

# RADIOACTIVITY MEASURING INSTRUMENTS CALIBRATION AND USE

## PURPOSE

This procedure gives methods and prescribes materials to be used in the calibration and use of the Radiological Health Department's laboratory equipment for analysis of radioactive samples and in vivo measurements of radioactivity.

## POLICY

Instruments that are used for making quantitative in vitro or in vivo measurements of radioactivity for determination of radiation doses, concentrations of radioactivity in effluents, or for verification of other aspects of regulatory compliance, shall be calibrated at regular intervals as appropriate for the particular instrument. Operating instructions, either supplied by the manufacturer or prepared by the user, shall be maintained in a manner that would allow a competent individual to use the equipment properly, even on an irregular basis.

## DEFINITIONS

Calibration - The determination of an instrument's response to a radioactive source in a particular physical condition and position relative to the detector.

## GAMMA SPECTROMETRY

### Germanium Detector and ND62 Multi-channel Analyzer

#### Equipment

ND62 multichannel analyzer  
ND 516 amplifier  
Tennelec TC205A Linear Amplifier  
PGT model 314 5 kV bias supply  
PGT model 315 5 kV bias supply  
PGT Intrinsic Germanium vertical detector

Refer to the ND62 operators manual and the 516 or TC205A amplifier instruction manuals for information

on the use of the instruments for acquiring and printing out the appropriate data.

#### Efficiency Calibration

Calibration of the detection efficiency of the Intrinsic Ge detector as a function of photon energy will be performed every two years using a NEN multi-nuclide reference solution NES-615. The detector will be calibrated in one or more of the basic geometries. In the off years, system calibration will be verified in geometry G with those energies that are still identifiable and useful in the sample from the previous year.

Geometry A: The vial that the solution is delivered in will be counted and the peaks found at distances of 2 cm from the face of the detector.

Geometry B: The standard will then be placed in a 50 mL Corning flask and filled with enough water to bring the total volume to 50 mL. The system is then calibrated with the flask placed against and centered on the face of the detector.

Geometry C: Equal aliquots of the contents of the 50 mL flask are then placed into two 50 mL tubes (of the same type used to count radiopharmacy urine samples) and enough water added to bring the total content of the tubes to 100 mL. The tubes are then counted by placing the pair of tubes against the side of the detector at the top of the detector and at the bottom of the detector.

Geometry D: The contents of the two 50 mL tubes are then placed into a 200 mL Corning flask with water added to bring it to 200 mL of liquid. The 200 mL flask is then placed against the detector and calibrated as before for geometry B.

Geometry E: The contents of the 200 mL flask is then placed into a marinelli beaker and filled with water added to bring it to the top of the beaker. The beaker is then placed over the detector and calibrated.

Geometry F: The next calibration is for solid material in Lab Tek disposable petri dishes VWR cat No. 25384-070. Quartz sand is put in the dish and the liquid from the marinelli beaker is evaporated in the dish. The lid is then placed on the dish and wrapped with tape at the circumference of the dish. The dish is then counted when centered and placed flat against the face of the detector.

Geometry G: The contents of the petri dish is the placed into a marinelli beaker with enough sand added to fill the beaker. The sand will be mixed to produce a uniform mixture. The beaker is then placed over the detector and counted.

In each of the counting geometries at least 20,000 net counts should be acquired for each energy peak represented in the sample. The energy peak efficiency is then found for each geometry as described in ANSI N42.14-1978. The curves and/or equations are then placed in a calibration book that is kept in the counting lab.

#### Sample preparation and counting

Low activity bulk solids e.g. ash:

Solid samples are to be placed in Lab Tek disposable petri dishes VWR cat No. 25384-070 or marinelli beaker. An empty dish and lid or beaker are prepared in the same manner and placed on the balance, the balance is then adjusted to indicate 0 mass. The sample is next placed on the balance and the net mass indicated is recorded. The sample is then counted in geometry F or G and the activity of isotopes in the sample determined using the appropriate efficiency. The specific activity can then be determined.

Low activity liquid samples:

Virtually any container can be placed next to the detector for a qualitative count. If the counting reveals some significant activity, the sample is then placed in a 50 or 200 mL Corning Flask or marinelli beaker for quantitative analysis. The sample is then counted in geometry B, D or E and the specific activity found by using the appropriate energy efficiency and knowing the volume of sample.

High activity liquids or solids:

These samples are placed in a 5 to 10 mL glass vial or test tube and counted, using the energy calibration of geometry A to determine the activity of the sample.

### THYROID COUNTING EQUIPMENT

Thyroid counting equipment consists of a scaler and a low-energy gamma detector. This equipment will be calibrated with an I-129 source NES-135S (to simulate I-125), to obtain a response factor for that will be used for I-125.

#### Equipment

Eberline Model E-6000 scaler-ratemeter  
Eberline Smart LEG-1 NaI low-energy gamma detector.  
Nuclear Associates Economy Neck Phantom

#### Calibration

Refer to the manufacturers' instruction manuals for information on how to set up and operate the instruments and acquire data.

During all parts of the calibration procedure the detector will be placed against the phantom in closest proximity to the sample well and with the edge of the detector even with the bottom of the well.

Acquire a one or two minute count of phantom background and a one or two minute count with the I-129 source in the phantom well. Subtract the background count from the I-129 counts to obtain the net counts, change the net counts to net counts per minute. Divide the net counts per minute by the activity of the standard (in nanocuries) to obtain the detection efficiency in cpm/nCi for I-129. Multiply I-129 efficiency by 1.9 to obtain the I-125 efficiency.

This efficiency will be used for determining I-125 thyroid burden. Before each use of the system to count thyroids the I-129 source should be recounted and be within 15% of the detection efficiency found at annual calibration.

#### Thyroid Counting

The net count to be used to determine I-125 activity in the thyroid is obtained by subtracting a background count

from the thyroid count. For screening measurements, the background count is obtained by counting the neck phantom without the check source. For verification measurements (i.e. if a positive result is obtained), the background count is obtained by counting the thigh of the individual being assayed. The net counts are then divided by the detection efficiency to obtain the activity in the thyroid.

## **WINDOWLESS PROPORTIONAL COUNTING FOR ALPHA AND BETA**

### Equipment

Ludlum 2200 scaler  
Eberline FC-1 proportional chamber  
NEN beta source set NES-269  
Eberline alpha set S94-1

This equipment is used only infrequently to count samples; it will therefore be calibrated just prior to use. Use the beta sources with appropriate backing material to duplicate backscatter conditions that will be used during sample counting. The voltage setting should be determined by making suitable plateau measurements.

## **LIQUID SCINTILLATION COUNTING FOR ALPHA OR BETA**

Consult the manufacturer's instruction manual on the operation of the unit.

### Equipment

Beckman LS 5000TD Liquid Scintillation System  
NEN NES-209 Unquenched LSC Mini-Vial Standard Set

### Calibration and sample analysis

The LSC is used primarily for qualitative or semi-quantitative analyses of wipe tests for removable contamination and, occasionally, for urine samples. When quantitative results are deemed necessary (e.g. quantitative assays of urine samples) and quenching must be accounted for, appropriate standards will be prepared to obtain the necessary curves of H number vs. efficiency (see manual section on H number and AQC automatic quench control) or the samples will be spiked with a known activity of the

isotope in question to make quantitative determination possible.

Semi-quantitative determination of removable contamination on wipes of surfaces can be inferred by using approximate efficiencies for groups of suspected nuclides. The assumption is made that quenching is fairly constant and due to self absorption in the wipe and attenuation by the plastic vial. As quenching for this situation is fairly small, count shifting from channel two to one is assumed to be unimportant and is not considered.

Program 2 is used for counting wipes of suspected H-3, C-14, S-35, or Ca-45 contamination. Also is used to estimate the activity of H-3 and C-14 waste vials before disposal.

The efficiency used will be determined by spiking and counting mini vials and standard vials containing 6mL and 10mL of Optifluor, respectively, with known quantities of H-3 and C-14.

Statistical tests of reproducibility will be performed monthly on the LSC. The test will be performed in accordance with recommendations found in ANSI N42.15-1980. A series of ten 1-minute counts will be made using program 4 with an NEN NES-209 source set. The data will then be analyzed to verify that the STD DEV and EST STD DEV are comparable and that the Chi Square falls in the acceptable 5% to 95% range.

### Urine Counting

For quantitative analysis of urine, three mini-vials will be prepared, each containing 6 mL of Optifluor. The first vial will have 1 mL of water added to use as a background. The second vial will have 1 mL of urine added. The third vial will have 1 mL of urine added, plus an appropriate spike of no more than 0.2 mL containing a known activity of the nuclide of interest. The counting efficiency for the nonspiked vial will be determined from the counts obtained from the spiked vial.

## **REFERENCES**

American National Standard: *Calibration and usage of germanium detectors for measurement of gamma-ray emission of radionuclides*, ANSI N42.14-1978.

American National Standard: *Performance verification of liquid scintillation counting systems*, ANSI N42.15-1980.

ND62 Multichannel Analyzer/Remote Terminal Operator's Manual, 07-0119, Nuclear Data, Inc.

Instruction Manual Models 514/516 Spectroscopy Amplifier, Mech-Tronics Nuclear.

Hardware Instruction Manual ND510 Spectroscopy Amplifier, 07-0029, Nuclear Data Inc.

ND Six Portable MCA user's guide, Nuclear Data Inc.

Technical Manual, Mini Scaler Model MS-3, Eberline Instrument Corporation.

Instruction Manual, Model 2200 Portable Scaler Ratemeter, Ludlum Measurements, Inc.

Instruction Manual, LS 7000 Liquid Scintillation System, Beckman Instruments, Inc.

Instruction Manual, TC205A Linear Amplifier, Tennelec

# RPR 53A. CALIBRATION OF A GERMANIUM DETECTOR AND THE ND62 MULTI-CHANNEL ANALYZER SYSTEM

## MULTI-NUCLIDE CALIBRATION STANDARD

The calibration standard shall be a multi-nuclide source traceable to the National Institute for Science and Technology (NIST, formerly the National Bureau of Standards) that will check the system at energies between 80 keV and 2,000 keV. In the past, Dupont model NE-615 multi-nuclide source has been used as the calibration standard.

Each detector shall first be calibrated utilizing the multi-nuclide reference source as a point source in the form received. The GeLi detector should be calibrated with the source at 2cm, 4cm, 8cm, 12cm, from the face of the detector. The Ge detector should be calibrated with the source 2cm from the face of the detector. The source should be counted each time, for a time period that produces a total of 20,000 counts.

After the point source calibration is completed. The source will be utilize to make up sources of the typical sample geometries to be used in subsequent calibrations.

## ND62 MULTI-CHANNEL ANALYZER SYSTEM CALIBRATION

### Setup

Turn the system on; either the message "HELLO", "MEM?", or "ACQ?" is displayed in the keyboard entry line. The message "HELLO" indicates the system is ready for operation. If "MEM" or "ACQ" is displayed, refer to the instruction manual.

### Initial System Setup and Spectrum Storage

Refer to instruction manual section 5-3 for initial system setup. Set systems equipment as follows:

#### Amplifier Settings

Gain: 50  
I: 2.5 - 40.6  
Shaping: 4  
BLR: Lo-rate  
Input: Neg.  
Output: unipolar  
5 KV Bias Power Supply  
Hi Voltage: Pos

Output: 3.1

Pot: 20.03

(Germanium detector bias voltage at 3000 volts.)

### Gain and Region of Interest Calibration

Using the ND62 MPOS and SPAN keys, the amplifier gain controls and the zero control, the system is calibrated to read 1 keV/channel. Sources to be used for this calibration are Co 60 and Ba 133.

- 1 Place Co 60 and Ba 133 sources in front of the Geli detector.
- 2 Press function-lock, using the PAGE key, select the SPECTRUM DATA PAGE screen. This is identified by the same title in the lower left hand corner of the screen.
- 3 Press ACQ then the RETURN key. Data will start to accumulate, when there is a well defined energy profile. Deactivate function Lock. Type in " OF " press return, this ends data accumulation.
- 4 Press Function Lock key, press MPOS key using it to move the marker to the top of the first (80.997 keV) energy peak. (Use +/- key to reverse marker direction.
- 5 Press SPAN key to separate out the right side marker. Move marker to the energy peak at (1332.486 keV).
- 6 Press function-lock to deactivate its function.
- 7 Type in LE space 80.997 then press return.
- 8 Type in RI space 1332.486 then press return.
- 9 Calibration check
  - a Type in ME space 303; press return; both RI & LE should read approximately 303.195.
  - b Type in ME space 1173; press return; both RI & LE should read approximately 1172.983.
  - c Type in ME space 2000; press return; both RI & LE should read approximately 2000.

d Press function-lock.

e Press ERAS then return; this clears the screen.

10 If step 9a does not produce the approximate ME readings as indicated; adjust the gain and redo steps 3-9. Continue until approximate readings are obtained.

### Counting Time

1 Press function-lock.

2 Display the TIME PAGE by pressing the PAGE key until it comes up; it will be identified by the same title in the lower left hand side of the screen.

3 Press function-lock to deactivate it's function.

4 Type in LI space then the counting time that is needed to produce greater than 20,000 counts for each isotope's primary energy peak. If it takes 2 hours & 30 minutes then type in LI 02:30:00 and press return.

5 Remove Co-60 & Ba-133 sources. System is now ready to count the calibration source.

### **POINT SOURCE CALIBRATION**

1 The multi-nuclide calibration source is to be counted at 2, 4, 8, and 12 cm from the face of the GeLi detector.

2 Place multi-nuclide source 2 cm from the face of the GeLi detector. Place all shield bricks in place.

3 Set count time for 15 minutes. Select TIME PAGE screen. To erase the data accumulated during the system calibration and reset LIVE and REAL times to zero. Press the function lock key, then ERAS key, then return key. Type in LI 00:15:00, press return key.

4 Start the counting by typing in ON and pressing the RETURN key. The count will stop automatically after 15 minutes when counting is completed.

The printer must be programmed before printing out the data.

### Programming the Printer

1 Type in LP press RETURN key.

2 Type in CO press RETURN key. (programs the printer)

3- Press CTRL key and O key. (compresses data)

4 Press CTRL key and Z key. ( takes printer out of program mode)

### Set Region of Interest

To avoid printing out all the data accumulated. Regions of interest must be set. There are 11 primary energy peaks in the multi- nuclide calibration source and each one will be a region of interest.

1 When counting is completed, using the PAGE key select the SPECTRUM DATA page.

2 Using the MPOS key, move the marker to the top of first primary energy peak. (88.03)

3 Press the EXPD key then press the RETURN key. (this activates the expand mode)

4 Press ZOOM key and hold it until the data has contracted enough to give a well defined energy peak. If the data continues to expand when the ZOOM key is pressed. Then press the -/+ key, this will cause the data to contract.

5 Move the marker to the base of the forward slope of the energy peak.

6 Using the SPAN key, separate out the right hand marker and move it to the base of the reverse slope of the energy peak.

7 With left and right markers positioned at the base on each side of the energy peak. Press ENTER key then the RETURN key. This sets the region between the markers as a region of interest.

8 Using the same procedure as above, set a region of interest for each of the 10 remaining nuclide.

### Data Print Out

1 Type in ST then press Return Key.

2 Type in PT then press Return Key.

3 Type in PR then press Return Key.

Printer should have been typing as each of the last three functions were entered.

When data print out is completed. Reposition multi-nuclide point source to the next calibration point which is 4cm from the face of the detector and repeat the count.

Continue this procedure until the multi-nuclide source has been counted at each of the 4 distances. Remember that each count must produce a minimum of 20,000 total counts. If the minimum counts is not produced, increase the count time and repeat the count.

After each of the 4 calibration point have been counted utilizing the GeLi detector. Connect the Germanium detector to the amplifier and count the multi-nuclide source at 2 cm from the face of the detector utilizing the above procedure.

After the point source calibration has been completed for both detectors and the data has been analyzed. The multi-nuclide source can be use to make up the sample geometry sources as per the following procedure. They are to be counted at each of the above calibration points for both detectors.

## PREPARATION PROCEDURES FOR REFERENCE SAMPLES

### Work Station Preparation

- 1 Cover work bench with absorbent paper. Obtain the following equipment.

- 100 mL volumetric flask
- 1000 mL 0.1 normal HCL diluent
- 20 mL volumetric glass pipette
- 10 mL volumetric glass pipette
- 5 mL volumetric glass pipette
- Lab coat and gloves
- Dispensing bulb
- Pasteur pipettes
- Heat lamp
- New 50mL tissue culture flasks
- Clean 100 mm x 15 mm plastic petri dishes
- Red top Vacutainer tubes
- HCL (concentrate)
- Distilled water
- Syringe (5cc) and 22 gage needle
- Balance and standard weight set

- 2 Before opening the ampoule, perform a wet simulation of the dilution procedure by preparing 7 control (blanks) which represent each of the 7 different sample geometries. Use the same solutions and clean glassware as will be used in the preparation of the Primary Standard. Refine and correct procedure or equipment inadequacies.

### Primary Standard Working Solution

- 1 Rinse and dry the exterior of the sealed ampoule. Shake the ampoule to empty the stem of liquid. Use a file to etch the glass neck of the ampoule as necessary.
- 2 Place the ampoule into a plastic petri dish. Holding the bottom of the ampoule firmly on the tabletop inside the secondary container (petri dish), snap open the ampoule. Place the stem in the petri dish.
- 3 Using a clean glass pasteur pipette and bulb. Transfer the contents of the ampoule into a 100 mL volumetric flask. Rinse the ampoule, stem, and the pasteur pipette repeatedly with 2-3 mL of the 0.1 N HCL diluent. Check the secondary containment (petri dish) for signs of spillage. If a spill did occur, rinse the dish repeatedly into the 100 mL volumetric flask.
- 4 Using a pasteur pipette to deliver the last few milliliters of diluent, fill the 100 mL volumetric flask to the marker with 0.1 N HCL (room temperature - 20° C).
- 5 Cap the volumetric flask and mix thoroughly. Label the volumetric flask "Primary Standard" and reference this procedure.
- 6 Clear the laboratory table top of unnecessary glassware and materials. Dispose of contaminated materials.

There is now 100 mL of Primary Standard working solution to be dispensed among the various standard counting geometries.

### Reference Sample Standard Counting Geometries

- 1 Liquids, urine and water
  - a One 50 mL tissue culture flask (Fig. 1).
  - b One 50 mL conical centrifuge tube (Fig. 2).
  - c Two 10 mL vacutainer tubes (Fig. 3).

## 2 Air Filter Samples

- a Two charcoal filter papers, one large and one small (Fig. 4).
- b One canister type charcoal filter (Fig. 5).

## 3 Incinerator Ash

- a One petri dish packed with 100 grams of reagent grade calcium phosphate. Which simulates the density of bone ash (Fig. 6).

### Liquid Standards Preparation

#### 1 50 mL tissue culture flask.

- a Test the tissue culture flask for cap tightness using distilled water.
- b Using a 20 mL Volumetric pipette, transfer 20 mL of the primary standard solution to one of the tissue culture flasks. Wipe the pipette tip with a chemwipe to remove excess liquid from the exterior of the tip. Be careful that the pipette is filled and that there has been no tip loss during transfer. Holding the pipette tip vertically and above the dispensed fluid level allow the primary standard to dispense. Do NOT force residual liquid from the pipette. With the primary standard dispensed into the tissue culture flask, add 30mL of HCL diluent to fill the flask to the 50 mL mark. Label the flask and set it aside-upright in a secondary container.
- c The blank for the tissue culture flask geometry consists of one tissue culture flask filled with 50 mL of 0.1 N HCL.

#### 2 50 mL Conical Test Tube

- a Test the conical centrifuge tube and cap to assure a tight seal.
- b Using the same 20 mL volumetric pipette as above, dispense 20 mL of the primary standard solution into the 50 mL test tube, Bring the total volume up to 50 mL with diluent, seal the cap, label and stand it upright in a secondary container.
- c The blank for the conical test tube consist of one conical centrifuge test tube filled with 50 mL of 0.1 N HCL.

## 3 Vacutainer Tube

- a Using two red-top vacutainer tube (approximately 10 mL volume each), pop the top to relieve the vacuum. Rinse the tubes with diluent and remove the vacutainer labels. Rinse the caps and allow the assembly to dry. Place the clean dry vacutainer tubes into a test tube rack and select the 5 mL volumetric pipette. Dispense exactly 5 mL of primary standard solution into each tube. Replace the caps and label the tubes. A syringe and 22-gage needle can be used to reestablish the vacuum within the tube. Wrap the cap and neck of the vacutainer tube with parafilm and label the tubes. Keep all tubes upright and avoid cap contamination.
- b The blank should consist of one red-top vacutainer filled with 5 mL of 0.1 N HCL and the vacuum reestablished.

## 4 Air Filter Samples

- a Two charcoal filter papers ( one large and one small) are centered individually into separate petri dishes. Impregnate each filter with 5 mL of the primary standard drop by drop. It will be necessary to allow the solution to dry (using a heat lamp) in order to confine the standard to the filter paper alone.
- b Prepare two charcoal filter paper blanks, one large and one small. By centering each filter into separate petri dishes and impregnate each filter with 5 mL of 0.1 N HCL. Allow to dry as a above.
- c One canister type charcoal filter (centered in a petri dish) must be impregnated with 5 mL of the primary standard. Use a needle to perforate the surface membrane on one side of the filter. Apply the primary standard drop by drop again confining the standard to the filter.
- d Makeup one canister type charcoal filter blank. By centering the filter in a petri dish and impregnate it with 5 mL of 0.1 N HCL, allow to dry.

## 5 Crematory Ash

- a Prepare one petri dish packed with 100 grams of reagent grade calcium phosphate, to simulate the density of bone ash. Impregnate drop by drop

evenly throughout the calcium phosphate 20 mL of the primary standard. Allow to dry, then seal the petri dish using another petri dish. Seal the seam with epoxy cement.

- b Prepare a blank utilizing a petri dish prepared same as above except it shall be impregnated with 20 mL of 0.1 N HCL instead of the primary standard.

With the remaining primary standard (20 mL) prepare:

ONE extra culture flask containing 10 mL of the primary standard diluted to 50 mL.

ONE charcoal filter canister (2") containing 5 mL of the primary standard solution.

ONE 50 mL conical test tube with the remainder (slightly less than 5 mL) of the primary standard plus the rinse from the empty 100 mL volumetric flask. This is an unknown bounded between 4 and 5 mL. Fill to 50 mL with diluent.

Label all containers and use parafilm where possible to seal the tubes.

Check all containers for gross contamination by wipe testing. Always use a plastic bag to cover the primary standards while they are near the detectors. Do not store the standards in the counting chamber.

### Approximate Counting Times

#### **Germanium-Lithium Detector**

Original source ampoule, treated as a "point source", should be counted:

- 15 minutes at 2 cm
- 30 minutes at 4 cm
- 60 minutes at 8 cm
- 120 minutes at 12 cm

For the Reference Sample Geometries, or after the source has decayed significantly, use a counting time that will allow accumulation of approximately 20,000 net counts in each peak of interest to the calibration, if possible.

### Calibration Data analysis

Enter the net counts for each of nuclide in the net count column of the data acquisition spread-sheet, which will calculate the efficiency in counts/gamma.

For each calibration position and sample geometry, plot an efficiency vs. energy curve.

Compare the efficiency vs. energy curves to the same curves obtained in the previous calibration. If there is a significant difference, contact the detector manufacturer.