4.5.2 Radiometrology

A-01-R

ALPHA RADIOASSAY

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CALIBRATION OF ALPHA-EMITTING RADIONUCLIDE SOLUTIONS AND SOURCES

A. Application.

The methods for primary and secondary calibrations of α-emitting solutions and sources are described for detection systems in use at EML.

B. Description of the system.

Primary calibrations of α-emitting radionuclide solutions and sources are performed at EML with a 2π windowless (internal) gas flow proportional detection system. A commercially obtained stainless steel counter with a three-position turntable and gas flow indicator (a bubbler) is capable of accepting samples up to 5.08 cm (2 in) diameter. A heavy walled cylindrically shaped active volume contains a loop anode of stainless steel wire. An EML designed and built preamplifier is connected directly to the top of the detector. The preamplifier acts as an impedance matching device for pulses from the detection volume to an electronic scaler. High voltage from a 0-5000 V regulated power supply is applied to the anode through a cable connection on the preamplifier. The signal from the anode passes through the preamplifier and a cable into an electronic scaler. The duration of the measurement interval is controlled with an electronic timer. Methane (99.99% chemically pure) is used as the counting gas.

Any source to be measured must be of a conducting material. In practice, all sources measured at EML in this system are on metal backings, usually platinum, stainless steel or aluminum.

C. Traceability to the National Institute of Standards and Technology (NIST).

Each day the detection system is used, its operating characteristics are checked with a NIST standard reference material (SRM) U_3O_8 source. After thoroughly flushing the system with the counting gas, a voltage plateau is determined with the NIST source. Generally, the plateau is 500-600 V in length at applied voltages of 2300-2900 (+). The slope of the voltage plateau is checked and should be < 1% per 100 V with methane as the counting gas. The operating voltage selected is 100 V below the upper "knee" of the plateau. The NIST source is measured for 2000 sec, sufficient for a 1 s Poisson standard deviation (SD) of the measurement of < 0.5%. The background count rate is determined at the same operating potential for 2000 sec. The background count should be 4-5 counts or about 0.003 counts sec^{-1} (cps). The net count rate obtained by subtracting the background count rate from the total count rate of the standard source measurement is compared with the certified NIST value. (Total count rate refers to the [sample + background] count rate.) The NIST certified value is given in terms of α particles sec^{-1} in a 2π configuration. This means that the α backscatter factor is part of the certified value and no correction needs to be made for this source characteristic. The EML value obtained must agree with the NIST certified value (within the error of the measurement) before other sources are measured.

As a further check of this procedure, when NIST α-emitting SRM sources are purchased, they are treated as unknowns and measured as described above. The value obtained at EML is then compared with the certified value.

Failure to obtain a flat or a long voltage plateau or failure to agree with the certified source value are immediate indicators of problems in the detection system which must be remedied before proceeding with additional measurements.

D. EML source preparation.

A system for the primary calibration of α-emitting solutions and sources is maintained at EML because all the nuclides required for in-house programs may not be available as NIST SRMs at any given time and it is cost effective. Both NIST SRM solutions and sources are initially expensive and the cost of recalibration by NIST is often equal to or more than the original cost. Since all EML α-emitting solutions and sources are recalibrated at least once a year, the cost of NIST recertification would be prohibitive.

Electrodeposited sources of α-emitting radionuclides are prepared on 17 mm diameter virgin platinum discs. These sources, once calibrated, are used as standards to determine the detection efficiencies of α-scintillation counters and solid-state α-spectrometry systems. In general, the activity of these sources is 15-20 Bq (450 pCi or 1000 dpm). The procedure for obtaining a "working standard" source is to measure the NIST SRM U_3O_8 source and background as described above, obtain a voltage plateau for the source to be calibrated, and measure the source at the proper plateau voltage for 2000 sec. The net count rate of the source is converted to an activity unit using the detection efficiency of 52%. A Poisson error term is calculated and attached to the activity value.

The calibration of an α-emitting solution differs from that for electrodeposited sources only in the method of source preparation. It is the practice at EML that sources from the solution to be calibrated be prepared in triplicate. The source mounts are 24 mm diameter virgin platinum discs. The solution to be calibrated must be essentially carrier-free. The activity of the solution should be on the order of 160-1600 Bq g^{-1}. To minimize α absorption in the source, a weighed amount of a Teflon suspension is transferred to the disc from a polyethylene transfer pipette (see Specification 7.11). About 30 µg of Teflon beads are delivered in the 0.1-g aliquot. A weighed aliquot of the solution to be calibrated is dispensed from the transfer pipette directly into the Teflon suspension bead. As a check on possible self-absorption in the dried source, aliquot weights are varied from 0.1-0.2 g in the triplication procedure. The sources are slowly dried near a heat lamp.

After the 2π proportional detection system has been checked as described above, the first source is heated in an alcohol burner flame just until the Teflon vaporizes. This drives off any residual waters of hydration which could cause self-absorption in the source. The flamed source is immediately placed in the methane atmosphere. A voltage plateau is obtained and the source is measured as described above. This procedure is repeated for the remaining sources. The activity and Poisson counting error are calculated for each source and corrected for the aliquot weight to obtain the activity g^{-1} of solution. A Gaussian mean and SD are calculated from the triplicate source measurements. The Gaussian SD is usually < 1%.

Dilutions of the calibrated solution may then be prepared by weight for use in various EML programs.

ALPHA SCINTILLATION MEASUREMENTS

A. Application.

The procedure was developed for α measurements of air dust samples and chemical precipitates. Perhaps its largest application has been in the determination of ^{222}Rn and ^{220}Rn concentrations from air filter samples. Routine applications include the determination of the α nuclide concentrations of low-level solutions and measurement of total α activity of radiochemically separated samples and investigations of materials for commercial uses.

The advantages of the use of ZnS(Ag) on Mylar phosphor discs were enumerated by Hallden and Harley (1960) and are paraphrased here.

Samples are measured with uniform high efficiency, since the sample is in direct contact with the phosphor and photomultiplier tube (PMT).

The phosphor sees only the sample and the face of the PMT, yielding a lower background than other α-scintillation systems and proportional counters.

Samples are completely enclosed, readily handled and stored without loss of sample integrity or cross contamination.

Counter maintenance is negligible, since the system cannot be contaminated under ordinary conditions.

Samples need not be conducting as with internal gas proportional counters.

Filter samples are held flat without curling or buckling producing a uniform counting configuration.

B. Description of the detection systems.

Essentially all α scintillation detection systems use ZnS(Ag) as the phosphor. There are basically two types of α scintillation counters in use at EML for total α measurements, those accommodating samples using ZnS(Ag) on Mylar (Hallden and Harley, 1960; see Specification 7.11), and those with the phosphor applied as a powder to the face of a PMT tube (Curran and Baker, 1948).

The EML α-scintillation counters were designed and built by EML's Instrumentation Division (now the Technical Program Services Division). The EML α-scintillation counters accommodate the 2.54 cm (1 in) diameter nylon ring and disc sample mounting system (see Specification 7.2). The sample to be measured is placed on the nylon disc. A 2.4 cm (15/16 in) diameter die-cut phosphor disc is placed against the sample, a strip of Mylar film (see Specification 7.3) is overlaid and the assembly is locked together with a nylon ring.

A spring-loaded 2.54 cm (1 in) diameter PMT is housed in a light tight, anti-magnetic shield, facing downward. The sample ring and disc assembly is placed in a space in a pullout drawer. The drawer is closed and a knob turned to the "up" position, placing the sample assembly in direct contact with the PMT. An emitter follower network is built on the base of the PMT. The high voltage supply for the PMT is an integral part of the EML Instrumentation Division's double width nuclear instruments modules (NIM) scaler-timer assembly.

It should be noted that the α-scintillation phosphor discs were and are intended for single use, that is, they and most of the samples are disposable as a unit.

C. Calibration of the instruments.

The detection efficiencies of the EML α-scintillation counters are determined by measuring electrodeposited "working standard" sources prepared at EML. The sources are traceable to the NIST through the process described in Section C, Traceability to NIST.

Measurements with single emitter sources with energies ranging from 3-6 MeV show that the detector response is independent of the energy of the α particle. Sources of ^{226}Ra and ^{228}Th in equilibrium with their short-lived progeny were measured in the 2π proportional counter and then mounted and measured in the α-scintillation detection systems. After applying the detection efficiency obtained from the "working standards", the values obtained by α-scintillation counting agreed with those of the 2π proportional counter within the error of the measurements. This implies that the detection efficiencies of the α-scintillation counters are independent up to 9 MeV.

Background count rates are determined for the materials commonly used in EML programs. These include platinum discs and cellulose, polystyrene and glass fiber filters. To determine the "true" background count rates of the systems, so called "nylon

background" measurements are performed in which the phosphor is placed in direct contact with the nylon disc. This is especially useful when assessing the performance of each new batch of phosphors. Typical background count rates for these materials are shown below.

Material	Background Count Rate (cpm)
Nylon	0.001 ± 0.001
Polystyrene filter	0.001 ± 0.001
Cellulose filter	0.002 ± 0.002
Glass fiber filter	0.100 ± 0.010
Platinum disc (17 mm dia.)	0.012 ± 0.002

The background count rates are determined for measurement intervals of 1000 min or more.

The average detection efficiency determined from measurements of a "working standard" source electrodeposited on platinum is 51%. The source is measured twice weekly for a 100-min counting interval.

Quality control charts are maintained for the background count rates and the detection efficiencies of the α-scintillation counters. The means and SDs of each type of measurement are calculated on a monthly basis.

SOLID-STATE ALPHA SPECTROMETRY

A. Application.

The solid-state α-spectrometry systems are used primarily to determine the concentrations of radionuclides in chemically separated samples. Virtually all radiochemical analyses for α-emitting radionuclides are performed with an isotopic tracer. For the transuranic nuclide determinations, the following tracers are used at EML: ^{236}Pu, ^{242}Pu, and ^{243}Am. Tracers used in the determination of naturally-occurring radionuclides are ^{208}Po, ^{209}Po, ^{229}Th and ^{232}U.

The systems are also used to determine the radiopurity of radionuclide solutions.

B. Description of the detection systems.

The solid-state α-spectrometry systems used at EML are divided into two groups of four detectors, one group is devoted to transuranic measurements and the other to natural radionuclide measurements. Both groups have several elements in common. All components of the systems are commercially available except as noted.

Four vacuum chambers are served by a single beltless vacuum pump. The interior diameters of the vacuum chambers allow samples to be placed as far as 5 cm from the detector. The sample holder, which may be moved vertically, has been modified at EML to an open drawer-slide arrangement. The drawer is locked in place and the slide, with a 2.54 cm diameter by 2 mm deep indentation to accommodate the sample mount, is removable. The sample is placed on a mount, the mount is placed in the indentation, and the slide is then replaced in the open drawer arrangement. This sample holder arrangement eliminates problems of source to detector geometry. The two kinds of solid-state detectors in use at EML will be discussed below. Voltage to the detectors is supplied by individual regulated power supplies. Each detector has a charge sensitive preamplifier and a linear amplifier. Signals from the detectors are directed through a router into a 4096 multichannel analyzer. One analyzer serves four detectors with data collection divided into four 1024 channel segments. A hard copy of the data is obtained with a high speed printer.

All α-spectrometry data are reduced "by hand" which ensures immediate and individual review of the spectra. By this practice, difficulties with the spectrometry systems or the samples are quickly noted and remedied.

The solid-state α detectors are operated at about 10 keV per channel, covering an energy range of about 3-13 MeV. By this practice, all α emitters present in a sample can be identified from their energies. (Virtually all α emitters, both natural and artificial are within this energy range.)

Because EML is primarily involved in the measurement of low-level environmental samples, the most common measurement period by α spectrometry is 5000 min. This time unit will be used in describing some of the characteristics of the detection systems.

The solid-state detectors in use at EML for research programs are conventional 500 mm^2 active area silicon surface barrier detectors. The resolution of the detectors, as measured routinely with EML prepared "working standard" sources, is 40-45 keV (FWHM). The systems are calibrated with "mixed α standards" prepared by micro-precipitation with NdF_3 (Hindman, 1983; Sill, 1981) on polystyrene filters. The back-

ground count rates determined with "blank" micro-precipitates in the energy regions of interest are generally 1 to 2 counts 5000 min^{-1}. The emitter mixture of the standards is ^{238,239,242}Pu, ^{244}Cm. The standard and sample measurements are performed at a distance of 1 mm from the lip of the detector housing. At this distance the average detection efficiency is 40%.

The detection efficiencies and resolution obtained with microprecipitated standards were checked against those obtained with electrodeposited standards and no differences were found.

The multichannel analyzers used for α-spectrometry measurements have been thoroughly tested by EML's Instrumentation Division to ensure stability over long measurement periods (5000-10,000 min). The stability of the systems is aided by placing them in a temperature controlled room. It is important to note that the room temperature needs to be stable. It is fluctuations in temperature that cause the detector response to shift or drift.

A commercially available hardware/computer controlled system is used for routine measurements. There are eight vacuum modules, each housing a 500 mm^2 ion implant detector. To accommodate the software requirements of the system, the detectors are calibrated with NdF$_3$ microprecipitated "working standards" containing only two emitters, ^{242}Pu and ^{244}Cm. The resolution of the detectors is about 50 keV FWHM and the detection efficiency about 40%. Background count rates are similar to our research systems.

B. Calibration of the alpha-spectrometry systems.

The detection efficiencies of the α-spectrometry systems are determined by measuring microprecipitated "working mixed standards" prepared at EML. The activity of the standards is determined by α-scintillation counting on systems calibrated with sources traceable to NIST through the process described in Section C, Traceability to NIST.

Measurements with single emitter sources with energies ranging from 3-6 MeV have shown that the energy and detection efficiency responses of silicon surface barrier detectors are constant. Sources of ^{228}Th in equilibrium with its short-lived progeny were measured and the activities of the individual progeny were the same within the error of the measurements. This provided empirical information that the energy response of the

detectors is constant up to 9 MeV and implies that the detection efficiencies are also constant.

It is the practice at EML to measure the background count rate of the detectors each weekend and during the week if the work load permits. Typical background count rates for the detectors are given above. The background count rates of the systems invariably increase with time due to recoil atoms depositing on the face of the solid-state detectors. It has been the experience at EML that the background count rates become unacceptable after two years of use. The only effective method of reducing the background count rates is to place the detectors in a high vacuum provided by a liquid nitrogen cooled diffusion pumping system over a weekend.

Each "working mixed standard" source has a total activity of about 15 Bq. The standards source is measured for 50 min before and after each sample measurement. The standard source measurements provide three important pieces of information: the detection efficiency, the detector resolution, and the energy calibration. Deviations from normal operating conditions in any or all of these characteristics are immediately investigated. These quality control records are very important in establishing the norm for each detector system.

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BETA RADIOASSAY

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CALIBRATION OF BETA-EMITTING RADIONUCLIDE SOLUTIONS

A. Application.

The methods for the primary calibration of emitting radionuclide solutions and routine measurements are described for detection systems in use at EML.

B. Description of the system.

Primary calibrations of β-emitting radionuclide solutions are performed at EML with gas flow 4π β-proportional and 4π β-proportional-4π γ coincidence detection systems. The commercially obtained aluminum proportional counters have a stainless steel wire anode in each half-cylinder. The total interior volume is 50 cm^3. The source mount is an aluminum washer, 3.8 cm in diameter with a 1.8-cm diameter center hole and four 0.3-cm holes in the rim to allow gas to flow between the halves with the source mount in place. The counting gas is 99.99% chemically pure methane, and the flow through the chamber is monitored with a bubbler. An EML designed and built emitter follower preamplifier is connected directly to the anodes. Signals from each half of the chamber are summed prior to further amplification. The amplifier is arranged to deliver triggered pulses to an electronic scaler. High voltage is supplied by a 0-5000 V regulated power supply. The chambers exhibit rather long (600 V) and flat (<0.5% per 100 V) counting plateau. The 4π β detection system is unshielded and has a background rate of 0.7 counts sec^{-1} at 3500 V, the usual operating voltage.

In the $4\pi\beta$-$4\pi\gamma$-coincidence detection system, the chamber is mounted between two 7.6 x 12.7 cm NaI(Tl) crystals. The entire detection assembly is lead shielded. The upper crystal is pneumatically raised and lowered to permit access to the chamber. The signals from the two crystals are summed, amplified, and processed through a single

channel analyzer, and simultaneously recorded into a scaler and a multichannel analyzer. High voltage to the crystals is supplied by a single 0-1500 V regulated power supply. As described above, the signals from the chamber are summed and amplified, but then split into the variable coincidence gate and a scaler. The output signals from the variable coincidence gate unit are fed into a third scaler. The three scalers are controlled by a master timer.

A detailed discussion of the $4\pi\beta$-γ-coincidence method can be found in the National Council on Radiation Protection and Measurements Report No. 58 (NCRP, 1985).

C. Traceability to the National Institute of Standards and Technology (NIST).

Traceability to NIST is difficult since suitable standard reference material (SRM) sources are not available for many radionuclides. Our approach over the years has simply been to recalibrate NIST SRM solutions or solutions obtained from commercial vendors who are traceable to NIST using EML source preparation techniques and detection systems. Except for discrepancies caused by differences in the use of decay scheme parameters, the EML results are in agreement with the certified values within the error of the measurements.

D. EML source preparation.

Two kinds of supporting films for the source mounts are used in the proportional counters, which are described below.

The first type of film support is prepared from clear vinyl paint[*] and is used with pure beta emitters with a maximum energy >0.25 MeV. It has been established experimentally, using NIST and commercial vendor certified standard solutions of ^{45}Ca, that the detection efficiency of this emitter is 100% for the source mounts described below.

About 0.1 mL of paint is dropped onto a 1-cm deep layer of clean cold water in a 50 cm x 75 cm tray, painted matte black on the interior surfaces. The paint spreads across the surface of the water and the film thickness is estimated visually, by color. A clear or light gray film is ~ 5 μg cm^{-2}, white is 10 μg cm^{-2}, and yellow is 20 μg cm^{-2}, which

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[*] *Flat Vinyl Ink 3900-99-Clear, obtained from: Colonial Printing Ink Co., 180-T East Union Ave., East Rutherford, NJ 07073*

has been determined experimentally. At EML, the 10 μg cm^{-2} films are used as they provide the necessary strength, while still remaining extremely thin. The film is lifted from the surface of the water with a wire loop and placed over the aluminum washer. This thin film planchette is dried and then given a thin gold coating of about 10 μg cm^{-2} on one side by vacuum evaporation in a small sputterer unit. Self- absorption in the source mount is minimized using a technique developed at EML (Hallden and Fisenne, 1963). A 0.1-g aliquot of a Teflon bead suspension in water[*] (100 μg of Teflon g^{-1}) is placed onto the nonconducting side of the film. The radioactive solution to be standardized is delivered by weight from a polyethylene transfer pipette into the Teflon bead suspension on the film. In general, a 0.1-g weight of the radionuclide solution is used in the preparation of a source. The source is air dried and then gold coated to produce an entirely conducting mount.

The second kind of source mount is a 2-μm thick film of polycarbonate[**] that is glued to the aluminum washer. These mounts are used for measurements by $4\pi\beta$-$4\pi\gamma$-coincidence counting, which will correct for the absorption within the film. The remainder of the source preparation procedure is the same as described for $4\pi\beta$ measurements. (Note: The thin vinyl films will only tolerate an HCl solution of <1N. The stronger polycarbonate will withstand HCl concentrations of up to 6N. In addition, the polycarbonate source mounts can be dried gently under a heat lamp.) The graph below shows the $4\pi\beta$ counter detection efficiency versus β- or β+ endpoint energies for sources mounted on polycarbonate films.

The primary radionuclide solution should be essentially carrier-free. The primary radionuclide solution to be calibrated is diluted to a concentration on the order of 1700 Bq g^{-1}. The calibration is performed with triplicate source measurements. The voltage plateau for each source is determined in order to select the proper counting voltage. The activity and Poisson counting error are calculated for each source and corrected for the aliquot weight to obtain the activity g^{-1}. Other corrections appropriate to the particular radionuclide decay scheme are made from the $4\pi\beta$-γ-coincidence measurements. A Gaussian mean and SD are calculated for the triplicate sources.

Appropriate dilutions of the calibrated solution are prepared for distribution within EML for various programs.

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[*] *Vanflex Teflon Emulsifier #30; obtained from: Interchem Corp., 120 Route 17N, Suite 115-T, Paramus, NJ 07652*

[**] *KIMFOIL; obtained from: Kimberly-Clark Corp., Schweitzer Division, Lee, MA 01238*

LOW BACKGROUND GAS-FLOW PROPORTIONAL COUNTING SYSTEM

A. Application.

The only routine beta analysis conducted at EML is the determination of ^{90}Sr by measurement of ^{90}Y. These measurements are performed in the LB4100^{TM} (Oxford Instruments Inc., Nuclear Measurements Group, 601 Oak Ridge Turnpike, Oak Ridge, TN 37831-2560) low background counting system. This system is also utilized for gross alpha/gross beta determination in water and air filter samples.

B. Description of the detection system.

The LB4100 is a four drawer, 16 detector, low background counting system that is intended for gross counting of α, β and low energy x-ray emitters. The detectors are of the gas-flow proportional type. The detector slide accepts samples prepared in solid form on disks either 2.54 cm or 5 cm in diameter. Two drawers (of four detectors each) are equipped with 5 cm detectors, which are used for gross alpha/gross beta determinations. The other two drawers are equipped with 2.54 cm detectors that are used for counting the ^{90}Y samples. The detectors are mounted on a 2.54 cm Whatman No. 42 filter paper and are covered with Mylar fastened with a ring and disc assembly.

Each sample detector is the signal source for a counting channel. Each detector activates a counting channel independently of the others. Each channel has its own time control. The channels are individually deadtime corrected. The interface between the counter and the user is a computer and its keyboard.

C. Calibration of the detection system for ^{90}Y determinations.

The detectors are checked for energy plateaus and source (^{90}Sr) efficiencies prior to use or when the gas tank is changed. The background is also measured prior to use with yttrium oxalate blanks. If the instrument is idle for an extended period of time, crosstalk and yttrium oxalate efficiencies are checked. The counters are standardized with ^{90}Y oxalate with detection efficiencies of ~ 40%.

LIQUID SCINTILLATION MEASUREMENTS

A. Application

The conventional liquid scintillation (LS) counting systems used at EML are designed to detect and measure low-energy (e.g., ^{3}H, ^{14}C) to high-energy beta particles (i.e., ^{90}Y, ^{106}Rh), and alpha particles. Samples containing mixtures of radionuclides that emit either alpha, beta, or conversion electrons are detected and quantified using variations of the LS counting technique. Depending on the LS cocktail, the alpha detection efficiency is generally > 95%, whereas the beta detection efficiency is dependent on energy, spectral shape and cocktail. Typically, beta particles with maximum energies > 0.250 MeV are detected with >90% counting efficiency.

One variation of the LS counting technique used at EML is Cerenkov counting (Scarpitta and Fisenne, 1996), which is an adjunct to LS counting that does not require a LS cocktail. Cerenkov counting in aqueous samples is applicable to beta particles with endpoint energies in excess of 0.263 MeV. The Cerenkov counting efficiency (CCE) is typically 30% per MeV for beta particles with endpoint energies above the Cerenkov threshold; alpha particles are not detected.

Another variation of the LS technique utilized at EML is photon-electron rejecting alpha liquid scintillation spectrometry (PERALS), which is designed specifically for low-level liquid scintillation alpha detection where beta/gamma interferences are eliminated and a high degree of sensitivity is desired.

The three counting methods described above require a clear liquid sample that can be counted in a short period of time (1 to 2 h). Presented in the following sections are descriptions of these methods.

B. Description of the detection system

EML's Packard Tri-Carb-2250CA LS instrument is a multitasker unit for quantitative detection of alpha, beta, positron, and conversion electron radiation. The system may be preprogrammed to select any of the 15 unique programs stored on the computer disk. The instrument can be operated in one of four counting modes: 1) conventional LS, 2) dual-DPM, 3) full spectrum analysis, or 4) efficiency tracing. The system provides computer

controlled data reduction of counting results after each sample or after every batch of samples identified by the user.

The scintillation detector well is located underneath the sample changer. Samples are automatically lowered into the detector well. The detector assembly consists of two facing bi-alkali high performance photomutiplier tubes, each coupled to an optical chamber. The shielding assembly consists of a light-tight detection chamber, magnetic shielding and 5 cm of lead to reduce the effect of external radiation.

Spectrum analysis forms the basis of the Packard LS system. Typically, a beta particle will take a few nanoseconds to dissipate all of its energy in the scintillation solution. This results in an analog pulse rising to its maximum amplitude and falling to zero. The amplitude of the analog pulse is converted to a digital value. The conversion is achieved in a high speed analog to digital converter (ADC), and the digital value, which represents the beta particle energy, is the memory slot of a 0-2000 keV spectrum analyzer. The conversion by the ADC is linear so that during the measurement of a sample, the spectrum analyzer will accumulate counts representing the complete energy spectrum of the radionuclide. The spectrum analyzer is calibrated in units of keV, and the user can select one of three regions of interest over a range of 0-2000 keV.

Quenching, in general terms, refers to interferences with any of the steps of energy transferred from the solvent (where most of the energy from the ionizing radiation is initially deposited) to the scintillant and subsequent light transmission to the PMT. When a sample is quenched, the spectrum usually is shifted toward the lower energy regions, resulting in an increase in counts (i.e., apparent increase in counting efficiency) in any low energy preset regions of interests. In most cases, a correction for this effect may be performed.

A quench-efficiency curve must be established for a given nuclide and scintillator cocktail if the sample differs in chemical composition from the reference standard used to calibrate the instrument. A series of quenched samples, containing a known constant activity for the nuclide of interest, are prepared with a measured volume of scintillation cocktail. Successively increasing amounts of a chemical quenching agent are added to the samples. The samples are then measured for spectrum analysis. A description of the quench corrections and dual-dpm analysis can be found in Scarpitta and Fisenne (1996).

C. Calibration of the system.

The LS stability and operational acceptance criteria are checked first before running any standards or samples. This is accomplished by counting the background, and a ^{3}H and ^{14}C standard provided by the instrument manufacturer using an appropriate spreadsheet program. The count rate is then compared with the quality control charts developed at EML for this instrument. Descriptions of the cocktail selection, determination of window settings, and detection efficiencies can be found in Scarpitta and Fisenne (1996).

D. Cerenkov Counting

Cerenkov radiation occurs when charged particles pass through a dielectric medium (liquid) where there is an exchange of energy from the charged particle to the molecules of the medium (Haberer, 1966). The exchange energy produces local electronic polarizations in the medium if the charged particle is moving at velocities comparable to the velocity of light in the medium. When these polarized molecules return to their normal state, the excess energy is released as electromagnetic radiation known as Cerenkov radiation.

A threshold energy exists for the production of Cerenkov radiation and it is a function of the refractive index, n, of the medium. For water (n = 1.33) the lower energy limit of electrons for the production of Cerenkov radiation is 0.263 MeV (Berger and King, 1985). With beta emitters, the maximum energy, E_{max}, has to substantially exceed this value because of the broad beta energy spectrum. Photoelectrons and Compton electrons from gamma emitting nuclides, having energies in excess of the threshold energy, can also be measured by Cerenkov counting. Some nuclides which can be measured by Cerenkov counting are ^{32}P, ^{35}S, ^{36}Cl, ^{40}K, ^{59}Fe, ^{89}Sr, ^{90}Sr/Y, ^{106}Rh(Ru), ^{137}Cs, ^{210}Pb/Bi, ^{234m}Pa (progeny of ^{238}U via ^{234}Th), and ^{99}Tc.

Cerenkov counting is an adjunct to LS analysis that does not require a scintillating cocktail. Various wavelength shifters (Haberer, 1966; Karamanos et al., 1975) have been used to enhance the CCE by 10-20% using commercially available LS spectrometers. Wave shifters are organic substances that shift the Cerenkov photon energies from a region of low detector sensitivity to a region of high detector sensitivity.

Standardized solutions, containing the equivalent of about 17 Bq (1000 dpm), are dispensed gravimetrically, in triplicate, into either 20 mL plastic or low borosilicate glass

vials. Each vial was counted for 15-30 min with window settings of 0-50 keV for Cerenkov counting so that the 1 sigma counting error is <2%.

For Cerenkov counting, water is typically used as the solvent. The average instrument background, using 20-mL plastic vials containing 3-18 mL of ultra-pure water is 0.225 ± 0.018 counts sec^{-1} (cps) for a 0-50 keV region of interest. The average background count rate for glass vials (0.346 ± 0.010 cps) is about 30% higher than that obtained using plastic vials. Based on replicate background measurements, the lower limit of detection for a 1 h count at the 95% confidence level, using water as a solvent, is 0.024 cps (1.44 cpm) and 0.028 cps (1.70 cpm) for plastic and glass vials, respectively. For specific alpha/beta applications, see Scarpitta and Fisenne (1996).

E. PERALS Spectrometry

EML's PERALS spectrometer (Ordela, Inc., 1009 Alvin Weinberg Drive, Oak Ridge, TN 37830) which is a single sample stand-alone Nim Bin type unit. The unit is interfaced to a personal computer which is capable of functioning as a multichannel analyzer. The PERALS technique employs a LS detector with a pulse-shape discriminator for beta-gamma rejection (>98%) and direct extraction of the alpha activity of interest into 2 mL of a water insoluble organic phase containing the scintillator. The discriminator takes advantage of the longer decay time of light produced by alpha particle interactions to select the alpha pulses for spectrometry and reject those from beta-particle or gamma-ray events.

The choice of commercially available extractants (ETRAC Corporation) and chemical composition of the aqueous phase allows for selectivity and flexibility in sample preparation (McDowell and McDowell, 1994). The degree of beta/gamma rejection allows an instrument background (with a blank scintillator in the detector) of 0.001 cpm or less over the entire alpha energy range (3-5 MeV). The advantages of the PERALS method are that it: 1) is extremely sensitive, 2) has a high alpha counting efficiency (100%), 3) requires a count time of 1-4 h per sample or less depending on the sample activity, and 4) generates a very small amount of organic waste (<2 mL per sample).

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Ga-01-R

GAMMA RADIOASSAY

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APPLICATION

This procedure is used for the nondestructive measurement of γ-ray emitting radionuclides from a variety of environmental matrices by high resolution germanium (Ge) detector γ-ray spectrometry and NaI(Tl) detector γ-ray spectrometry. It is applicable to nuclides emitting γ-rays with energies > 20 keV for Ge detectors and 50 keV for NaI(Tl) detectors. For typical counting systems and sample types, activity levels of about 40 Bq are easily measured and sensitivities as low as .002 Bq can be achieved for many nuclides. Count rates in excess of 2000 counts sec^{-1} (cps) should be avoided because of electronic limitations. High count rate samples can be accommodated by dilution or by increasing the sample to detector distance.

The procedure is used for either qualitative, quantitative or relative determinations. In tracer work, the results may be expressed by comparison with an initial concentration of a given nuclide which is taken as 100%. For radioassay, the results may be expressed in terms of known standards for the radionuclides known to be present. In addition to the quantitative measurement of γ-ray radioactivity, γ-ray spectrometry can be used for the identification of specific emitters in a mixture of radionuclides. General information on radioactivity and the measurement of radiation has been published. Information on the specific application of γ-ray spectrometry is also available in the literature.

DESCRIPTION OF THE SYSTEM

Gamma-ray spectra are measured at EML with modular equipment consisting of a detector, an amplifier, a pulse-height analyzer, memory, and a permanent data storage device. Lithium-drifted germanium, Ge(Li), or high purity Ge detectors (p-type or

n-type) are used for the analysis of complex γ-ray spectra because of their excellent energy resolutions. These Ge detectors, however, are characterized by high cost and require cooling with liquid nitrogen. Thallium activated sodium-iodide crystals, NaI(Tl), can be operated at ambient temperatures and are often used at EML as γ-ray detectors in spectrometer systems. However, their energy resolutions limit their use to the analysis of single nuclides or simple mixtures of a few nuclides.

Upon completion of the γ-ray assay, the spectral data are interpreted and reduced to nuclide activities in Bq (disintegrations per second) or related units suited to the particular application. At this time, the spectral data may be inspected on the CRT to identify the γ-ray emitters present. This is accomplished by reading the channel number from the x-axis and converting to γ-ray energy by multiplying by the appropriate keV/channel (system gain). If the system is calibrated for 1 keV per channel with channel zero representing 0 keV, the energy will be equal to the channel number. The channel number or γ-ray energy in keV is usually displayed on the CRT. Identification of nuclides is aided by catalogs of γ-ray spectra and other nuclear data tabulations. Because of the reduced spectral resolution obtained with NaI(Tl) detectors, this technique can only be applied to samples of single nuclides or very simple combinations of nuclides.

Data reduction of spectra taken with Ge spectrometry systems is usually accomplished by integration of the photopeaks above a definable background (or baseline), and by subsequent activity calculations using a library that includes data such as nuclide name, half-life, γ-ray energies and associated abundance (intensity or branching ratios). Computer programs for data reduction of NaI(Tl) detector data have been used extensively at EML. Data reduction of spectra involving mixtures of nuclides is usually accomplished by least-square fitting routines to a library of standard spectra of the individual nuclides acquired under individual conditions.

Variation of the physical geometry of the sample and its relationship with the detector will produce both qualitative and quantitative variations in the γ-ray spectrum. To adequately account for these geometry effects, calibrations are designed to duplicate all sample counting conditions including source-to-detector distance, sample shape and size.

Electronic problems, such as erroneous deadtime correction, loss of resolution, and random summing, may be avoided by keeping the gross count rate below 2000 cps and also by keeping the deadtime of the analyzer below 5%. Total counting time is governed

by the radioactivity of the sample, the detector-to-source distance, and the acceptable Poisson counting uncertainty.

In complex mixtures of γ-ray emitters, the degree of interference of one nuclide in the determination of another is governed by several factors. If the γ-ray emission rates from different radionuclides are similar, interference will occur when the photopeaks are not completely resolved and overlap. If the nuclides are present in the mixture at markedly different levels of activity, nuclides of higher energies that are predominant can cause serious interferences with the interpretation of minor, less energetic, γ-ray photopeaks. The complexity of the analysis method is due to the resolution of these interferences and, thus, one of the main reasons for computerized systems.

Cascade summing may occur when nuclides that decay by a γ-ray cascade are analyzed. Cobalt-60 is an example; 1172 and 1333 keV γ-ray from the same decay may enter the detector to produce a sum peak at 2505 keV or a count in the continuum between the individual peaks and the sum peak; thus, causing the loss of counts from one or both of the other two peaks. Cascade summing may be reduced by increasing the source-to-detector distance. Summing is more significant if a well-type detector is used.

Random summing is a function of counting rate and occurs in all measurements. The random summing rate is proportional to the total count squared and the resolving time of the detection system. For most systems, random summing losses can be held to < 1% by limiting the total counting rate to 1000 cps.

The density of the sample is another factor that can affect quantitative results. Errors from this source can be avoided by preparing the standards for calibration in solutions or other matrices with a density comparable to the sample being analyzed. Another approach is to apply attenuation corrections to all calibration standards and samples based on sample weight, known volume, γ-ray path length and average atomic number of the sample matrix.

APPARATUS

A γ-ray spectrometer consists of the following components:

A. Detector assembly.

1. <u>Germanium detector</u> - The detector should have a volume of at least 50 cm^{-3}, with a full width at one half the peak maximum (FWHM) < 2.2 keV at 1332 keV, certified by the manufacturer. A charge-sensitive preamplifier using low noise field effect transistors should be an integral part of the detector assembly. A convenient support should be provided for samples of the desired form. Vertical systems allow the standard/sample to be placed directly on the detector end cap.

2. <u>NaI(Tl) detector</u> - The sodium iodide crystal, activated with about 0.1% thallium iodide, should contain < 5 µg g^{-1} of K and should be free of other radioactive materials. The crystal should be attached and optically coupled to a multiplier phototube. (The multiplier phototube requires a preamplifier or a cathode follower compatible preamplifier with the amplifier.) The resolution (FWHM) of the assembly for the photopeak of ^{137}Cs should be < 7% for a 75-mm by 75-mm detector.

3. <u>Shield</u> - The detector assembly should be surrounded by an external radiation shield made of massive metal, equivalent to 102 mm of Pb in γ-ray attenuation capability. It is desirable that the inner walls of the shield be at least 127 mm in distance from the detector surfaces to reduce backscatter. If the shield is made of Pb or a Pb liner, the shield must have a graded inner shield of 1.6 mm of Cd or tin lined with 0.4 mm of copper, to attenuate the 88 keV Pb γ-rays. The shield must also have a door or port for inserting and removing samples.

4. <u>High-voltage power/bias supply</u> - The bias supply required for Ge detectors usually provides a voltage up to 5000 V and 1 to 100 µA. NaI(Tl) detectors require a high-voltage power supply of a range of usually from 500 to 3000 V and up to 10 mA to operate the multiplier phototube. The power supply should be regulated to 0.1% with a ripple of not more than 0.01%. Line noise caused by other equipment should be removed with filters and additional regulators.

5. <u>Amplifier</u> - A spectroscopy amplifier compatible with the preamplifier and with the pulse-height analyzer should be used.

B. Data acquisition and storage equipment.

1. <u>Data acquisition</u> - A multichannel pulse-height analyzer (MCA) or stand-alone analog-to-digital converter (ADC) under software control of a separate computer, performs many functions that are required for γ-ray spectrometry. An MCA or computer collects the data, provides a visual display, and outputs final results or raw data for later analysis. The four major components of an MCA are the ADC, the memory, control, and input/output. The ADC digitizes the analog pulses from the detector amplifier. The magnitude of these pulses is proportional to the energy of the photon deposited in the detector. The digital result is used by the MCA to select a memory location (channel number) which is used to store the number of events which have occurred with that energy. Simple data analysis and control of the MCA is accomplished with microprocessors. These processors control the input/output, channel summing over set regions of interest, and system energy calibration, etc.

2. <u>Data storage</u> - Because of the use of microprocessors, modern MCAs provide a wide range of input and output (I/O) capabilities. Typically, these capabilities include the ability to transfer any section of data to one or more of the following: terminal, line printer, cassette tape, floppy or hard disk, X-Y plotter, and to computer interfaces via a serial or parallel port.

SAMPLE/STANDARD CONTAINERS

Sample mounts and containers must have a convenient reproducible geometry. Considerations include commercial availability, ease of use and disposal, and the containment of radioactivity for protection of the personnel and working environment from contamination. The evaporation of liquid samples to dryness is not necessary and liquid samples up to 1 L may be used. Massive samples may cause significant self-absorption of low energy γ-rays and may degrade the higher energy γ-rays. A ß absorber consisting of about 6 mm of Al, Be, or plastic may be used for samples that have a significant ß activity and high ß energies.

CALIBRATION AND STANDARDIZATION

This section describes the analysis of mixtures of radionuclides with Ge detectors or single or simple mixtures of radionuclides with NaI(Tl) detectors. If complex mixtures of radionuclides are to be analyzed with NaI(Tl) detectors, refer to page 2 of this procedure.

A. Preparation of apparatus.

Follow the manufacturer's instructions, limitations, and cautions for the setup and the preliminary testing of all of the spectrometry equipment to be used in the analysis. This equipment would include, as applicable: detector, power supplies, preamplifiers, amplifiers, multichannel analyzers, and computing systems.

Place an appropriate volume of a standard or a mixed standard of radionuclides in a sealed container and place the container at a desirable and reproducible source-to-detector distance. For environmental analysis, most standards/samples are counted at the detector end cap. The standard should provide about 100 cps in the peaks of interest and should be made up of standard sources traceable to a nationally certified laboratory. In all radionuclide measurements, the volumes, shape, physical and chemical characteristics of the samples, standards and their containers must be as identical as practicable for the most accurate results.

B. Energy calibration.

The energy calibration (channel number of the MCA versus the γ-ray energy) of the detector system is accomplished at a fixed gain using standards containing known radionuclides. The standards should be in sealed containers and should emit at least four different γ-ray energies covering the range of interest, usually 50 keV to 2000 keV in order to test for system linearity. Some commercially available nuclides suitable for energy calibration are: ^{210}Pb, 46.5 keV; ^{241}Am, 59.5 keV; ^{109}Cd, 88 keV; ^{141}Ce, 145 keV; ^{51}Cr, 320 keV; ^{137}Cs, 662 keV; ^{54}Mg, 835 keV; ^{22}Na, 511 and 1275 keV; ^{88}Y, 898 and 1836 keV; ^{60}Co, 1173 and 1332 keV; equilibrated ^{226}Ra, 186, 352, 609, 1120, and 1765 keV. A mixed γ-ray standard for energy and efficiency calibration is also commercially available. This standard can be obtained in solid form in a user supplied container. The radionuclide purity of the standards should be verified periodically to ensure against

accidental contamination or the presence of long-lived impurities by comparing the observed spectra with the spectra published in the literature.

A MCA should be calibrated to cover the range of interest. If the range is 50 to 2000 keV, the gain of the system should be adjusted until the ^{137}Cs photopeak, 662 keV, is about one-third full scale. Leaving the gain constant, locate at least three other photopeaks of different energies, covering the same range. Determine and record the MCA channel number corresponding to the maximum count rate for each of the four energies. Germanium detectors will have a linear relationship if the equipment is operating properly. Similarly, MCAs and NaI(Tl) detectors being produced today are capable of producing an almost linear energy response. Samples should not be analyzed if this relationship is not obtained. Follow the appropriate manufacturer input instructions for the determination of the slope and intercept. During each day in which the spectrometry system is being used to analyze samples, the above sequence of operation shall be repeated using at least two different energies. If the slope and intercept are essentially unchanged, the energy calibration data remain valid. If an appreciable change in the slope or intercept is evident, the entire calibration procedure must be rerun.

C. Photon detection efficiency calibration.

Accumulate an energy spectrum using sealed, calibrated radioactivity standards in a desired and reproducible counting geometry. At least 10,000 net counts (total counts minus the compton continuum and ambient background) should be accumulated in each full-energy γ-ray peak of interest.

Correct the radioactivity standard source γ-ray emission rate for the decay from the time of standardization to the time at which the count rate is measured.

Calculate the full-energy peak efficiency Ef as follows:

$$Ef = Np/Ng$$

where

Ef = full-energy peak efficiency (counts per γ-ray emitted),
Np = net γ-ray count in the full-energy peak of interest (cps), and
Ng = γ-ray emission rate (rays sec^{-1}).

If the standard source is calibrated as to activity, the γ-ray emission rate is given by:

$$Ng = A*Pg$$

where

    A   = number of nuclear decays per second, and

    Pg = probability per nuclear decay for the γ-ray.

For Ge detectors, plot the values for the full-energy peak efficiency versus γ-ray energy. The plot will allow the determination of efficiencies at energies for which standards are not available, and will show that the algorithms used in computerized systems are providing valid efficiency calibrations.

Once the efficiencies have been determined, it is unnecessary to recalculate them unless there is a change in resolution, geometry, or system configuration.

SAMPLE MEASUREMENTS

After the spectrometer system has been set up, the energy and efficiency calibrations are performed, then the unknown sample can be measured.

Following the general concepts of quantitative analytical chemistry, transfer the sample to the specimen container and position it in the same manner as was done during system calibration.

Measure the sample for a period of time long enough to acquire a γ-ray spectrum which will meet the minimum acceptable counting uncertainty.

PEAK AREA CALCULATIONS

Spectral data obtained with a Ge detector are only corrected for background when these peaks may alter the final results. In many experiments, the background may not affect the results but is still monitored to ensure the integrity of the system.

The underlying aim of this procedure is to subtract the continuum or baseline from the spectral data where it underlies a photopeak of interest. For operator-directed calculations, the choice of the baseline level may be straightforward. The simplest way, using a plot of the spectral data, is to draw a straight line, using judgement and experience, that best describes the baseline. Then the baseline data can be read directly from the plot and subtracted.

Photopeaks lying on a sloping baseline or one with curvature will be analyzed, independent of the method, with increased uncertainty. Use of data from these peaks should be limited to those cases where there is no other alternative. Photopeaks that overlap with each other will also increase the uncertainty of the final result.

In order to determine nuclide concentrations, the photopeak areas corrected for background and interferences are divided by the count time and efficiency for the energy of the γ-ray being calculated to give photons sec^{-1} for the peak of interest. If, as is the case for some nuclides, the branching ratio is not accurately known and a direct calibration was made with the same nuclide, the branching ratio and efficiency will be one number that converts cps to Bq sec^{-1} for the nuclide and photopeak of interest. If not, the photons sec^{-1} are converted to disintegrations sec^{-1} by dividing the photons sec^{-1} by the photons per disintegration, for the nuclide and photopeak of interest. The results are then corrected for attenuation or decay, or both.

Canberra Industries MicroSampo Version 2.0 (a commercial software package) is used at EML to perform these calculations.

While the uncertainty due to counting and calibration may represent a significant proportion of the total uncertainty in the measurement, systematic uncertainties should be determined and included in the above calculation. Systematic uncertainties include, but are not limited to, reproducibility of sample position, peak analyses, decay calculations,

 background subtraction, pulse pile-up, cascade summing corrections, and self-absorption corrections.

ANALYSIS OF COMPLEX MIXTURES
OF NUCLIDES WITH NAI(Tl) DETECTORS

Because of the inherent energy resolution of NaI(Tl) detectors, γ-ray peaks in complex mixtures of nuclides may not be separated sufficiently for quantification as outlined above. It may not even be possible to visually locate individual peaks if their energies are similar or their intensity is too low in relation to other γ-rays present in the spectrum. Complex mixtures of as many as 10 to 20 radionuclides can be quantified mathematically with computer programs using linear least squares techniques.

When using these techniques, care should be given to the following parameters.

A. System gain and zero energy channel.

The exact gain and zero energy channel of the spectrometer must be monitored and recorded. If the computer analysis program performs gain and/or baseline (zero energy channel) corrections on sample data, then the library of standards data must be obtained under uniform and precise calibration conditions.

B. Library standards.

The least squares analysis technique is a linear combination of all of the data contained in the standards library. Therefore, the standards library must contain a spectra of every component in the sample; in addition, these spectra must be obtained from the purest radionuclides available.

C. Counting of library standards.

All γ-ray spectra will contain a background component. The activity of the library standards must be high enough so that this background component will be insignificant, even though all computer programs make some kind of a background correction.

The duration of the counting period for the standard library spectra should be long enough to obtain statistically valid data, but it should be short enough so that analyzer gain and baseline drifts are insignificant.

The activity of the library standards should be chosen so that the counting rates of the predominant photopeaks are all about the same.

A very important data evaluation technique to be used with a least squares program is a superimposed plot of the original sample data and the computed spectral data. A plot of residuals (the difference between the original and computed spectra) is also very important. The residuals plot is very sensitive to errors that are caused by omitting radionuclides present in the sample from the library standards.

SAMPLE MEASUREMENTS

After the spectrometer system has been set up, the energy calibrations performed, and individual pulse-height spectra for nuclides expected to be present in samples are obtained, then the unknown specimens can be measured and quantified.

Following the general concepts of quantitative analytical chemistry, transfer the sample to the specimen container and position it in the same manner as was done during system calibration. Measure the sample for a period of time long enough to acquire a γ-ray spectrum which will meet the minimum acceptable counting uncertainty.

COMPUTER CALCULATIONS

WLSQ is an EML least squares Fortran computer program that can be run on a VAX or IBM-compatible PC to resolve complex spectra.

Systematic uncertainties include, but are not limited to: reproducibility of sample position, peak analyses, decay calculations, background substraction, pulse pile up, cascade summing operations, and self-absorption corrections.

The uncertainty obtained from the least squares analysis can be substituted for the uncertainty in counting and should be included to obtain an overall uncertainty of the analysis. The uncertainties obtained from the least squares analysis are the square roots of the diagonal elements of the inverse matrix used to solve the linear set of simultaneous equations representing the sample spectra.

QUALITY CONTROL

The following quality control procedures are required so that the γ-ray spectrometers maintain their energy calibrations. In addition, the systems are to be monitored so that degradation in performance will be noticed as soon as possible.

A. Daily calibration checks.

The energy calibration of each Ge γ-ray detector is determined daily with a mixed nuclide source consisting of ^{241}Am and ^{60}Co.

In order to maintain an energy calibration of 0.5 keV/channel, count the Am-Co source to obtain well-defined peaks. The 59.5 keV γ-ray line from ^{241}Am should fall in channel 119. The 1332.5 keV γ-ray line from ^{60}Co should fall in channel 2665.

If the ^{241}Am and ^{60}Co peaks do not fall in the correct channels, first adjust the DC offset of the amplifier so that the 59.5 keV γ-ray line falls in channel 119. Then adjust the fine gain of the amplifier so that the 1332.5 keV γ-ray line falls in channel 2665.

Recount the Co-Am calibration standard to verify the peak positions and readjust the amplifier if necessary.

The energy calibration of each NaI(Tl) γ-ray detector is determined daily with a nuclide source consisting of ^{207}Bi.

In order to maintain an energy calibration of 10 keV/channel, count the ^{207}Bi source to obtain well-defined peaks. The 570 keV γ-ray line should fall in channel 57. The 1064 keV γ-ray line should fall in channel 106.

If the ^{207}Bi peaks do not fall in the correct channels, first adjust the DC offset of the amplifier so that the 570 keV γ-ray line falls in channel 57. Then adjust the fine gain of the amplifier so that the 1064 keV γ-ray line falls in channel 106.

Recount the ^{207}Bi calibration standard to verify the peak positions and readjust the amplifier if necessary.

B. Weekly efficiency calibration and resolution checks.

Each week the same ^{137}Cs calibration standard is counted, recorded, analyzed, and the date is entered into a permanent data base for each γ-ray spectrometry system.

Count the ^{137}Cs calibration standard in the same manner as unknown samples. Record the data for permanent storage and perform the usual data reduction analysis.

Enter the results of the analysis (Bq) and the resolution of the ^{137}Cs peak (full width at half maximum in keV) in the γ-quality control data base.

Report any deviation from the expected values before samples are analyzed. If remedial action is necessary, the cause and solution of the problem must be recorded in the laboratory logbook. A complete recalibration must be performed if any remedial actions have been taken.