definitions above may not be appropriate for those cases where the number of counts is small, for example, less than 10 to 20 counts **(40)**.

10.5.2 Where complex NaI(Tl) spectra are being analyzed by a weighted least squares unfolding analysis and the components that are being sought are not completely separable by instrument resolution, consider the *a priori* MDA detection limit as 3.29 times the standard deviation (68 % confidence level) of the nuclide~~'~~'s activity when a blank matrix is evaluated. The blank matrix should be evaluated using the library of nuclides routinely applied for such matrix unfolding applications.

10.5.3 In computing S_x, consider the sample and background counting rates, that is:

~~$S_x = \sqrt{(N/t^{2}) + (N_B/t_B{}^{2})}$ cps~~ (9)

$$U = \sqrt{(C/t^2) + (C_b/t_b{}^2)} \quad (10)$$

If the sample and background counting times are equal ($t = t$~~$_B$~~$_b$), *use a slightly different form:*

~~$S_x = \sqrt{N + N_B}$ counts~~ (10)

$$U = \sqrt{C + C_b}\ \text{counts} \quad (11)$$

11. Calculation and Symbols

11.1 To calculate the ~~amount of activity~~ count rate of a substance, *X*, use the following general method:

~~$C = 1/VY\,[(N/t) - (N_B/t_B)$~~ (11)

$$R_n = \frac{1}{VY}\left(\frac{C}{t} - \frac{C_b}{t_b}\right) \quad (12)$$

Cb tb Rn

where:
~~N~~ C = number of counts accumulated,
t = sample counting period, s,
~~N_B~~ C_b = number of background counts accumulated,
t~~$_B$~~$_b$ = background counting period, s,
~~C~~ = net counts per second per millilitre, cps/mL,
R_n
V = volume of sample, mL, and
Y = fractional recovery of species (unity in methods where complete recovery is assumed).

11.2 Calculate the ~~disintegration rate~~ activity concentration (~~D~~ AC) *as follows:*

~~$D = (C - R)/E$~~ (13)

$$AC = (R_n - R_{rb})/E \quad (13)$$

E Rrb

where:
~~D~~ AC = disintegrations per second per millilitre, dps/mL or Bq,
E = fractional efficiency of counter, cps/dps, and
R_{rb} = reagent blank correction (cps) measured on an actual blank sample.

11.3 In gamma spectrometry use the following:

~~$E = GP$~~ (13)

$$E = IE_{pp} \quad (14)$$

Epp

where:
~~G~~ I = fractional abundance of the gamma ray concerned, and
~~P~~ = photopeak detection efficiency (fraction).
E_{pp}

In beta counting use the following:

~~$E = f_d\, f_a\, f_s\, f_m$~~ (14)

$$E = E_d\, K_a\, K_s\, I \quad (15)$$

Ka Ks I

where:
~~f~~E_d = detector efficiency factor for a source of given beta energy,
~~f~~K_a = absorber factor for the total absorber thickness (detector window + air space + absorber + cover) for a source of given beta energy,
~~f~~K_s = self absorption and backscatter factor for a given precipitate thickness and given beta energy, and
~~f_m~~ I = number of charged particles per disintegration.

~~The concentration of substance *A* may be calculated as follows:~~

~~$A = \frac{D}{3.7 \times 10^{4}}$~~ (15)

~~where *A* = concentration in microcuries per millilitre (µCi/mL). For~~

For decay correction, use the expression:

~~$$D = D° \exp(-0.693\, t_{/T}) \quad (16)$$~~

$$AC = AC_0 \exp(-0.693\, t_e/t_{1/2}) \quad (16)$$

tet1/2

where:

~~$D°$~~ AC_0 = disintegration rate at time zero, that is, reference time,
t_e = elapsed time between measurement and reference time, and
~~T~~ $t_{1/2}$ = half-life of the nuclide.

Parent-daughter relationships are commonly shown by use of subscripts 1, 2, etc., for count rates, disintegration rates, or values of t and T.

ALPHA COUNTING

12. Scope

12.1 This practice covers the measurement of the alpha particle radioactivity of water. It is applicable to alpha emitters having energies above 3 MeV at activity levels above 0.02 ~~dps~~ Bq per sample.

13. Summary of Method

13.1 Alpha particles are characterized by intense loss of energy in passing through matter. This intense loss of energy is used in differentiating alpha radioactivity from other types through the dense ionization or intense scintillation it produces. This high rate of loss of energy in passing through matter, however, also makes sample preparation conditions for alpha counting more stringent than is necessary for other types of radiation.

14. Alpha Detectors

14.1 Alpha radioactivity is normally measured by one of several types of detectors in combination with suitable electronic components. The detector devices most used are ionization chambers, proportional counters, silicon semiconductor detectors, and scintillation counters. The associated electronic components in all cases would include high-voltage power supplies, preamplifiers, amplifiers, scalers, and recording devices.

15. Detection Technique

15.1 In all of these systems, the initial event is converted to an electrical pulse that is amplified to a voltage sufficient to operate the scaler mechanism where provision is made for recording each pulse. The number of pulses per unit of time is directly related to the disintegration rate of the test sample. The efficiency of the system can be determined by counting standards prepared in the same manner as the samples. An arbitrary efficiency factor can be defined in terms of a different radionuclide, such as natural ~~uranium, polonium-210, plutonium-239, or americium-241.~~ uranium, ^{210}Po, ^{239}Pu, or ^{241}Am.

16. Measurement Variables

16.1 The measured alpha-counting rate from a sample will depend on a number of variables. The most important of these variables are geometry, source diameter, self-absorption, absorption in air and detector window, coincidence losses, and backscatter. These are discussed in detail in the literature **(2, 3)** and in many cases can be measured or corrected for by holding conditions constant during the counting of samples and standards. These effects may be described by the following relationship:

~~$$\text{dps} = \text{cps}\,(G_p)(f_{bs})(f_{aw})(f_d)(f_{ssa})(f_c)$$~~

$$A = RK_gK_{bs}K_{aw}K_dK_{ssa}K_eK_c \quad (17)$$

R KgKbsKawKdKssa Ke Kc

where:

~~cps~~ A = recorded counts per second, corrected for background,
~~dps~~ R = alpha disintegrations per second,
~~G_p~~ K_g = point source geometry, which is the solid angle subtended by the sensitive area of the detector. The effect of this variable is eliminated by maintaining a constant geometry for both standard and sample counts.
~~f~~ K_{bs} = backscatter factor, or ratio of cps with sample backing to cps without backing. This is not important in alpha counting since backscatter is small. In samples mounted on copper or stainless-steel planchets for counting 2π geometry, backscatter may be taken as 2 % (backscatter factor equals 1.02) without any serious error. In samples mounted on platinum, backscatter may be taken as 4 %.
~~f~~ K_{aw} = factor to correct for losses due to absorption in air or window of external counters.
~~f~~ K_d = factor to correct for dispersion of the source from a point configuration. The effect of this variable is eliminated by preparing the standard in the same configuration as the samples.

~~f~~$\underline{K}_{ssa}$ = factor to correct for the absorption and scattering of alpha particles in the sample and its mount. This is covered further in Section 19.
~~f_c~~$\underline{K_e}$ = factor to correct for losses due to the resolving time of the detector and its associated electronics.
~~f~~$\underline{K_c}$ = $(1 - nt)$ where n is the observed counting rate, cpm, and t is the resolving time in minutes.

16.2 Alpha counters have low backgrounds and high efficiencies. However, some counters are easily contaminated internally and care should be taken to avoid contamination. Silicon detectors operated in vacuum may become contaminated due to recoil from sources. Recoil contamination can be eliminated by maintaining an air absorber of 12 µg/cm^2 between the source and the detector **(4).**

17. Interference

17.1 Some alpha counters are sensitive to beta radiation with a degree of efficiency depending on the detector **(2, 3).** In these cases, electronic discrimination is often used to eliminate the smaller pulses due to beta particles.

18. Apparatus

18.1 *Ionization Chambers*—Alpha particles entering the sensitive region of an ionization chamber produce dense ionization of the counting gas. The electrons are collected at the anode, thus developing a voltage pulse. Gridded ion chambers are used for alpha spectrometry but have been replaced by solid-state detectors. The major advantage of the ion chamber is its ability to measure large area samples with essentially a 2π geometry. The chambers are operated at an over-pressure with gas mixtures such as 10 % methane-90 % argon or 2 % ethylene-98 % argon. The peak resolution of such a detector is about 50-keV FWHM (see Practice D 3084).

18.2 *Proportional Counters*:

18.2.1 Alpha particles entering the sensitive region of a proportional counter produce ionization of the counting gas. In this case, the electrons are accelerated towards the anode, producing secondary ionization and developing a large voltage pulse by gas amplification. Proportional counters are usually operated in the "limited proportional" range, where the total ionization is proportional to the primary ionization produced by the alpha particle.

18.2.2 Proportional detectors are generally constructed of stainless steel or aluminum (see Fig. 6). No additional shielding is required for alpha proportional counting. The counter should be capable of accepting mounts up to 51 mm in diameter. Proportional detectors are available in two types, either with or without a window between the sample and the counting chamber. The manufacturer's specifications for either type should include performance estimates of background count rate, length and slope of the voltage plateau, and efficiency of counting a specified electrodeposited standard source, along with the type of gas used in the tests. For a window flow counter, the window thickness, in milligrams per square centimetre, should also be specified. With windowless low counter the sample and sample mount should be made of an electrical conductor in order to avoid erratic behavior due to static charge buildup.

18.2.3 Alpha emitters are counted with proportional instruments in 2π, or 50 %, configuration. Two-π geometry is obtained by placing the sample on a flat planchet inside the detector. Half the alpha particles are emitted downward into the planchet, of which approximately 2 % are backscattered into the upward direction. The other half are emitted upward into the gas volume.

18.2.4 Typical parameters for the alpha windowless flow counter are: background count rate—10 counts/h; length of voltage plateau—300 V; and slope of voltage plateau—1 %/100 V for an electrodeposited source. For a window flow counter, typical values are: window thickness—1 mg/cm^2; background count rate—10 counts/h; length of voltage plateau—300 V; slope of voltage plateau—1 %/100 V for an electrodeposited source; and efficiency—35 to 40 % for an electrodeposited source. Gases commonly used in both types of alpha proportional counters are 10 % methane-90 % argon, pure methane, or pure argon.

18.3 *Scintillation Counters*

18.3.1 In a scintillation counter, the alpha particle transfers energy to a scintillator, such as zinc sulfide (silver activated). The transfer of energy to the scintillator results in the production of light at a wavelength characteristic to the scintillator, and with an intensity proportional to the energy transmitted from the alpha particle. The scintillator medium is placed in close proximity to the cathode of a multiplier phototube; light photons from the scintillator strike the photocathode and electrons are emitted. The photoelectrons are amplified by the multiplier phototube and a voltage pulse is produced at the anode.

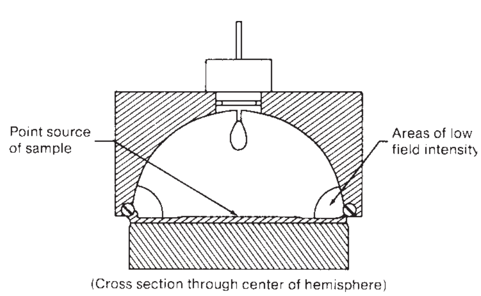


FIG. 6 Typical Chamber Geometry for Proportional Detector

18.3.2 The counter size is limited by the multiplier phototube size, a diameter of 51 mm being the most common. Two types of systems may be employed. In the first, the phosphor is optically coupled to the multiplier phototube and either covered with a thin (<1 mg/cm ^{2}) opaque window or enclosed in a light-proof sample changer. With the sample placed as close as possible to the scintillator, efficiencies approaching 40 % may be obtained. The second system employs a bare multiplier phototube housed in a light-proof assembly. The sample is mounted in contact with a disposable zinc sulfide disk and placed on the phototube for counting. This system gives efficiencies approaching 50 %, a slightly lower background, and less chance of counter contamination.

18.3.3 A major advantage of alpha scintillation counting is that the sample need not be conducting. For a 51-mm multiplier phototube with the phosphor coupled to the tube, typical values obtained are: background count rate—0.006 counts/s; and efficiency for an electrodeposited standard source—35 to 40 %. With a disposable phosphor mounted on the sample, typical values are: background count rate—0.003 counts/s; and efficiency for an electrodeposited standard source—45 to 50 %. For both systems, voltage plateau length is 150 V with a slope of 5 %/100 V.

18.3.4 Liquid scintillation counting of alpha emitters with a commercially available instrument overcomes many of the problems inherent in other techniques **(6, 9, 10, 23).** Typical background counting rates range from 0.1 to 0.2 counts/s. Sample preparation involves mixing the sample aliquot with a suitable liquid scintillator solution or gel phosphor before counting. Planchet preparation is eliminated, volatile components are retained, and the completely enclosed sample cannot contaminate the counting chamber. The sample is uniformly distributed in the scintillator so there is no self-absorption, resulting in a counting efficiency of almost 100 %. Because of the high alpha energies, considerable chemical quenching effects can be tolerated before counting efficiency is reduced. Coincidence losses are small in liquid scintillation counting at count rates up to 2×10^{4} cps. For samples that contain both alpha and high-energy beta emitters, difficulties do arise in distinguishing between the two. The problem is due primarily to the broad continuum of beta energy distribution up to the maximum energy and the poor resolution of liquid scintillation spectrometers. This problem is aggravated because the light yield per million electronvolt of alpha particles in most liquid scintillators is approximately tenfold lower than a beta particle of equivalent energy, putting the pulses from alphas and high-energy betas in the same region. Correction for beta activity may be made by certain mathematical or graphical techniques. It is preferable to separate the alpha emitter from the bulk of the beta activity by chemistry.

18.4 *Semiconductor Detectors*

18.4.1 The semiconductor detectors used for alpha counting are essentially solid-state ionization chambers. The ionization of the gas in an ionization chamber by alpha particles produces electron-ion pairs, while in a semiconductor detector electron-hole pairs are produced. The liberated charge is collected by an electric field and amplified by a charge-sensitive amplifier. In general, silicon surface barrier detectors are used for alpha counting. These detectors are *n*-type base material upon which gold is evaporated to make a contact. The semiconductor material must have a high resistivity since the background is a function of the leakage current. This leakage current is present in an electric field since the starting material is a semiconductor, not an insulator. The reversed bias that is applied reduces the leakage current and a "depletion layer" of free-charge carriers is created. This layer is very thin and the leakage current is extremely low; therefore, the interactions of photons with the detector will have negligible effect. Since the detector shows a linear response with energy, any interactions of beta particles with the detector can be eliminated by electronic discrimination. The semiconductor is of special interest in alpha counting where spectrometric measurements may be made since the average energy required to produce an electron-hole pair in silicon is 3.5 ± 0.1 eV compared to the 25 to 30 eV needed to produce an ion pair in a gridded ionization chamber. Consequently, silicon detectors provide much improved resolution and also normally have lower background count rates.

18.4.2 The detector size is generally less than 25 mm in diameter since the resolution decreases and cost increases with detector size. For best results, the sample should be electrodeposited to make a lower mass source. The detector is operated in a vacuum chamber. Typical backgrounds range from 8×10^{-5} to 2×10^{-4} ~~counts/s.~~ cps. Additional specifications for detectors may be found in Practice D 3084.

19. Absorption

19.1 The range of alpha particles is a few centimetres in air and a few thousandths of a centimetre in solids. Even fairly short path lengths in air and passage through thin windows will result in the absorption of some fraction of the alpha radiation.

20. Calibration

20.1 Calibrate alpha-counting equipment for specific nuclide measurement using a standard of similar alpha energy measured under exactly the same conditions as the sample that is to be counted.

20.2 When the gross alpha activity of a sample is to be measured (Test Method D 1943), calibrate the counting equipment using a standard. The standard should contain the same weight and distribution of solids as the sample and be mounted in an identical manner. If the samples contain variable amounts of solids or carrier, prepare a calibration curve relating the weight of solids present to counting efficiency. Express the efficiency factor (*E*) as a fraction of the disintegration rate of the standard.

20.3 Calibrated standards of ~~plutonium-239, americium-241, polonium-210, thorium-228, radium-226, uranium-233, uranium-235,~~ ^{239}Pu, ^{241}Am, ^{210}Po, ^{228}Th, ^{226}Ra, ^{233}U, and uranium (natural) are readily available either from the National Institute of Standards and Technology or commercial organizations. Calibrated standards of other alpha-emitting radionuclides may be procured from the above suppliers upon special request.

21. Source Preparation

21.1 Appropriately mount the sample whose alpha activity is to be determined for the final measurement. This can be accomplished in a number of ways, some of which are dependent upon the counting method used. In all cases, however, mount the sample in a uniform and reproducible manner.

21.2 The most straightforward and commonly used method of preparing a sample for alpha counting is by evaporation onto a suitable planchet. Evaporated sources are usually counted in end-window or windowless proportional counters or in zinc sulfide scintillation counters. Producing a thin, uniform, and reproducible source by this technique is difficult and considerable care is necessary. Carry out evaporations slowly to avoid spattering and usually perform under infrared lamps.

21.3 Mounting of solid-free samples resulting from radio-chemical separations of specific nuclides is best accomplished by evaporating the sample in the center of a planchet. Define an area in the center of the planchet and keep the sample within this area during evaporation. This can usually be accomplished by carefully evaporating the sample in small portions. Samples that contain solids present the problem that the material does not evaporate uniformly but tends to deposit in crystals and aggregates, producing uneven deposits consisting of rings, ridges, or localized amounts of solids. This will be the case when water samples are prepared for alpha counting without chemical separation of specific nuclides. These samples are best mounted by allowing the sample to evaporate on the entire surface of the planchet. This will produce a more uniform source and minimize the self-absorption effects. Surfactants can be used to spread the sample during evaporation. Dissolution of the solids on the planchet with water and re-evaporation may be necessary to obtain a uniformly distributed source.

21.4 Samples that contain carriers can be mounted using a precipitation technique. Keep the amounts of carrier used small (<10 mg) so as to minimize self-absorption. Precipitate the carrier by suitable chemical procedures and mount the precipitate for counting by either filtration through a filter or by evaporation of a slurry of the precipitate onto a planchet. The final surface density of such a deposit should be less than 1 mg/cm ^{2}. Count these samples in an end-window proportional counter or in a zinc~~-~~ sulfide scintillation counter because they are nonconductors.

21.5 Filtration of a precipitate onto a filter produces a more uniform and reproducible source than the evaporation technique. When evaporating a slurry of a precipitate onto a planchet, take care to obtain a uniform and reproducible source. However, this is usually more easily obtained with a slurry than when evaporating a solution.

21.6 Electrodeposition is the most effective and widely used technique for preparation of a thin, uniform, and reproducible source for alpha counting. Electroplated sources can be counted in any alpha-counting equipment except, of course, a liquid scintillation counter. The chemical separation of specific nuclides from the sample is required prior to electrodeposition. Electrodeposit the alpha-emitting element onto a flat metal disk. Platinum, stainless steel, nickel, and tantalum have been used for this purpose. Information on electrodeposition of alpha emitters has been published **(24-26).** The electrodeposition technique produces the most suitable sources for alpha spectrometry. One disadvantage of this technique is that for some samples quantitative deposition is not always obtained and a recovery correction factor must be applied. Electrodeposition followed by alpha spectrometry is particularly useful for alpha analysis in which yield measurements are determined by the addition of a known amount of an isotope of the same element to be measured (see Practice D 3084).

21.7 The use of liquid scintillation counting avoids some of the difficulties encountered in preparing samples for solid source counting, but has the disadvantage of blindly counting all alphas, as does any gross method. Self-absorption problems are eliminated and counting efficiencies are 90 to 100 %. Prepare samples for liquid scintillation counting by dissolving the sample in a scintillation mixture **(27, 28)** or extracting the alpha-emitting element into a scintillation mixture containing the extractant **(29).**

22. Calculation

22.1 This method is useful for comparing activities of a group of samples as in a tracer experiment. The following equation may be used to calculate the results:

~~$\text{Activity, } C = \frac{1}{VY}\left(\frac{N}{t} - \frac{N_B}{t_B}\right)$~~

$$R_n = \frac{1}{VY}\left(\frac{C}{t} - \frac{C_b}{t_b}\right) \qquad (18)$$

Cb tb Rn

where:

~~N~~ C = number of counts accumulated,
t = sample counting period, s,
~~N_B~~ C_b = number of background counts accumulated,
~~t_B~~ t_b = background counting period, s,
~~C~~ = net counts per second per millilitre,cps/mL,
R_n
V = volume of sample, mL, and
Y = recovery of species (unity in methods where complete recovery is assumed).

22.2 The disintegration rate or concentration in microcuries may be calculated as follows:

~~Disintegration rate, $D = C/E$~~

$$AC = R_n/E \qquad (19)$$

where:

~~D~~ AC = disintegrations per second per millilitre, dps/mL or Bq, and

E = efficiency of counter (fraction), cps/dps.

~~Concentration, $A = D/(3.7 \times 10\ 4)$~~

~~where A = concentration in microcuries per millilitre (µCi/ml).~~

22.3 Results may also be reported in terms of equivalent natural uranium activity, employing an efficiency determined by use of a natural uranium reference standard. ~~Alpha activity equivalent to natural uranium, Bq/mL = C/Eu~~

~~where~~

~~E_u = efficiency of counter for natural uranium (fraction).~~

22.4 To properly evaluate the result, calculate the uncertainty associated with the concentration in accordance with the recommended procedure in Section 10.

BETA COUNTING

23. Scope

23.1 This practice covers the measurement of the beta particle radioactivity of water. It also covers the general techniques used to prepare and measure the activity resulting from radiochemical separation of specific nuclides or groups of nuclides in water samples. It is applicable to beta emitters with activity levels above 0.4 ~~dps~~ Bq per sample for most counting systems. For samples of lower activity, see Practice D 3085 for specific techniques. The method is not applicable to samples containing radionuclides that are volatile under conditions of the analysis. General information on radioactivity and measurement of radiation may be found in the literature **(7, 23).**

24. Summary of Method

24.1 Beta radioactivity may be measured by one of several types of instruments that provide a detector and a combined amplifier, power supply, and scaler. The most widely used detectors are proportional or Geiger-Mueller counters, but scintillation systems offer certain advantages.

24.2 Among the gas ionization-type detectors, the proportional-type counter is preferable because of the shorter resolving time and greater stability of the instrument. For preparing solid sources from water samples for beta activity measurement, the test sample is reduced to the minimum weight of solid material having measurable beta activity by precipitation, ion exchange, or evaporation techniques. For measuring solid sources resulting from individual radiochemical separation procedures, the precipitate is appropriately mounted for counting.

24.3 Beta particles entering the sensitive region of the detector produce ionization or scintillation photons that are converted into an electrical pulse suitable for counting. The number of pulses per unit time is directly related to the disintegration rate of the sample by an overall efficiency factor. This factor combines the effects of sample-to-detector geometry, sample self-shielding, backscatter, absorption in air and in the detector window (if any), and detector efficiency. Because most of these individual components in the overall beta-particle detection efficiency factor vary with beta energy, the situation can become complex when a mixture of beta emitters is present in the sample. The overall detection efficiency factor may be empirically determined with prepared standards of composition identical to those of the sample specimen, or an arbitrary efficiency factor can be defined in terms of a single standard such ~~as cesium-137~~ as ^{137}Cs or other nuclide. Gross counts can provide only a very limited amount of information and therefore should be used only for screening purposes or to indicate trends.

24.4 Liquid scintillation counting avoids many sources of error associated with counting solid beta sources: such as (*1*) self-absorption, (*2*) backscattering, (*3*) loss of activity during evaporation due to volatilization or spattering, and (*4*) variable detection efficiency over a wide beta-energy range. In addition to the greatly improved accuracy offered by liquid scintillation counting, sample preparation time and counting times are significantly shorter. Sample preparation involves only adding a sample aliquot to the scintillator or gel phosphor. Because every radioactive atom is essentially surrounded by detector molecules, the probability of detection is quite high even for low-energy beta particles. Radionuclides having maximum beta energies of 200 keV or more are detected with essentially 100 % efficiency. Liquid scintillation can, at times, be disadvantageous due to chemiluminescence, phosphorescence, quenching, or the typically higher backgrounds.

24.5 Organic scintillators, such as *p*-terphenyl plus a wave shifter in a plastic monomer, are polymerized to form sheet material of any desired thickness. The plastic phosphor counting system **(8)** has its widest use as a beta particle detector for separated, solid samples rather than for beta spectrometry applications.

24.6 The plastic beta scintillator phosphor is mounted directly on the sample and is discarded after counting. The phosphor-sample sandwich is placed in direct contact with the photomultiplier tube yielding essentially a 2π configuration. Since the output pulse of the detector system is energy dependent, the counting efficiency for a given phosphor thickness of 0.25 mm yields the highest counting efficiency with the lowest background.

24.7 Solid samples (precipitates from radiochemical separations) containing 3 to 5 mg/cm ^{2} of stable carrier are measured in such a system.~~For yttrium-90~~ For ^{90}Y a solid sample of this type would have a counting efficiency of 45 to 50 %.

24.8 A plastic scintillator/phosphor system with a 25-mm photomultiplier tube shielded with 12.7 mm of lead has background in the order of 4×10^{-2} cps. For very low backgrounds, about 4×10^{-3} cps, the photomultiplier tube and sample assembly are fitted into a well-type hollow anode Geiger tube operated in anti-coincidence. The entire assembly is then placed in a heavy shield.

24.9 The system has many advantages but reduction of background is probably most important. The reduction occurs since the scintillation does not see the surrounding mechanical components of the counter. The additional advantage of keeping the counter itself free from contamination by enclosing the phosphor-sample sandwich is also important.

24.10 A note of caution is advisable at this point. Any beta particle detection system, whether internal gas counters or scintillation counters, will detect alpha particles. It is not possible to electronically discriminate against all the alpha pulses.

24.11 If a sample is suspected of containing alpha activity, a separate alpha measurement must be made to determine the alpha contribution to the beta measurement.

25. Solid Source Counting

25.1 The observed count rate for a solid source is the result of the interaction of many variables. The most important of these are the effects of geometry, backscatter, radiation, source diameter, self-scatter and self-absorption, absorption in air and the detector window (for external counters), and coincidence counting losses. These effects have been discussed **(17, 23)** and in many cases can be reduced or corrected for by counting test standards and samples under identical conditions. For absolute measurements of a single, specific nuclide, apply appropriate correction factors. These effects may be described by the following relation:

~~cps = dps (G_p)(f_{bs})(f_{aw})(f_d)(f_{ssa})(f_c)~~

$$A = RK_g K_{bs} K_{aw} K_d K_{ssa} K_e K_c \quad (20)$$

RKgK bsKaw K d Kc

where:

~~*cps*~~ A = recorded counts per second, corrected for background,

~~*dps*~~ R = disintegrations per second (Bq) yielding beta particles,

~~G_p~~ K_g = source-to-detector geometry, which is the solid angle subtended by the sensitive area of the detector. This is corrected for most easily by maintaining constant counting configuration and geometry for standards and sample measurements.

~~*f*~~K_{bs} = backscatter factor, or ratio of cps with backing to cps without backing. This is the phenomenon in which particles originally emitted away from the detector are scattered back toward it by reflection off the source-backing material. Its value ranges from 1 to almost 2, depending on the thickness and atomic number of the backing material. The backscatter error may be minimized by using identical backing material for counting standard and samples.

~~*f*~~K_{aw} = factor to correct for losses due to absorption in the air and window of external counters. It is equal to the ratio of the actual counting rate to that which would be obtained if there were no absorption by the air and window between the source and the sensitive volume of the detector. It is related to the absorption coefficient and density of the absorber by the approximate equation: ~~*f*~~$K_{aw} = \exp(-\mu x)$, where μ = absorption coefficient, in square centimetres per milligram, and x = absorber density in milligrams per square centi- metre. In practice, it can be accounted for, together with the geometric factor, by maintaining the geometry of the counting configuration constant for standards and samples.

~~f_d~~ K_d = factor to correct a disk-source counting rate to the counting rate of the same activity as a point source on the same axis of the system. This can be corrected for by preparing and counting the standard in the same configuration as the samples.

~~*f*~~K_{ssa} = factor to correct for the absorption and scatter of beta particles within the material accompanying the radioactive element. This is further discussed in 28.1.

~~*f*~~K_c = factor to correct the counting rate for instrument-resolving time losses and defined by the simplified equation ~~*f*~~$K_c = (1 - nt)$, where n = observed counts per second and t = instrument resolving time in seconds. Generally, the sample size or source-to-detector distance is selected to preclude counting losses. Such losses are negligible for count rates of less than 13 000 counts per second if proportional detectors are used. Information on the effect of instrument-resolving time on the sample count rate, as well as methods for determining the resolving time of the counting systems, may be found in the literature **(23).**

26. Liquid Scintillation Counting

26.1 The observed count rate for a liquid scintillation sample is directly related to the beta (plus conversion electron) and positron emission rate in most cases. The important exceptions are: (*1*) beta emitters whose maximum energy is below 200 keV, and (*2*) counting systems wherein quenching decreases the expected photon yield, thereby decreasing the overall detection efficiency significantly below 100 %. Low-energy beta emitters, such as tritium~~ or carbon-14,~~ or ^{14}C, can be measured accurately only when the appropriate detection efficiency factor has been determined with a known amount of the same radionuclide counted under identical conditions. Quenching losses are greatest at low beta energies. Quenching may be evaluated by comparison to known-quench standards of the same radionuclide, using the channel ratio technique, or with other techniques as described in the manufacturer's instructions. For absolute measurements of low-energy beta emitters, apply appropriate correction factors as follows:

~~$\mathrm{Bq} = \frac{\mathrm{cps}}{(fe)(fq)}$~~

$$A = \frac{R_b}{EK_q} \tag{21}$$

RbE Kq

where:

~~Bq~~ A = disintegrations per second yielding beta particles,

~~cps~~ R_b = recorded counts per second, corrected for background,

~~fe~~ E = detection efficiency factor (see Note); observed cps/dps ratio for known standard of some radionuclide counted under identical conditions with no quenching, and

~~fq~~ K_q = quench correction factor; observed ratio of cps quenched to cps unquenched for known standard of same radionuclide with equivalent quenching.

NOTE 1—The detection efficiency may be assumed to be unity for beta emitters having maximum energies of 200 keV or greater.

26.2 In tracer studies or tests requiring only measurements in which data are expressed relative to a defined standard, the individual correction factors cancel whenever sample composition, sample weight, and counting configuration and geometry remain constant during the standardization and tests.

26.3 The limit of sensitivity for both Geiger-Mueller and proportional counters is a function of the background counting rate. Massive shielding or anti-coincidence detectors and circuitry, or both, are generally used to reduce the background counting rate to increase the sensitivity **(23).** For a more complete discussion of this, see Practice D 3085.

27. Interferences

27.1 *Solid Sources*:

27.1.1 Material interposed between the test sample and the detector, as well as increasing mass in the radioactive sample itself, produces significant losses in sample counting rates. Since the absorption of beta particles in the sample solids increases with increasing mass and varies inversely with the maximum beta energy, sample residue must remain constant between related test samples and should duplicate the residue of the evaporated standard.

27.1.2 Most beta-particle counters are somewhat sensitive to alpha, gamma, and X-ray radiations, with the degree of efficiency dependent upon the type of detector **(23).** The effect of the interfering radiations on the beta counting rate is more easily evaluated with external-type counters where appropriate absorbers can be placed between the sample and the detector to evaluate the effects of interfering radiation.

27.2 *Liquid Scintillation*—Liquid scintillation samples are subject to interference by substances that quench or enhance the scintillation process. This includes any chemiluminescence that changes the photon yield. Substances such as oxidants, organohalides, ketones, and aldehydes are to be particularly avoided. A second type of quenching is color quenching, in which a colored solution impairs the light collection efficiency for the photons produced.

28. Apparatus

28.1 *Beta Particle Counter*—The end-window Geiger-Mueller tube and the internal or external proportional gas-flow chambers are the two most prevalent types of detectors. Other types of detectors include scintillators and solid-state detectors. The material used in the construction of the detector and its surroundings should contain a minimal level of detectable radioactivity. If the detector is of the window-type, the window thickness may be used in calculating beta-ray attenuation; however, direct calibration of the entire counting system with standards is recommended. The manufacturer should provide all settings and data required for reliable and accurate operation of the instrument. Detectors requiring external positioning of the test sample shall include a support of low-density material (aluminum or plastic), which ensures a reproducible geometry between the sample and the detector. Because different sample-to-detector geometries are convenient for differing sample activity levels, the sample support may provide several fixed positions ranging from 5 to 100 mm from the detector.

28.2 *Liquid Scintillation*—Liquid scintillation counting systems use an organic phosphor as the primary detector. This organic phosphor is combined with the sample in an appropriate solvent that achieves a uniform dispersion. A second organic phosphor often is included in the liquid scintillation "cocktail" as a wavelength shifter. The wavelength shifter efficiently absorbs the photons of the primary phosphor and re-emits them at a longer wavelength more compatible with the multiplier phototube. Liquid scintillation counting systems use either a single multiplier phototube or two multiplier phototubes in coincidence. The coincidence counting arrangement is less likely to accept a spurious noise pulse that occurs in a single phototube, and thus provides lower background. The requirement that both multiplier phototubes respond to each has a slight effect on the overall detection efficiency of betas with E-max >200 keV; however, system response to beta E-max <200 keV will be significant. The need to minimize detectable radioactivity in the detector and its surroundings is likewise important in liquid scintillation counting. To achieve this, scintillation-grade organic phosphors and solvents are prepared from ~~low carbon-14~~ low ^{14}C materials such as petroleum. The counting vials are of low potassium glass or plastic to minimize counts due ~~to potassium-40.~~ to ^{40}K. Liquid scintillation provides a fixed geometry from a given size counting vial and liquid volume.

28.3 *Detector Shield*— The detector assembly should be surrounded by an external radiation shield of massive metal equivalent to approximately ~~5100~~ 51 mm of lead and lined with 3.2-mm thick aluminum. The material of construction should be of minimal detectable radioactivity. The shield should have a door or port for inserting or removing specimens. Detectors having other than completely opaque windows are light-sensitive. The design of the shield and its openings should eliminate direct light paths to the detector window; beveling of the door and the opening generally is satisfactory. Liquid scintillation counting systems must provide an interlock that protects the photocathode of the multiplier phototube from light when the sample counting chamber is opened.

28.4 *Associated Electronic Equipment* —The high-voltage power supply amplifier, scaler, and mechanical register normally are contained in a single chassis. The power supply and amplifier sections are matched with the type of detector to produce satisfactory operating characteristics and to provide sufficient range in adjustments to maintain stable conditions. The scaler should have a capacity for storing and visually displaying at least 9×10^5 counts. The instrument should have an adjustable input sensitivity matched to that of the detector, and variable high-voltage power supply. (An adjustable power supply and meter are unnecessary for liquid scintillation systems.) Counting chambers of Geiger-Mueller and proportional counters contain a suitable counting gas and an electrode. Counting rates that exceed 200 cps must be corrected for dead-time loss when using a Geiger-Mueller tube. As the applied voltage to the electrode is increased, the counting chamber exhibits responses that are characteristic of a particular voltage region. At low voltages of the order of 100 V, there is no multiplication of the ionization caused by a charged particle. At voltages approaching 1000 V, there is appreciable amplification of any ionization within the counting chamber; however, the size of the output pulse is proportional to the amount of initial ionization. When operated in this voltage region, the device is known as a gas-proportional counter. Usually, there is a region at least 100 V wide, known as a plateau, wherein the count rate of a standard is relatively unaffected. The operating voltage for proportional counters is selected to approximate the middle of this plateau in order to maintain stable responses during small voltage shifts. The plateau region is determined by counting a given source at voltage settings that differ by 25 or 50 V. The number of counts at each setting is recorded, and the resultant counts versus voltage are plotted as shown in Fig. 7. Voltage plateau curves are to be remeasured periodically to ensure continued instrument stability, or whenever an instrument malfunction is indicated. If the voltage is increased beyond the proportional region into the 1500 to 2000-V region, the pulse size increases and the dependence on the initial ionization intensity disappears. This is the beginning of the Geiger-counting region, where a single ion pair produces the same large pulse as an intense initial ionization.

28.5 *Alpha Interference*—Alpha particle interference can be substantial and must be considered with any type of beta counter. One technique involves placing a thin absorber between the solid source and the detector. The absorber diameter should exceed that of the detector window. The absorber should be placed against the window to minimize beta particle scatter. Any absorber that stops alpha particles will also attenuate low-energy beta particles somewhat. For example, an aluminum absorber of 7 mg/cm^2 will absorb 48 % of beta particles of 0.35-MeV maximum energy. The alpha-particle absorber is not recommended for use with internal beta-particle detectors, especially when either the composition or activity ratios of the radioelements or radioactivity level might vary significantly between samples. Chemical separation of the alpha- and beta-particle emitters produces a higher degree of accuracy for internal detector measurements. Published information on beta-particle absorption **(23)** should be used as a guide for use of an absorber. In liquid scintillation spectra, the alpha component appears as a peak on the beta continuum and thus provides a basis for resolving the two **(30).**

28.6 *Self-Absorption and Backscatter* :

28.6.1 Radioisotopes emit radiation uniformly in all directions. If the radioisotope is intimately mixed with the detector, as in the case with liquid scintillation counting, essentially all beta particles interact with the detector. With solid source counting, however, the source and detector usually are related by a solid angle of 2π or less. Even so, the number of beta particles reaching the detector can be significantly affected by other factors.

28.6.2 When a radioactive source decays by beta emission, the beta particles (electrons) are emitted with a distribution of energies ranging from zero to definite maximum value. The lower energies are absorbed by the sample itself and this self-absorption is particularly large for certain nuclides, such ~~as carbon-14 and sulfur-35,~~ as ^{14}C and ^{35}S, in which all the beta particles are of low energy. It is therefore desirable to make solid samples as thin as possible.

28.6.3 Self-absorption corrections can be made, if necessary. A series of counts is made on a number of weighed samples of homogeneous material, with the material spread uniformly over the sample mount. The data are corrected for background and graphed on a semilog plot, with the logarithm of the counting rate per milligram of sample plotted against the number of

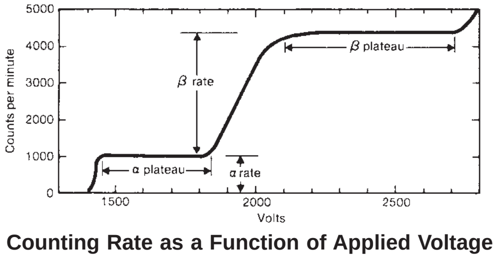


FIG. 7 Counting Rate as a Function of Applied Voltage for a Proportional Counter Exposed to a Source Emitting Both Alpha and Beta Particles

milligrams. The resulting curve can be extrapolated to zero sample weight to give the counting rate per milligram corrected for self-absorption **(3).**

28.6.4 Samples consisting of the final product from radiochemical separations generally are of the same approximate weight from determination to determination because they usually contain the same amount of carrier, and the self-absorption is essentially constant. Corrections can be made by running a standard amount of activity of the nuclide through the procedure, with sample activities expressed in terms of this reference standard.

28.6.5 Beta particles are easily deflected in passing through matter. Some beta particles emitted in a direction away from the detector can be deflected by the source-backing material and scattered toward the detector. This phenomenon leads to an increased number of beta particles reaching the detector over that expressed from geometric considerations alone. Backscatter increases rapidly with increasing thickness and atomic number of the backing material. Backscatter effects may be held constant by maintaining sample size and mounts constant for relative measurements. If backscatter corrections must be made, these corrections can be determined by counting a sample first on thin film mount, and then recounting when backed by the desired backing material. Backscatter does have the effect of increasing the number of particles that reach the detector, thereby increasing the counting efficiency.

29. Calib~~s~~ration and Standardization

29.1 *Calibration and Standardization for General Measurements—Solid Sources*—Place a known amount~~ of cesium-137~~ of ^{137}Cs or other activity (approximately 20 to 200 Bq, with the smaller amount more appropriate for Geiger-Mueller detectors) in a volume of water containing salts equivalent to those of the test samples and prepare for counting as directed. Throughout the experiment, the evaporation, mounting, counting, and the density of the plate solid of this reference must be identical with those of the test samples. Count for the length of time necessary to produce the desired statistical reliability. Then express combined efficiency factor as a percentage of the disintegration rate of the reference standard.

29.1.1 **Caution:** This factor is inaccurate for beta particles whose energies differ appreciably from those~~ of cesium-137;~~ of ^{137}Cs; nevertheless, count rates for solid beta sources of undefined energy are often reported relative to~~ the cesium-137~~ the ^{137}Cs standard. The fact that the 662-keV gamma ray~~ of barium-137~~ of ^{137m}Ba is significantly converted accounts for the fact that about 1.10 electrons leave the source for every disintegration. For this reason,~~ calibrate cesium-137~~ calibrate ^{137}Cs standards in terms of combined beta plus electron emission rate.

29.2 *Liquid Scintillation*—Add a known amount~~ of cesium-137~~ of ^{137}Cs (approximately 370 Bq) to the liquid scintillation "cocktail." The volume and composition of the cesium solution and the "cocktail" should simulate those normally used. For samples without serious quenching problems the counting efficiency should approach 100 %. (Note in 29.1~~ that cesium-137~~ that $_{137}$Cs standards must be calibrated in terms of beta plus conversion electron emission rate.)

29.3 *Calibration and Standardization for Tracer Experiments*—Add a known quantity of activity of a reference solution of the tracer (approximately 200 Bq) to a nonradioactive standard test sample and process.

30. Source Preparation

30.1 *Solid Sources*:

30.1.1 Appropriately mount the sample whose activity is being determined for the final measurement. The exact form of the mount is somewhat dependent upon the particular instrument for counting. In general, most counters will accept either flat plate or dish mounts.

30.1.2 Water samples can be evaporated on either flat disks or dishes, with the dishes being preferred for high solids samples. The dishes should have a flat or concentrically ringed bottom whose diameter is no greater than that of the detector window, preferably having 3-mm high side walls with the angle between the dish bottom and side equal to or greater than 120 deg to reduce side-wall scattering. Sample dishes with verticle side walls may be used but the exact positioning of these dishes relative to the detector is very important. This factor becomes critical for dishes having the same diameter as the detector. Dishes having side walls more than 3 mm in height are not recommended. Dishes should be of a material that will not corrode under the plating conditions and should be of uniform surface density, preferably great enough to reach backscatter saturation **(23).**

30.1.3 Samples resulting from radiochemical separations are customarily in the form of a precipitate on a filter. These can be mounted on plates, dishes, or other suitable backing. Fix the filter paper to the backing with a double-backed adhesive strip or a liquid adhesive, and cover to prevent sample loss and contamination of the counting equipment. Such covering can be accomplished with a cover of transparent plastic tape or film, or by spraying the precipitate with a collodion. Do not use nonconducting coverings in internal flow counters.

30.2 *Liquid Sources*:

30.2.1 Liquid samples of sufficiently high activity level may be added directly to an appropriate amount of liquid scintillator solution. For aqueous samples the liquid scintillator solution must contain a solvent, such as dioxane, which is miscible with water, or a surfactant to maintain a single phase with immiscible solvents. Low-activity liquid samples may be concentrated by evaporation before addition to the liquid scintillator solution.

30.2.2 Liquid samples are sometimes concentrated or separated, or both, by extraction into a suitable organic solvent. This solvent often is suitable for direct addition to the liquid scintillator solution. Caution is advised to avoid solvents that cause substantial quenching.

31. Radioactive Decay and Decay Curves

31.1 Decay curves are useful for identifying radionuclides by their characteristic half-lives and in determining the presence and number of interfering radioactive substances. When two or more beta-emitting isotopes are present in a source, an analysis of the decay curve is required to resolve them since the semilog plot of the disintegration rate versus time is a curve whose slope changes as the isotopic composition changes. After sufficient time the longest-lived activity predominates. The half-life of this long-lived component can be determined from extrapolation of the straight line portion of the curve to t_0, the original count rate for that radionuclide, as shown in Fig. 8. In this graph, the extrapolated line of the 4.6-day half-life component subtracted from the original curve yields a curve that represents all other components; this residual curve may be processed again in the same way to reduce (in principle) any complex decay curve into its component parts. In actual practice, uncertainties in the observed data place a practical limit to a three-component system except in unusual cases, and often a two-component system may not be satisfactorily resolved because of similar decay constants. Since the residual curve in Fig. 8 (1-day half-life) does not deviate from the straight line, and the original curve is concave except in the final portion, a two-component system of independently-decaying activities is indicated. In accumulating decay data, care must be taken to avoid disturbing the source and to locate the sample in the same

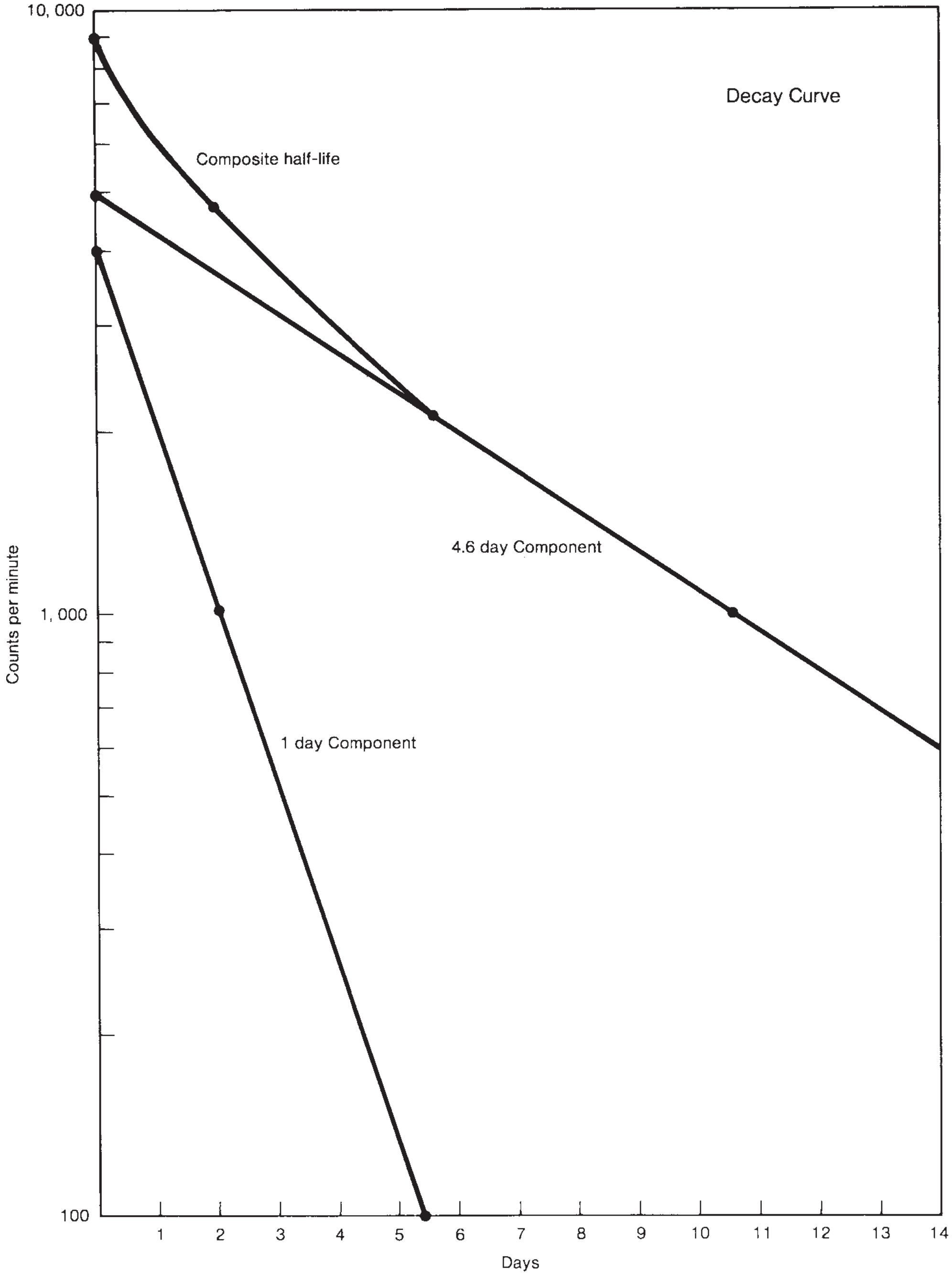


FIG. 8 Decay Curve

geometrical position for each measurement. If the instrument is calibrated for the specific characteristics of the source and for the energies of the radiations measured, the count rate shown at the ordinate intercept of the extrapolated straight lines at t_0 will be equal to the disintegration rate at t_0. However, this technique requires specific beta-energy knowledge for each component of the sample, as well as counting-instrument standardization at these same energies under identical conditions. In practice, gamma spectrometry is preferred (see Test Method D 2459) for resolving mixtures of all radionuclides except pure beta emitters. An additional complication in decay-curve interpretation is the growth of radioactive daughters. See Ref **(17, 23)** for a discussion of this.

32. Beta Particle Maximum Energy

32.1 When a radioactive source decays by beta emission, the beta particles (electrons) are emitted with a distribution of energies ranging from zero to a definite maximum value. The maximum energy of the electrons is characteristic of a particular nuclide and is the "beta energy" shown in nuclear data tables. Determining the approximate maximum energy of the beta particles from a radioactive source thus aids in radionuclide identification.

32.2 The combined effects of a continuous beta spectrum and scattering produces an approximately exponential absorption law for a beta-particle source of a given maximum energy. In interposing varying thicknesses of aluminum between the source and the detector and plotting the count rate versus the thickness of aluminum absorber in milligrams per square centimetre on semilog paper, an empirical absorption curve is produced. The plotted data can be compared to a standard absorption curve determined for a particular nuclide to check for other radionuclides. If the sample and absorber are as close as possible to the detector and only one radionuclide is present, the absorption curve is nearly a straight line until it tails into the constant background count rate of the instrument, which in most cases includes the gamma rays from the source. Visual inspection of the point at which the beta activity is not detectable above the total background gives an approximate thickness value, although this is usually low. Better results can be obtained by subtracting the background from the total absorption curve, as shown in Fig. 9, to obtain the 220-mg/cm ^{2} component. The point at which the straight-line extrapolation intersects the count-rate error of the background is the absorber thickness value. The maximum beta energy can then be obtained from the range energy relation curve (Fig. 10). If the lower energy region of the curve deviates from the straight line extrapolation to zero absorber thickness, a second component is indicated. The values on the extrapolated line subtracted from the second curve produced the 40-mg/cm ^{2} component. The sum of the count rate for both components at zero absorber thickness is the true count rate.

32.3 The best absorption curve is obtained with the absorber against the detector and the source positioned for minimum clearance of the thickest absorber to be used. The source must not be moved after the first count rate, which is obtained with no absorber between the source and the detector. The activity of the source should be close to the maximum allowed by the resolving time of the instrument. Absorption coefficients given in the literature and half-thickness data utilize the initial portions of the absorption curve and are not as accurate as the total absorption determination. More accurate methods for determination of the beta particles' range, from absorption curves, are the comparison methods of Feather **(23)** and Harley, et al **(31).**

32.4 Many liquid scintillation counting systems provide an output suitable for accumulation and storage of beta spectra of known maximum energy. To obtain the most accurate beta maximum energy measurement, the pulse height-energy relationship of the system must be calibrated **(32).** With the aid of this relationship, the pulse height distribution of the sample may be converted to a Kurie plot **(33)** and extrapolated to obtain maximum beta energy. Excellent agreement with beta-energy spectrometry values has been obtained with this technique **(34).** A Kurie plot is shown in Fig. 11.

33. Calculation

33.1 Results may be expressed in observed counts per minute per millilitre for comparing relative activities of a group of samples as in tracer experiments.

~~$C = \frac{1}{V}\frac{N}{t} - \frac{N_B}{t_B}$~~ (24)

$$R_n = \frac{1}{VY}\left(\frac{C}{t} - \frac{C_b}{t_b}\right) \tag{22}$$

Cb tb V Rn

where:

~~N~~ C = number of counts accumulated,
t = sample counting period, s,
~~N_B~~ C_b = number of background counts accumulated,
~~t_B~~ t_b = background counting period, s,
V = volume of initial sample, mL, and
~~C~~ R_n = net counts per second per millilitre (cps/mL).

33.2 Results may also be expressed in terms of ~~equivalent cesium-137~~ equivalent ^{137}Cs activity, using the empirical efficiency determined for the reference standard:

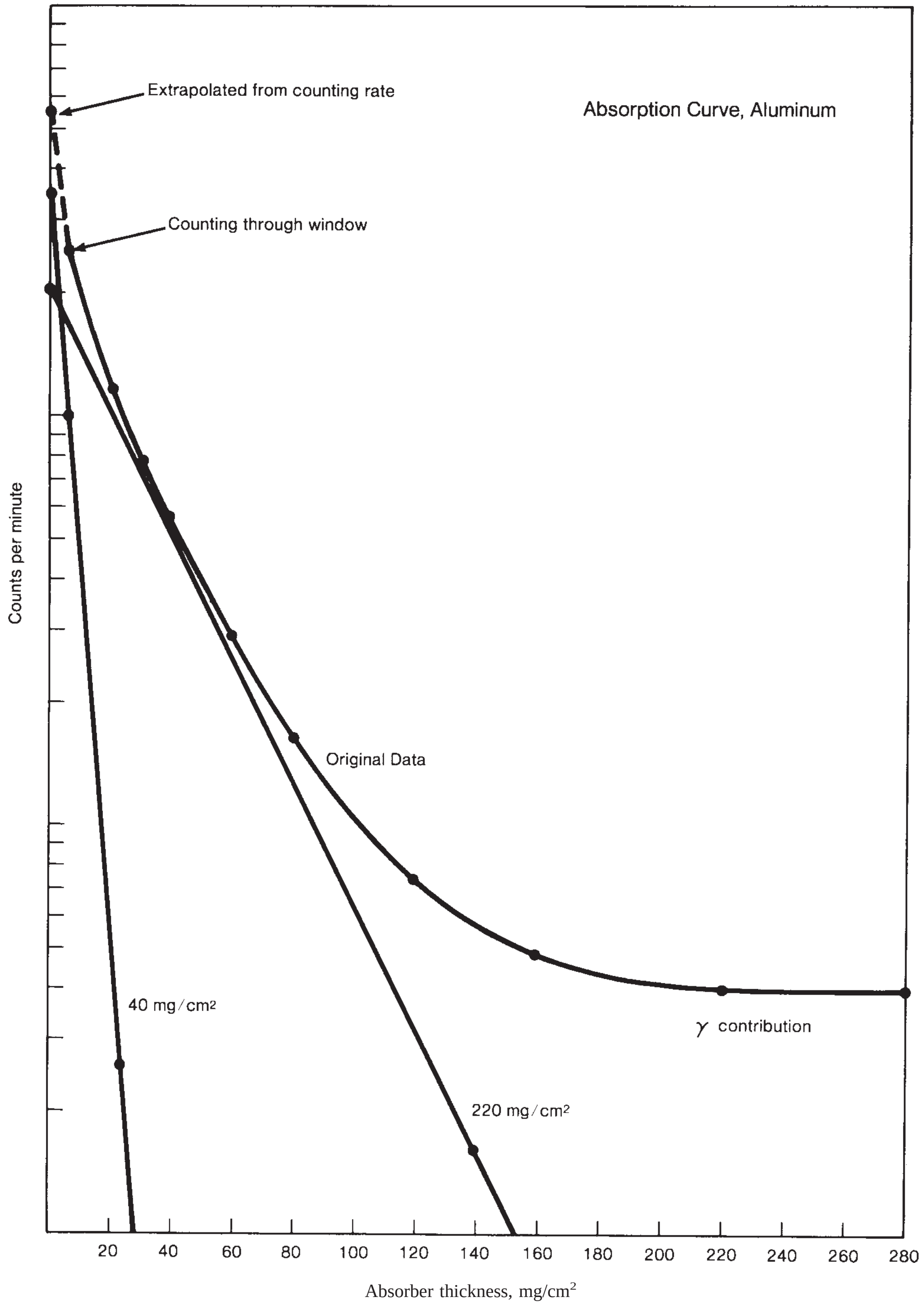


FIG. 9 Absorption Curve

~~$D_{Cs} = C/E_{Cs}$~~ (23)

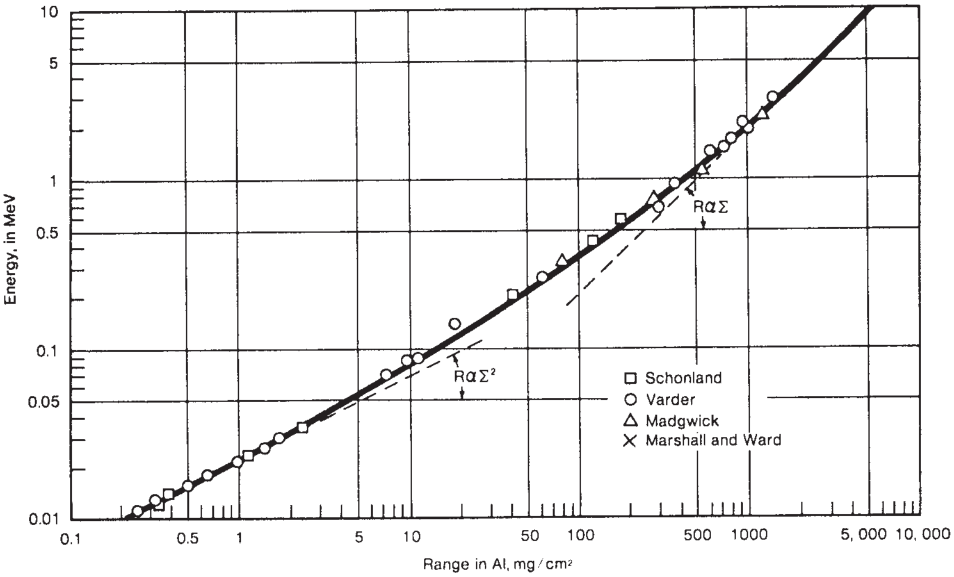
$$A_{a,Cs} = R_{n,v}/E_{Cs} \qquad (23)$$

where:

~~C~~ $R_{n,v}$ = net counts per second per millilitre (cps/mL), and

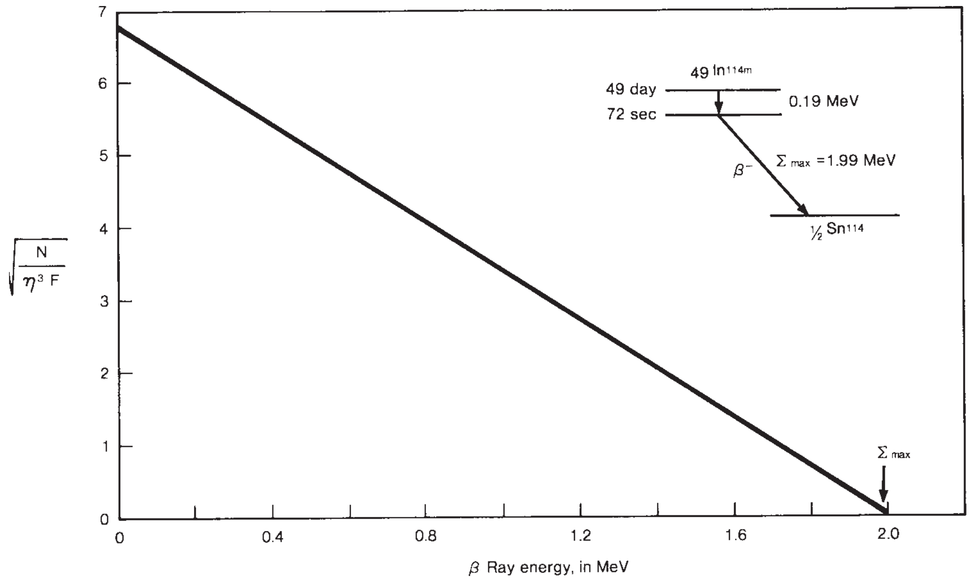
E_{Cs} = efficiency for counting cesium-137 beta particles plus conversion electrons (count rate ÷ emission rate).

33.3 If it is known that only one nuclide is present, its disintegration rate may be determined by use of the efficiency factor measured with a reference standard of the same nuclide. The results may be calculated as follows:



NOTE 1—Experimental values by several observers on monoenergetic electrons are shown. For monoenergetic electrons, the range coordinate refers to the extrapolated range. For continuous beta-ray spectra, the energy coordinate refers to the end-point energy *E* ..., and the range coordinate becomes the maximum range. The smooth curve represents the empirical relationship, developed by Katz and Penfeld **(36).**

FIG. 10 Empirical Range-Energy Relationship for Electrons Absorbed in Aluminum



NOTE 1—Note especially that *N* means *N* (*n*), the number of beta rays in a momentum interval, Δ, of constant size. The horizontal coordinate is the kinetic energy, *E*, which corresponds to the midpoint of the momentum interval $n + \Delta_n$. When spectral data give a straight line, such as this one, the *N* (*n*) is in agreement with the Fermi momentum distribution. The intercept of this straight line, on the energy axis, gives the disintegration energy *E* max (E_s), if the rest mass of the neutrino is zero **(37).**

FIG. 11 Kurie Plot of the Beta Spectrum in ^{114}In

~~$$D = \frac{1}{EV_Y}\left[\frac{N}{t} - B\right] \quad (26)$$~~

$$A_a = \frac{1}{EV_aY}\left[\frac{C}{t_a} - C_b\right] \quad (24)$$

CbAa E Va

where:

~~N~~ C = number of counts accumulated,

~~B~~ C_b = background in counts per second (cps),
~~D~~ A_a = disintegration rate per second per millilitre (Bq/mL),
E = efficiency of counter for the specific nuclide (counts÷ disintegrations),
V_a = volume of test specimen, mL,
Y = recovery of species (unity in cases where complete recovery is assumed), and
t_a = count period, s.

~~33.4 If it is desired that the activity be expressed in units of microcuries, the following expression is used:~~

~~$A = D/(3.7 \times 10^{4})$ (27)~~

~~where:~~
~~A = concentration, µCi/mL,~~
~~D = disintegration rate per second per millilitre (Bq/mL), and~~
~~3.7×10^{4} = conversion factor, disintegration per second per microcurie.~~

GAMMA COUNTING

34. Scope

34.1 This practice covers the measurement of gross gamma radioactivity of water. Since gamma radiation is a penetrating form of radiation, it can be used for samples of any form and geometry as long as standards of the same form are available and are counted at the same geometry to calibrate the detector. Because of this penetrating nature, small variations in sample density or sample thickness are usually not significant and gamma counting is the preferred method in general radiochemical work. When a standard cannot be obtained in the matrix being counted, a correction for the different absorption in the matrices must be made.

34.2 Since different nuclides emit distinct and constant spectra of gamma radiation, the use of an energy-discriminating system provides identification and measurement of all the components present in a mixture of radioactivity. This technique is covered in Test Method D 2459. Gamma counting and gamma spectrometry are applicable to levels of about 0.4 ~~dps~~ Bq or above. General information on gamma-ray detectors and gamma counting is covered in the literature **(13, 23)**.

35. Summary Of Practice

35.1 Gamma counting is generally carried out using solid detectors since a gas-filled detector will not provide adequate stopping power for energetic gammas. In solids such as NaI(Tl) or CsI, the gammas interact by excitation of atoms and energy is transferred to orbital electrons and then released as light photons when the orbits are refilled. The scintillations are easily detected and amplified into useable electrical pulses by a multiplier phototube. The NaI(Tl) detector is the recommended detector for gross gamma counting.

35.1.1 In semiconductor detectors such as Si(Li) and ~~Ge(Li),~~ HPGe, the gamma photons produce electron-hole pairs and the electrons are collected by an applied electrical field. A charge-sensitive preamplifier is used to detect the charge transferred and produce a useable electrical pulse. The semiconductor detectors are widely used in gamma spectrometry (see Test Method D 2459) and are not reviewed in this practice.

35.2 The output pulses from the multiplier phototube or preamplifier are directly proportional to the amount of energy deposited, which could either be total and included in the photopeak, or fractional and included in the continuum or escape peaks, in the detector by the incident photon. The pulses may be counted using a scaler or analyzed by pulse height to produce a gamma-ray spectrum.

35.3 Gamma photons interact with the detector by three distinct processes. The photoelectric effect results in complete absorption of the photon energy and produces the full energy or photopeak shown. The Compton effect results in a particle absorption of the photo energy and a scattered photon of lower energy results. The scattered photon carries energy away and the Compton continuum shown in Fig. 12 results. The third interaction is pair production, which occurs at energies above 1.02-MeV and results in the disappearance of the photon when an electron-positron pair is produced. The electron and positron give up their kinetic energy to the detector and the resulting electron joins the electron population of the detector; the positron, however, is annihilated in combining with an electron and produces two gamma photons of 0.511-MeV each. One or both of the 0.511-MeV photons may escape from the detector without interacting and the "single escape" and "double escape" peaks shown in Fig. 13 result. The Comptons, from a higher energy photon, always present an interference problem in the counting of gamma photons and appropriate corrections must be made for this effect. Pair production can also be considered as an interference since the escape peaks may have an energy equal to the lower energy gamma of interest.

35.4 The change of the absorption coefficient with gamma energy results in a wide variation of detection efficiency. The detection efficiency falls rapidly as gamma energy increases for a fixed size of detector. Two other important effects are seen as a result of the variation of the absorption coefficient; firstly, low-energy photons may be absorbed in massive samples, such as large bottles of water, and erroneous results may be obtained. A similar absorption effect is seen in ~~Ge(Li)~~ HPGe systems where the can around the detector acts as an absorber for very low-energy gammas and the efficiency passes through a maximum usually around

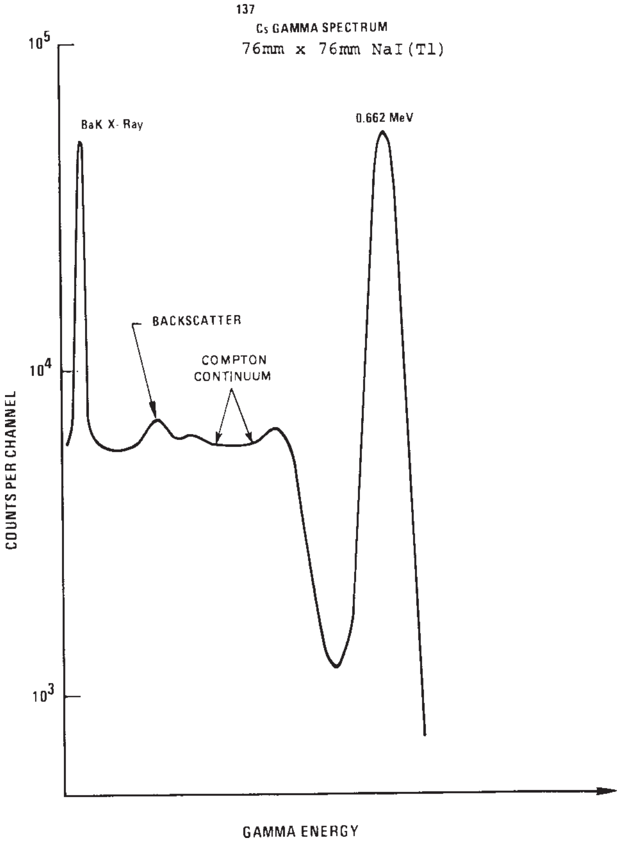


FIG. 12 Compton Continuum

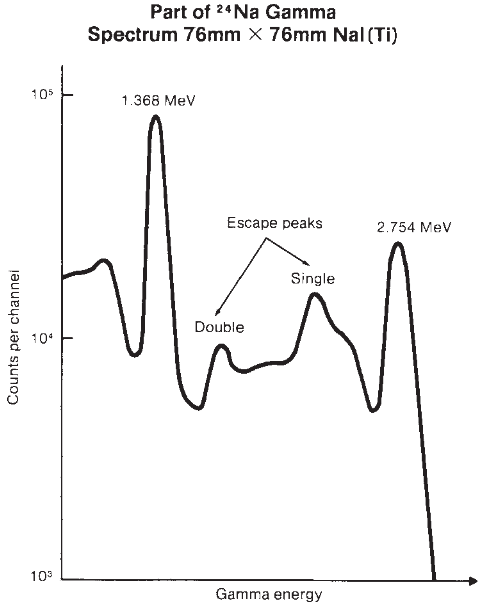


FIG. 13 Single and Double Escape Peaks

100~~K~~ keV. The second result is that for low-energy gammas a thin detector may be as efficient as a much thicker one since the low-energy gammas are easily stopped in the thin detector.

35.5 Because of this variation in efficiency and the possible interferences from other activities, gross gamma counting is only reliable when used to compare standards and samples of the same nuclide. The use of gross gamma monitoring systems should be avoided when possible and, in all cases, proper allowance must be made for the lack of accuracy.

36. Interferences

36.1 The natural background present at all locations is detected very efficiently by gamma detectors and presents a significant interference that must be reduced by the use of shielding. Low-radioactivity-level lead or steel should be used for a 76 by 76-mm NaI(Tl) detector. Approximately 102-mm of lead or 150-mm of steel shielding produces an acceptable background for most work. Details of shield designs are given by Heath **(35).** Lead shields are a source of X-rays when high-activity samples are counted. This X-ray emission can be reduced in amount and energy by using a graded liner, such as 1.6-mm of cadmium and 0.4-mm of copper, the copper being nearer the detector. The cadmium strongly absorbs the lead X-rays and in turn emits its own X-rays at a much lower energy. These are strongly absorbed by the copper. Any residual copper X-rays are usually below the energy level of interest.

36.2 The "Compton" and "Pair Production" effects discussed in 35.3 can be very significant interferences and must be corrected for.

36.3 At high count rates "random sum peaking" may occur. Two absorptions may occur within the resolving time of the detector and are summed and seen as one pulse. For a detector of resolving time, t, and a count rate of A counts per unit time, the time window available for summing is $2\,At$ (since the count summed could occur as early as t before or as late as t after the other count) and the probability of another count at any time is simply A. Therefore, the sum count rate will be $2A^2t$ in unit time. Random summing is strongly dependent on the count rate A and, if summing occurs, it can be reduced by increasing the sample to detector distance.

36.3.1 Well counters that have very high efficiencies are prone to summing since, for a given source strength, the count rate is higher than for a detector of lower efficiency. For moderate and high-source strengths, the trade-off is a poor one and the well counter is best suited for low-level work where its high efficiency is an important advantage.

36.4 Cascade summing may occur when nuclides that decay by a gamma cascade are counted. Cobalt-60 is an example; 1.17-MeV and 1.33-MeV from the same decay may enter the detector and be absorbed, giving a 2.50-MeV sum peak. Cascade summing may be reduced and eventually eliminated by increasing the source-to-detector distance.

36.5 The resolution of a gamma detector is the effective limit to its utility even when complex data-reduction methods are used. A typical 76 by 76-mm NaI(Tl) detector will give full widths at half maximum (FWHM) of approximately 60 keV at 662-keV gamma energy and approximately 90 keV at 1.33-MeV gamma energy.

37. Apparatus

37.1 *Sodium Iodide Detector Assembly* —A cylindrical 76 by 76-mm NaI detector is activated with about 0.1 % thallium iodide, with or without an inner sample well, optically coupled to a multiplier phototube, and hermetically sealed in a light-tight container. The NaI(Tl) crystal should contain less than 5 ppm of potassium and be free of other radioactive materials. In order to establish freedom from radioactive materials, the manufacturer shall supply a gamma spectrum of the background of the detector between 0.08 and 3.0 MeV. The resolution of the detector for ~~the cesium-137~~ the ^{137}Cs gamma at 0.662-MeV should be less than 60 keV FWHM or less than 9 %.

37.2 *Shielding* for the detector shall be constructed as needed from low-radioactivity-level lead or steel. A thickness equivalent to 102 mm of lead usually provides adequate shielding. If the shield is made of lead a graded liner should be used (35.1) unless the shield-to-detector distance is more than 250 mm.

37.3 *High-Voltage Power Supply*—500 to 2000 V dc regulated to 0.1 % with a ripple of not more than 0.01 %.

37.4 *Preamplifier*— Linear amplifier system to amplify the output from the multiplier phototube to a maximum output of 10 V.

37.5 *Analyzer with Scaler and Timer* —A single-channel discrimination system will accept all or any part of the output from the amplifier and pass it to the scaler. Any pulses lying outside the preset limits are rejected. The lower limit is usually referred to as the "threshold" and the difference between the two limits is the "window."

37.6 *Sample Mounts and Containers* may consist of any reproducible geometry ~~container that is commercially available.~~ container. Other considerations are cost, ease of use, disposal, and effective containment of radioactivity for the protection of the workplace and personnel from contamination.

37.7 *Geometry Control System*—A system of shelves or supports for the various sample containers that allows the user to place a sample in any of several preset and reproducible geometries.

37.8 *Beta Absorber*— A beta absorber of 3 to 6 mm of aluminum, beryllium, or poly(methyl methacrylate) should completely cover the upper face of the detector to prevent betas from reaching the detector.

38. Energy Efficiency Relationship

38.1 Because of the rapid falloff in gamma absorption as gamma energy rises, the detection efficiency shows a similar effect, and Fig. 14 shows the efficiency versus gamma energy plot for a 76 by 76-mm NaI(Tl) detector. The portion of the curve at low energy shows that as the absorption coefficient increases geometry becomes the limiting factor. The maximum efficiency seen is well below 50 % due to the presence of the beta absorber and the containment of the detector. The 76 by 76-mm NaI(Tl) detector is the most widely used size and a large amount of data are available in the open literature on the use of this size and results obtained. Heath **(35)** has written a comprehensive review and supplied many gamma-ray spectra in both graphical and digital form.

38.2 Other sizes of detectors may be used. However, the following should be noted:

38.2.1 Smaller detectors, such as 38 by 38-mm, will give efficiencies that are low and fall off more rapidly as gamma energy increases. Small or thin detectors are useful for the measurement of low-energy gammas since they are less responsive to high-energy gammas and the interference from Compton effects is reduced.

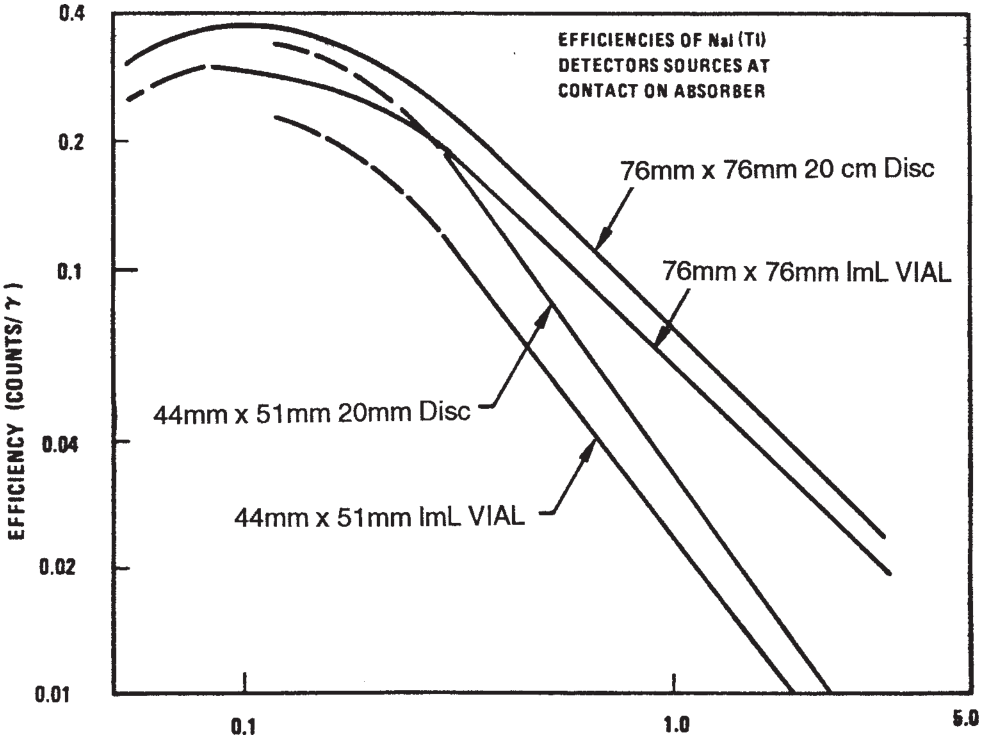


FIG. 14 Efficiency versus Gamma Energy

38.2.2 Larger detectors will give higher efficiencies and less fall off as gamma energy increases. Larger detectors are useful for situations where the highest attainable efficiency is desired and for the assembly of complete absorption detectors. The increase in efficiency is accompanied by an increased background count rate and an increase in the probability of summing in the detector.

38.2.3 Well detectors will give very high efficiencies, up to about 80 % for low- and moderate-energy gammas. The well detector is useful for low levels of activity and the background of a well detector is essentially the same as that of a plain cylindrical detector of the same overall dimensions. Summing becomes a definite problem at high activities since both random and cascade summing result from the high efficiencies and the high geometry of the well detector.

39. Calibration and Standardization

39.1 Calibrate the system for energy or gain by using~~ at least~~ two different sources of different gamma energies. Cesium-137 at~~ 662 keV and cobalt-60~~ 0.662-keV and ^{60}Co at 1.17-MeV and 1.33-MeV are generally adequate. Use these sources to establish two parameters; (*1*) the required MeV or keV per volt of amplifier output, and (*2*) the condition that 0 MeV is equal to 0-V output. Carry out subsequent efficiency calibrations with the same gain settings.

39.2 Obtain efficiency calibrations by counting known amounts of single nuclides in the geometry and matrix that will be used for the unknown samples. Standards are available from the National Institute of Standards and Technology and other sources. Take extreme care in the preparation of subsamples of standards and, whenever possible, use gravimetric dilution and division techniques. Obtain the threshold and window levels used in volts using the energy calibration data obtained previously.

39.3 In comparing a standard and samples of the same nuclide, accurate results are possible. When an arbitrary standard is used to estimate the activity of a mixture of nuclides, the data are not accurate since the real detection efficiency varies with energy. Because of this gross gamma count, data obtained on mixtures of nuclides are only useful as an indication of approximate activity. Such data can be used for sample screening to select aliquot sizes for gamma spectrometry, comparison of process or waste samples with each other, and as a general screening for liquid wastes.

39.4 Report data obtained using an arbitrary standard for a mixture of nuclides as being based on a "calibration using nuclide-X" or as being "relative to nuclide-X." Data obtained by using this technique *should not be reported*~~*as dpm or Bq*~~ *in units of Bq without qualification*.

40. Source Preparation

40.1 Prepare samples in an identical manner to the standards and count in the same geometry under identical conditions. For gamma counting, samples may be in liquid, solid, or gaseous form contained in suitable containers.

40.2 The evaporation of liquid samples to dryness before counting is not necessary. However, samples that have been evaporated to dryness for gross beta counting can also be gamma counted. Evaporation may also be used as a sample concentration technique, especially with pure water such as evaporator condensates, where a large sample may be evaporated to small volume and counted with a higher efficiency.

40.3 Gaseous samples can be collected in any type of gas container but take care to ensure that all the gas containers of a particular type have a constant wall thickness. Wall thickness variation can be a significant problem in counting radioactive xenons that have gammas of 80 and 250-keV energy.

41. Calculation

41.1 Results may be expressed in observed counts per minute per millilitre for comparing relative activities of a group of samples as in tracer experiments:

~~$C = \frac{1}{V}\frac{N}{t} - \frac{N_B}{t_B}$~~ (28)

$$R_n = \frac{1}{VY}\left(\frac{C}{t} - \frac{C_b}{t_b}\right) \quad (25)$$

Cb tb V Rn

where:
~~N~~ C = number of counts accumulated,
t = sample counting period, s,
~~N_B~~ C_b = number of background counts accumulated,
t~~$_B$~~$_b$ = background counting period, s,
V = volume of initial sample, mL, and
~~C~~ = net counts per second per millilitre (cps/mL).
R_n

41.2 Results also may be expressed in terms of ~~equivalent cesium-137~~ equivalent ^{137}Cs activity, using the empirical efficiency determined for the reference standard:

~~$D_{Cs} = C/E_{Cs}$~~ (29)

$$A_{a,Cs} = R_{n,v}/E_{Cs} \quad (26)$$

where:
~~C~~ $R_{n,v}$ = net counts per second per millilitre (cps/mL), and ~~ECs~~ ~~=plus conversio~~

41.3 If it is known that only one nuclide is present, its disintegration rate may be determined by use of the efficiency factor measured with a reference standard of the same nuclide. The results may be calculated as follows:

~~$D = \frac{1}{EV_Y}\left[\frac{N}{t} - B\right]$~~ (30)

$$A_a = \frac{1}{EV_aY}\left[\frac{C}{t_a} - C_b\right] \quad (27)$$

CbAa Va

where:
~~N~~ C = number of counts accumulated,
~~B~~ = background in counts per second (cps),
C_b
~~D~~ = disintegrations per second per millilitre (Bq/mL),
A_a
E = efficiency of counter for the specific nuclide (cps/Bq),
V_a = volume of test specimen, mL,
Y = recovery of species (unity in cases where complete recovery is assumed), and
t_a = count period, s.

~~41.4 If it is desired that the activity be expressed in units of microcuries, use the following expression:~~

~~$A = D/(3.7 \times 10^4)$~~ ~~(31)~~

~~where:~~
~~A = concentration, µCi/ml,~~
~~D = disintegration rate per second per millilitre (Bq/mL), and~~
~~3.7×10^4 = conversion factor, disintegrations per second per microcurie.~~

42.

~~43.~~ Keywords

4~~3~~2.1 alpha counting; beta counting; gamma-ray counting; radioactivity detection; radioactivity measurement; water

REFERENCES

(**1**) Pate, B. D., and Yaffe, L., “Disintegration Rate Determination by 4π Counting,” *Canadian Journal of Chemistry,* Vol 33, 1955, pp. 15, 610, 929, and 1656.

(**2**) Blanchard, R., Kahn, B., and Birkoff, R. D., “The Preparation of Thin, Uniform Radioactive Sources by Surface Absorption and Electrodeposition,” *Health Physics,* Vol 2, 1960, p. 246.

(**3**) Hallden, N. A., and Fisenne, I. M., “Minimizing Self-Absorption in 4π-8 counting, “*International Journal of Applied Radiation and Isotopes,* Vol 14, 1963, p. 529.

(**4**) Merritt, J. S., Taylor, J. G. V., and Campion, P. J., “Self-Absorption in Sources Prepared for 4π-β Counting,” *Canadian Journal of Chemistry,* Vol 37, 1956, p. 1109.

(**5**) Gunnick, R., Colby, L. J., and Cobble, J. W., *Analytical Chemistry,* Vol 31, 1959, p. 796.

(**6**) Friedlander, G., Kennedy, J. W., and Miller, J. M., *Nuclear and Radiochemistry,* John Wiley and Sons, New York, 1964, 2nd ed.

(**7**) Price, W. J., *Nuclear Radiation Detection,* McGraw-Hill, New York, N.Y., 1964, 2nd ed.

(**8**) Campion, P. J., Taylor, J. G. V., and Merritt, J. S., “The Efficiency Tracing Technique for Eliminating Self-Absorption Errors in 4π Beta Counting,” *International Journal of Applied Radiation and Isotopes,* Vol 8, 1960, p. 8.

(**9**) Curtis, M. L., Heyd, J. W., Olt, R. G., and Eichelberger, J. F., *Nucleonics,* Vol 13, May, 1955, p. 38.

(**10**) Watt, D. E., and Ramsden, O., *High Sensitivity Counting Techniques,* The MacMillan Company, New York, N.Y., 1964.

(**11**) Crouthamel, C. E., Adams, F., and Dams, R., *Applied Gamma-Ray Spectrometry,* Pergamon Press, New York, N.Y., 2nd ed., 1970.

(**12**) Nielson, J. M., *Gamma-Ray Spectrometry in Physical Methods of Chemistry,* A. Weissberger and B. W., Rossiter, eds. Vol. 1, Part III D, Chap. X, John Wiley and Sons, Inc. New York, N.Y., 1972.

(**13**) Perkins, R. W., *Nuclear Instruments and Methods,* 1965, p. 33. Wogman, N. A. and Perkins, R. W., *ibid,* 1968.

(**14**) Cooper, J. A., Ranticelit, L. A., Perkins, R. W., Haller, W. A., and Jackson, A. L.,“ An Anti-Coincidence Shielded Ge(Li) Gamma-Ray Spectrometer and Its Application to Neutron Activation Analyses”, *Report BNWL-SA-2009,* Pacific Northwest Laboratory, Richland, Wash., 1968.

(**15**) *Metrology of Radionuclides, Proceedings of a Symposium,* Oct. 14–16, 1959, International Atomic Energy Agency, Vienna.

(**16**) Nielsen, J. M., and Kornberg, H. A.,“ Multidimensional Gamma-Ray Spectrometry and Its Use in Biology,” *Radioisotope Sample Measurement Techniques in Medicine and Biology, Proceedings of a Symposium,* May 24–28, 1965, International Atomic Energy Agency, Vienna, p. 3.

(**17**) Lapp, R. E., and Andrews, H. L., *Nuclear Radiation Physics,* Prentice Hall, New York, 1948. Englewood Cliffs, N.J., 2nd ed., 1954.

(**18**) Jarrett, Allen A., “Statistical Methods Used in the Measurement of Radioactivity with Some Useful Graphs and Nomographs,” *AECU 262.*

(**19**) Bell, C. G., and Hayes, F. N., *Liquid Scintillation Counting,* Pergamon Press, New York, 1958.

(**20**) Birks, J. B., *The Theory and Practice of Scintillation Counting,* Pergamon Press, New York, 1964.

(**21**) Bransome, E., Jr., ed. *The Current Status of Liquid Scintillation Counting,* Greene and Stralton, New York, 1970.

(**22**) *Environmental Radioactivity Surveillance Guide,* ORP/SID 72-2 USEPA 1972.

(**23**) Friedlander, G., Kennedy, J. W., and Miler, J., *Nuclear and Radiochemistry,* John Wiley and Sons, Inc., New York, NY, 2nd ed., 1964. Overman, R. T., and Clark, H. M., *Radioisotope Techniques,* McGraw-Hill Book Co., Inc., New York, NY, 1960. Price, W. J., *Nuclear Radiation Detection,* McGraw-Hill Book Co., Inc., New York, NY, 1964. Flynn, K. F., Glendenin, L. E., and Prodi, V. *Absolute Counting of Low Energy Beta Emitters Using Liquid Scintillation Counting Techniques in Organic Scintillators and Liquid Scintillation Counting,* D. L. Horrocks and Chim-Tzu Peng, eds., Academic Press, New York, 1971, p. 687.

(**24**) Sill, C. W., and Olson, D. G., “Sources and Prevention of Recoil Contamination of Solid-State Alpha Detector,” *Analytical Chemistry,* Vol 42, 1970, p. 1956.

(**25**) Puphal, K. W., and Olson, D. R., “Electrodeposition of Alpha Emitting Nuclides from a Mixed Oxalate-Chloride Electrolyte:, *Analytical Chemistry,* Vol 44, 1972.

(**26**) Mitchell, R. F., “Electrodeposition of Actinide Elements at Tracer Concentrations,” *Analytical Chemistry,* Vol 32, 1960, pp. 326–328.

(**27**) Bell, C. G., and Hayes, F. N., *Liquid Scintillation Counting,* Pergamon Press, New York, NY, 1958.

(**28**) Moghissi, A. A., “Low Level Counting by Liquid Scintillation,” *Organic Scintillators and Liquid Scintillation Counting,* Academic Press, New York, NY, 1971.

(**29**) Horrocks, D. L., “Alpha Particle Energy Resolution in a Liquid Scintillator,” *Review of Scientific Instruments,* Vol 55, 1964, p. 334.

(**30**) Bogen, D. C., and Welford, G. A., “Application of Liquid Scintillation Spectrometry for Total Beta and Alpha Assay,” *Proceedings of the International Symposium of Rapid Methods for Measuring Radioactivity in the Environment, LAEA-SM-14/3,* 1971, p. 383.

(**31**) Harley, J. H., Hallden, N. A., and Fisenne, I. M., “Beta Scintillation Counting with Thin Plastic Phosphors,” *Nucleonics,* NUCLA, Vol 20, 1962, p. 59.

(**32**) Flynn, K. F., Glendenin, E., Steinberg, E. P., and Wright, P. M., “Pulse Height-Energy Relations for Electrons and α-Particles in a Liquid Scintillator,” *Nuclear Instruments and Methods,* Vol 27, 1964, p. 13.

(**33**) “Tables for the Analyses of β Spectra, *National Bureau of Standards Applied Mathematics Series Reports No. 13,* U. S. Government Printing Office, Washington, DC, 1952.

(**34**) Flynn, K. F., and Glendenin, L. E., “Half-Life and β-Spectrum of Rubidium-87,” Physics Review, Vol 116, 1959, p. 744.

(**35**) Heath, R. L.,“ Scintillation Spectrometry Gamma-Ray Spectrum Catalog” *IDO 16880,* and *ANCR-1000.*

(**36**) Katz and Penfeld, “Reviews,” *Modern Physics,* Vol 24, 1952, p. 28.

(**37**) Lawson and Cork, *Physics Review,* Vol 57, 1940, p. 982.

(**38**) Currie, L. A., “Limits for Qualitative Detection and Quantitative Determination,” *Analytical Chemistry* , Vol 40, No. 3, 1968, pp. 586–593.

(39) Paar, J. G., and Porterfield, D. R., “Evaluation of Radiochemical Data Usability,” *Report ES/ER/MS-5 (OSTI DE97003857),* Lockheed Martin Energy Systems, Oak Ridge, TN, April 1997.

(40) Strom, D. J., and MacLellan, J. A., “Evaluation of Eight Decision Rules for Low-Level Radioactivity Counting,” *Health Physics,* Vol 81, 2001, p. 27.

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