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REGULATORY GUIDE

DIRECTORATE OF REGULATORY STANDARDS

REGULATORY GUIDE 5.9

SPECIFICATIONS FOR Ge(Li) SPECTROSCOPY SYSTEMS FOR MATERIAL PROTECTION MEASUREMENTS

PART I: DATA ACQUISITION SYSTEMS

A. INTRODUCTION

Section 70.51, "Material Balance, Inventory, and Records Requirements," of 10 CFR Part 70, "Special Nuclear Material," requires, in part, that licensees authorized to possess at any one time more than one effective kilogram of special nuclear material establish and maintain a system of control and accountability such that the limit of error of any material unaccounted for (MUF), ascertained as a result of a measured material balance, meets established minimum standards. The selection and proper application of an adequate measurement method for each of the material forms in the fuel cycle is essential for the maintenance of these standards.

This is the first in a two-part series of guides which present specifications for lithium-drifted germanium, Ge(Li), gamma ray spectroscopy systems. This guidance applies to the selection of a special nuclear material (SNM) assay system which utilizes gamma ray spectroscopy for the quantitative determination of the SNM content and a qualitative determination of the radionuclide abundances. Within each of the guides in this series, Data Acquisition and Data Reduction, variations of a basic spectroscopy system are defined and individual specifications provided. The procedures for applying these systems to specific materials and the analysis of the reduced data is the subject of a later guide.

B. DISCUSSION

1. Background

Gamma ray spectroscopy systems have been used for the nondestructive assay (NDA) of various special nuclear material forms encountered in the fuel cycle both for quantitative determination of the special nuclear material content, and for the determination of radionuclide abundances. In addition to the NDA of bulk materials, gamma ray spectroscopy is used in the analysis of specially prepared, homogeneous laboratory samples.

There is no single gamma-ray spectroscopy system available which is satisfactory for all applications nor is there a standard which defines and specifies the type or types of systems to be used in each of the above applications. This guide defines and details the specifications for gamma ray spectroscopy data aquisition systems appropriate for special nuclear material assay.

The scope of this guide is limited to the consideration of Ge(Li) gamma ray spectroscopy systems. No discussion of thallium-activated sodium iodide, NaI(Tl), gamma ray systems is presented. In addition, no discussion of applications of gamma ray spectroscopy is presented. The measurement procedures (including calibration), analysis methods, inherent limitations, and overall precision and accuracy are specific to each application and are therefore the subject of separate application guides.

An elementary introduction to the concepts associated with the application of Ge(Li) spectroscopy to problems of nuclear material assay is available.[1] Descriptions of the physical processes of gamma ray detection, discussions of important instrumentation

* Indicates change from previous issue.

[1] L. A. Kull, "An Introduction to Ge(Li) and NaI Gamma-Ray Detectors for Safeguards Applications," ANL-AECA-103 (1973).

characteristics, and a step-by-step description of a simple assay problem are included in this document. Relevant information presented at a somewhat higher technical level, including nomenclature and definitions, is contained in two useful standards documents.[2,3] These describe detailed techniques for defining and obtaining meaningful performance data for Ge(Li) detectors and amplifiers. The glossary of technical terms found in both these standards documents will prove valuable to those unfamiliar with gamma-ray spectroscopic nomenclature.

Finally, there is a considerable amount of valuable background material published by the manufacturers of detectors and associated electronic hardware which is available from them on request.

2. Functional Description

A block diagram of those components of the Ge(Li) spectroscopy system which perform the data acquisition function in material protection measurements is shown in Fig. 1. The function of these components is first to convert the charge produced by the interaction of an incident gamma ray with the Ge(Li) detector into an amplified, analog electrical signal. The analog signal is then converted into digital information which can be stored, displayed, and otherwise processed by appropriate data reduction and analytical modules.

3. Types of Systems

There are three variations of the basic data acquisition system presented in this guideline. This variance in the basic configuration is the result of attempts to optimize each system to obtain specific assay information from certain types of material forms.

The three variations of the basic system are described below and will be referred to by Roman numeral in the remainder of the document. (For example, System II refers to paragraph II below.)

I. A moderate to high efficiency system having an energy resolution which is adequate for assays of materials for the fissile isotopes ^{241}Pu, ^{239}Pu, ^{235}U, and ^{233}U. It can also be used to perform assays of materials for fertile isotopes such as ^{232}Th and ^{238}U and to determine the "age" of plutonium samples from measurements of their americium-241 content. This system is used in those applications where NaI resolution is inadequate to accurately resolve the gamma ray lines of the isotopes of interest from those from an interfering background and where the lower efficiency Ge(Li) detector still provides sufficient sensitivity for practical assay work. The system is designed to measure gamma rays with energies greater than 120 keV.

II. A moderate to high efficiency system which can satisfy all the requisites for System I and which, in addition, has the improved energy resolution necessary to assay for the plutonium isotopes 238 through 241. This system is commonly used to determine the relative radionuclide abundances and is designed to measure gamma rays with energies greater than 120 keV.

III. A system designed specifically for low-energy gamma ray and X-ray spectroscopy (at gamma ray energies less than 200 keV) having an energy resolution adequate to perform quantitative and qualitative assays of specially prepared samples for the isotopes of plutonium (238-241) and uranium (235 and 238).

4. Equipment Acceptance Practices

Standard practices regarding the final acceptance of equipment are usually prescribed by individual companies, laboratories, or departments. However, some of the following procedures have been found to be useful in providing the user with the assurance that he will acquire equipment which will perform as expected in nuclear material assay applications.

Equipment descriptions (including the theory of operation) and instructional material covering operation, maintenance, and servicing of all electronic components should be supplied for individual components or complete systems. Such descriptions should include complete and accurate schematic diagrams for possible in-house equipment servicing. Carefully specified operational tests of system performance should be made at the vendor's facility and the original data supplied to the user before equipment delivery is scheduled, with final acceptance based on the user's own performance data taken at the user's facility.

It is necessary to have calibration sources on hand to verify the operational capabilities of the system. The following radioactive sources (with appropriate activities) will provide sufficient counting rates to perform the tests specified in the regulatory position:

^{60}Co–1-30 μCi
^{57}Co–1-10 μCi

C. REGULATORY POSITION

Lithium-drifted germanium, Ge(Li), gamma ray spectroscopy data acquisition systems meeting the operating specifications given below are considered adequate for use in special nuclear materials assay. The selection of a system meeting these specifications is considered necessary but not sufficient for accurate gamma ray spectroscopic assay requiring resolution better than obtainable with NaI. No guarantee of measurement quality as a result of the application of such systems should be assumed.

[2] "Test Procedure for Amplifiers and Preamplifiers for Semiconductor Radiation Detectors," IEEE Std 301-1969, The Institute of Electrical and Electronics Engineers, Inc. (1969).

[3] "Test Procedures for Germanium Gamma-Ray Detectors," IEEE Std 325-1971, The Institute of Electrical and Electronics Engineers, Inc. (1971).

The emphasis here is on the operating specifications related to the overall performance of the entire data acquisition system. Component specifications have been included in Appendix A to provide guidance in the selection of original or replacement components which are essential if adequate system performance is to be attained. The system operating performance should not be deduced from the component performances; overall system performance should be checked independently and compared to the operating specifications presented here.

1. Energy Resolution and Peak Shape

(Systems I, II, III) The energy resolution of the system should be measured according to the procedure specified in IEEE Standard 325-1971,[4] with the following additional stipulations: (1) the peaking time[5] for the shaping amplifier should be no greater than 4 μsec; (2) the total number of counts in the center channel of the peak should be no less than 10^4 counts; (3) the count rate during the measurement should be in the range 10^2 to 10^3 counts per second as measured with a total count rate meter. The full width of the peak at half maximum (FWHM) and full width at tenth-maximum (FWTM) are as defined in IEEE Standard 325-1971.[6] The full width at 1/50 maximum (FW.02M) is defined in a similar manner. The energy resolution and peak shape specifications for each of the systems (I, II, III) are given in Table 1 and the measured values should be no greater than those shown here. These values have been determined to be necessary for the applications defined in B.3. above.

2. Detection Efficiency

(Systems I, II) The full energy peak efficiency (in percent) is defined relative to the full energy peak efficiency of a 3 in. x 3 in. NaI(Tl) scintillation detector for 1.33 MeV gamma rays (^{60}Co) at a source-detector distance of 25.0 cm. The detailed procedures for determining the efficiency in accordance with this definition are presented in IEEE Standard 325-1971.[7]

The efficiency required for specific assay applications should be determined by estimating the gamma ray intensity at the detector from a sample of known strength and the counting rates required to collect a statistically significant number of counts under the spectrum peaks of interest in a reasonable period of time. Estimates should be corrected for sample-to-detector distance and the effects of absorbing materials placed between the sample and detector. Whenever possible, it is advisable to make preliminary measurements on the samples under consideration with an available detector, and the efficiency of the optimal detector determined by extrapolating the measured results. A nominal estimate of the detector efficiency (as defined above) required for most applications is approximately 8%; however, detectors with efficiencies in the range of 5% to 20% are in use for nuclear material assays. (To assist in providing some perspective here, an 8% detector as specified above has an active volume of about 40 cc while 5 to 20% detectors have volumes of about 25 cc to 110 cc, respectively. An 8% detector has absolute detection efficiencies of about 15 x 10^{-4} @ 185 keV, 4.5 x 10^{-4} @ 411 keV, and 0.96 x 10^{-4} @ 1.33 MeV at a source-detector separation of 25 cm.)

(System III) The method described above for determining the detection efficiency with a high energy gamma ray source is not relevant for detectors used in low-energy gamma ray spectroscopy. Instead, it is more appropriate to specify, (1) the active volume of the detector and (2) the maximum effect of absorbing materials (absorbing materials include detector surface "dead layers," gold surface plating, and the end cap window of the cryostat). The following specifications are therefore given for the low-energy gamma ray system:

a. detector volume—1.0 to 1.5 cc
b. drift depletion depth—0.5 to 0.7 cm
c. layers of absorbing material between the radiation source and the active volume of the detector must be thin enough so that the 14.4 keV peak from a ^{57}Co source is at least 5 times the continuum background under the peak.[8]

3. Count Rate Capabilities

The following specifications are related to a system's ability to maintain adequate energy resolution at high count rates.

(Systems I, II) The system should be capable of operating at a total counting rate of 10^4 cps from a ^{60}Co source (as measured with a total count rate meter) with less than a 10% relative increase in the 1.33 MeV peak width at 1/10 the maximum peak height (FWTM) as compared to the FWTM value measured at 10^2 to 10^3 cps.

(System III) The system should be capable of operating at a total counting rate of 5 x 10^3 cps from a ^{57}Co source (as measured with a total count rate meter)

[4] IEEE Std 325-1971, op. cit., Section 4.

[5] Peaking time—the time required for a pulse to reach its maximum height. Peaking times can be easily measured with an oscilloscope and are less susceptible to misinterpretation than are RC time constants. The relationship between RC time constants and peaking time varies as their is no standard method for defining RC time constants in semi-Gaussian shaping networks.

[6] IEEE Std 325-1971, op. cit., Section 3.

[7] Ibid., Section 5.2.

[8] Care should be taken to ensure that the ^{57}Co source encapsulation is thin enough (<100 mg/cm^2 plastic or the equivalent) so that self absorption in the source itself is not significant.

with less than a 10% relative increase in the FWHM and FW.02M of the 122 keV peak as compared to the values obtained at 10^3 cps.

4. Peak-to-Compton Ratio

(Systems I, II) The peak-to-Compton ratio for the 1.33 MeV peak from a ^{60}Co source as defined in IEEE Standard 325-1971[9] should be greater than the values specified in Table 2 for corresponding detector efficiencies.

(System III) This specification is not applicable.

5. Linearity and Stability

(Systems I, II, III) The integral non linearity of the data acquisition system's energy calibration should be less than 0.2% over the top 95% of the ADC range. The system nonlinearity should be measured using a set of well-known gamma ray sources and the procedure described in the literature.[10,11]

The long-term stability requirement for the system's zero channel and gain should be defined as follows: the drift in the position of a spectrum peak from a calibration source should be less than 0.1% (compared to full scale) in a 24-hour period at constant room temperature. (For example, the centroid of a calibration peak placed in approximately channel 4000 of a 4096 channel spectrum should not vary in position by more than 4 channels over a 24-hour period.) The temperature coefficient of the system's zero channel and gain should be less than 0.02%/°C in the temperature range from 0° to 50°C.

[9] IEEE Std 325-1971, op. cit., Section 3.4.

[10] R. C. Greenwood, R. G. Helmer, and R. G. Gehrke, "Precise Comparison and Measurement of Gamma-Ray Energies with a Ge(Li) Detector I. 50-420 keV," Nucl. Instr. and Methods 77, 141 (1970).

[11] R. G. Helmer, R. C. Greenwood and R. G. Gehrke, "Precise Comparison and Measurement of Gamma-Ray Energies with a Ge(Li) Detector II. 400-1300 kev," Nuclear. Instr. and Methods *96*, 173 (1971.)

APPENDIX A

COMPONENT SPECIFICATIONS

1. Detector Crystal Geometry

(Systems, I, II) The detector should be of the closed end, coaxial drift, right circular cylinder type; this configuration has the maximum fraction of usable active volume for detectors of moderate to high efficiency. The crystal diameter should be approximately equal to its length to minimize any unusual efficiency vs. geometry effects. The active volume of the detector should comprise at least 90% of the total crystal volume with the undrifted core diameter kept as small as economically possible. This maximizes the probability that a gamma-ray interaction will appear in the full energy peak of the spectrum. (Note: The specification on peak-to-Compton ratio given in Section C.4 is directly related to the crystal's active/total volume atio.)

(System III) The detector should be of the planar type. Small detectors of this configuration offer the best resolution available for low-energy gamma rays. Operating specifications are given in Section C.2 that define the allowable thickness of detector surface "dead layers" which absorb low-energy gamma rays before they interact in the detector's active volume.

(Systems I, II, III) Methods for specifying the physical size for the detector crystals are covered in Section C.2.

2. Detector Mounting and Cryostat Description

(Systems I, II, III) There are four detector cryostat configurations which are typically available: (1) right angle dip-stick, (2) upright dip-stick, (3) gravity feed, and (4) side entry (portable). Of these, the right angle dip-stick is widely used for Systems I and II and the upright dip-stick for System III; the configuration selected should be that considered to be most useful for a specific application. For reliable operation, the vacuum in the detector housing should be maintained by a zeolite getter. It is recommended that the liquid nitrogen Dewar have a minimum capacity of about 30 liters and a holding time of at least 10 days. The Dewar should have a connection which allows replenishment of the liquid nitrogen supply without removing the cryostat. A separate high-voltage input to the cryostat housing should be provided in the event it is necessary or desirable to apply a detector bias which exceeds the rating of the preamplifier's high-voltage input. It is recommended that the high-voltage input be clearly marked and located at least 2.0 cm from the preamplifier signal output. The distance between the detector's front surface and the window in the housing should be less than or equal to 1.0 cm to allow one to achieve minimal detector-sample separations when necessary.

3. Preamplifiers

(Systems I, II) In many cases preamplifiers compatible with nuclear material spectroscopy applications are purchased in combination with a Ge(Li) crystal as a package. The detector specifications therefore relate to the detector-preamplifier combination; however, the following additional specifications should be included in the selection of an optimal system. A charge sensitive preamplifier should be mounted on the cryostat near the detector. The field effect transistor (FET) in the first stage of the preamplifier should be operated at room temperature (~300°K).[12] The detector should be d.c. coupled (as opposed to capacitively coupled) to the gate of the input stage of the preamplifier for better energy resolution.

The following procedures are recommended to minimize the probability of destroying the FET due to detector warmup or high-voltage transients. Positive high voltage should be used, and there should be at least one filter section placed in the high-voltage system internal to the cryostat. At least one filter should also be placed external to the cryostat to reduce the possibility of short circuiting due to condensate formation on the internal filter. The total RC time constant of the filter network should be at least 30 seconds.

(System III) Same as above for Systems I and II except that the FET in the preamplifier's first stage should be located within the cyrostat and operated at liquid nitrogen (LN) temperature. An LN cooled FET is required to achieve the excellent energy resolution characteristics of this system.

4. Main Amplifier

(Systems I, II, III) A main amplifier with adjustable gain should include unipolar, semi-Gaussian pulse shaping networks with adjustable time constants corresponding to peaking times between 1 and 8 μsec. (1 to 4 μsec peaking times are typically used for Systems I and II while peaking times as long as 8 μsec could be used in System III.) This choice of amplifier provides minimum resolving time for a given energy resolution and sufficient flexibility to optimize the amplifier characteristics for most counting conditions. Nominal specifications to aid in identifying this class of amplifiers, commonly referred to as spectroscopy amplifiers, include the following: linear range 0 to 10V, integral nonlinearity <0.05%, temperature stability <100 ppm gain shift/°C, and thermal noise <5μV rms

[12] (System II only) The preamplifier's first stage FET may be located within the cryostat and operated at liquid nitrogen temperatures, but in order to facilitate possible FET replacement, it is recommended that a detector be selected which attains adequate energy resolution with an uncooled FET

referred to the input for 4 μsec peaking times (the noise level varies inversely with the peaking time). The main amplifier should be a standard NIM[13] module.

At counting rates greater than ~10^3 cps, problems such as degradation of the energy resolution resulting in a loss of counts in the spectrum peaks begin to occur. These effects are due to the overlap of portions of two or more pulses in time and to baseline fluctuations. The magnitude of these effects can be minimized by the inclusion of the following features in the amplifier's design: (1) a baseline restorer (BLR) circuit at the amplifier output and (2) pole-zero cancelled coupling networks. The BLR circuit should be adjustable for both low and high counting rate conditions.[14]

5. Analog to Digital Converter (ADC)

(Systems I, II, III) The ADC should be capable of digitizing pulse amplitudes from the amplifier in the range of 0 to 10 volts in at least 4096 channels. The frequency of the internal clock should be at least 50 megahertz to handle high counting rates with nominal ADC dead time losses. The integral nonlinearity should be less than 0.15% over the top 95% of full scale and the differential nonlinearity should be less than 1.0% over the top 95% of full scale for semi-Gaussian pulses with peaking times of 1 to μsec. These linearity specifications are not stringent, but are adequate to enable identification of unknown peaks which may appear in a spectrum.

The short-term zero channel and gain drifts should be $\leq$ .01%/°C and $\leq$ .02%/°C, respectively (the percentage refers to full scale), in the temperature range from 0° to 50°C. For long term stability, the peak from a stable pulser should not shift by more than one channel over a 24-hour period for a line voltage of 115V ± 10%, 50-65 Hz, and at constant room temperature. (Note: The ADC drift and linearity specifications are closely related to the overall system stability and linearity operating specifications described in Section C.5.)

The ADC should be capable of being DC coupled to the main amplifier in order that BLR circuits can be used. A digital offset capability in the ADC is recommended. (Note: In some systems the ADC is an integral part of a multichannel analyzer, a unit which also performs the functions of data storage, display, and sometimes rudimentary analysis. These latter functions are taken up in Part 2 of this series. In multichannel analyzer systems, however, the ADC function is usually specified separately and can be compared with the above recommendations.)

(System I) For certain applications where energy resolution is definitely not critical, all the ADC specifications above are applicable with the exception that a 1024 channel capacity with a 1024 digital offset may be adequate to provide a sufficiently small energy interval per channel (keV/channel) to cover a limited energy range of interest. It should be emphasized, however, that this choice may restrict the effective use of the system for other applications.

6. Power Supplies

(Systems I, II, III) The system power supplies (detector high voltage, preamplifier, and NIM bin) should be capable of operating the system within the operating specifications listed in Section C.1 when supplied with 115 volts (± 10%) at 50 to 65 hertz (at constant room temperature). The detector bias power supply should have an adjustable output that is short circuit protected with automatic power restoration after removal of the short. The maximum output voltage is determined by detector requirements; 5 kilovolts is sufficient for most applications.

[13] NIM-Nuclear Instrument Module, see USAEC Technical Information Document, Standard Nuclear Instrument Modules, Revision 3, TID-20893 (1969).

[14] For more details on BLR circuits see V. Radeka, "Effect of 'Baseline Restoration' on Signal-to-Noise Ratio in Pulse Amplitude Measurements," Rev. Sci. Instr. *38*, 1397 (1967).

TABLE 1

ENERGY RESOLUTION AND PEAK SHAPE SPECIFICATIONS

SYSTEM I

Calibration Source Gamma Ray Energy	FWHM (keV)	FW.02M/FWHM
^{57}Co- 122 keV	1.6	less than 2.7
^{60}Co–1332 kev	2.5	less than 2.8

SYSTEM II

^{57}Co–122 keV	1.0	less than 2.7
^{60}Co–1332 keV	1.9	less than 2.8

SYSTEM III

^{57}Co–5.9 keV (Fe X-ray)	0.32	less than 2.5
^{57}Co–122 keV	0.55	less than 2.5

TABLE 2

PEAK-TO-COMPTON RATIO VS. DETECTOR EFFICIENCY

Detector Efficiency (As defined in Section C.2)	Minimum Peak-to-Compton Ratio
5%	20:1
10%	30:1
15%	35:1
20%	38:1

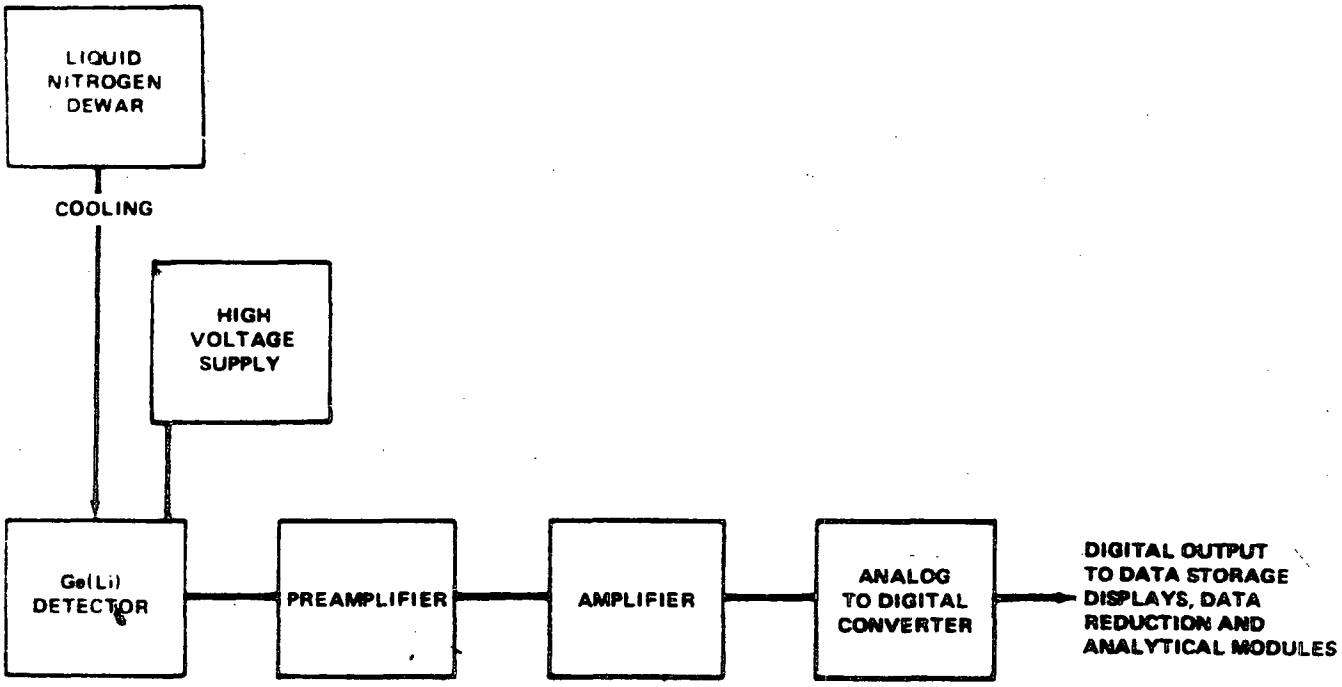

Figure 1.–BLOCK DIAGRAM OF A Ge(Li) DATA ACQUISITION SYSTEM