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U.S. NUCLEAR REGULATORY COMMISSION

REGULATORY GUIDE

OFFICE OF NUCLEAR REGULATORY RESEARCH

REGULATORY GUIDE 5.9
(Task SG 042-2)

GUIDELINES FOR GERMANIUM SPECTROSCOPY SYSTEMS FOR MEASUREMENT OF SPECIAL NUCLEAR MATERIAL

A. INTRODUCTION

Section 70.51, "Material Balance, Inventory, and Records Requirements," of 10 CFR Part 70, "Domestic Licensing of 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 so that the standard error (estimator) of any inventory difference, 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.

Many types of nondestructive assay (NDA) measurements on special nuclear material (SNM) can involve, or even require, a high-resolution gamma ray spectroscopy system. This guide is intended both to provide some general guidelines acceptable to the NRC staff for the selection of such systems and to point out useful resources for more detailed information on their assembly, optimization, and use in material protection measurements.

Any guidance in this document related to information collection activities has been cleared under OMB Clearance No. 3150-0009.

B. DISCUSSION

1. BACKGROUND

Gamma ray spectroscopy systems are used for NDA of various special nuclear material forms encountered in the nuclear fuel cycle, both for quantitative determination of the SNM content and for the determination of radionuclide abundances.

Applications of high-resolution gamma ray spectroscopy have multiplied greatly in recent years. The samples encountered range from fresh fuel rods and reprocessing solutions to boxes and cans of uncharacterized waste material. Measurement conditions also vary widely from controlled laboratory environments to the unpredictable plant environment that can be hostile to the measurement equipment and can often contribute serious background interferences to the spectral data. As a result, there is no single gamma ray assay system that can be effective in all cases. The system chosen for a particular NDA task must therefore be determined from careful consideration of all factors that may affect the measurement and of the requirements for the precision and accuracy of the assay.

The scope of this guide is limited to the consideration of high-resolution gamma ray spectroscopy with lithium-drifted germanium, Ge(Li), or high-purity germanium, HPGe (also referred to as intrinsic germanium, IG), detectors. No discussion of thallium-activated sodium iodide, NaI(Tl), or lithium-drifted silicon, Si(Li), gamma ray systems is presented. In addition, no discussion of specific NDA applications of gamma ray spectroscopy is provided. The measurement procedures (including calibration), analysis methods, inherent limitations, and overall precision and accuracy attainable are specific to each application and are therefore the subject of separate application guides. Guidelines for measurement control, calibration, and error analysis of NDA measurements are dealt with in detail in Regulatory Guide 5.53, "Qualification, Calibration, and Error Estimation Methods for Nondestructive Assay," which endorses ANSI N15.20-1975, "Guide to Calibrating Nondestructive Assay Systems."[1] ANSI N15.20-1975 was reaffirmed in 1980.

All of the major commercial vendors of Ge(Li) and HPGe detectors and the associated electronics maintain up-to-date documentation on the specifications of currently available equipment, as well as a variety of useful and informative notes on applications. This literature is available

*The substantial number of changes in this revision has made it impractical to indicate the changes with lines in the margin.

[1]Copies of this standard may be obtained from the American National Standards Institute, Inc., 1430 Broadway, New York, New York 10018.

from the manufacturers upon request, and the potential customer may use this literature as a source of the most current information on the highest quality systems available.

Finally, the potential user ought to consult with those individuals currently active in the field of nondestructive assay of special nuclear material and seek their advice in the particular assay problem being considered.

2. BIBLIOGRAPHIC INFORMATION

An annotated bibliography is included in this regulatory guide to provide more detailed information on spectroscopy systems and their use.

Elementary introductions to the concepts associated with the application of high-resolution gamma ray spectroscopy to problems of nuclear material assay are available in Augustson and Reilly and in Kull. These works discuss the physical processes of gamma ray detection and important instrumentation characteristics. More advanced discussion of gamma ray detectors and associated electronics may be found in Knoll and in Adams and Dams. A thorough treatise on the associated electronics is available in Nicholson. In addition, extensive discussion of a variety of NDA techniques and the implementation of some of these techniques with high-resolution gamma ray spectroscopy may be found in Sher and Untermeyer, in Rogers, and in Reilly and Parker. Detailed descriptions of detector efficiency and energy calibration procedures are available in section D of Knoll and also in Hajnal and Klusek; in Hansen, McGeorge, and Fink; in Hansen et al.; and in Roney and Seale.

Relevant technical information beyond the introductory level, including nomenclature and definitions, is contained in three useful standards of the Institute of Electrical and Electronics Engineers, ANSI/IEEE Std 301-1976, "Test Procedures for Amplifiers and Preamplifiers for Semiconductor Radiation Detectors for Ionizing Radiation,"[2] ANSI/IEEE Std 325-1971, "Test Procedures for Germanium Gamma-Ray Detectors"[2] (reaffirmed in 1977), and ANSI/IEEE Std 645-1977, "Test Procedures for High-Purity Germanium Detectors for Ionizing Radiation,"[2] which supplements ANSI/IEEE Std 325-1971. These describe detailed techniques for defining and obtaining meaningful performance data for Ge(Li) and HPGe detectors and amplifiers.

3. FUNCTIONAL DESCRIPTION

A block diagram of a typical high-resolution gamma ray spectroscopy system is shown in Figure 1. In such a system, the solid state Ge(Li) or HPGe detector converts some or all of the incident gamma ray energy into a proportional amount of electric charge, which can be analyzed by the subsequent electronics. The detector output is converted into an analog voltage signal by the preamplifier, which is an integral part of the detector package. The preamplifier signal is further amplified and shaped and is then converted into digital information that can be stored, displayed, and otherwise processed by the data reduction and analytical components of the system.

4. TYPES OF SYSTEMS

High-resolution gamma ray spectroscopy systems are distinguished primarily by the type (p-type or n-type) and the configuration (planar or coaxial) of detector used. For assay applications involving the measurement of low-energy gamma radiation (i.e., energies below approximately 200 keV), a thin planar HPGe or Ge(Li) crystal is most appropriate. A coaxial detector crystal with a larger volume is much better suited for higher energy gamma ray measurements (i.e., for energies above approximately 120 keV). The distinction between these two types of detectors is not sharp. For instance, there may be some applications above 120 keV in which a planar detector would be useful to render the system less sensitive to interferences from ambient high-energy gamma radiation.

It should be noted that Ge(Li) detectors have no real advantage over HPGe detectors with comparable performance specifications. In addition, Ge(Li) detectors require constant liquid nitrogen (LN) cooling, even when not in operation. HPGe detectors are, of course, also operated at LN temperature, but they can be stored at room temperature. This is an advantage to potential users who may have extended plant shutdowns. It also prevents complete loss of a detector due to operator procedure error, which can happen with a Ge(Li) detector when LN cooling is not continuously maintained. This added convenience and the greater ruggedness of the HPGe detectors make them especially attractive for in-plant NDA applications.

5. EQUIPMENT ACCEPTANCE PRACTICES

Equipment descriptions and instructional material covering operation, maintenance, and servicing of all electronic components are supplied by the manufacturer for all individual modules or complete systems. Such descriptions should include complete and accurate schematic diagrams for possible in-house equipment servicing. Complete operational tests of system performance are to be made at the vendor's facility, and the original data are supplied to the user upon delivery of the equipment. Extensive performance testing of all systems by the user is generally not necessary.[3] However, qualitative verification of selected equipment performance specifications and detector resolution is recommended.

It is necessary to have calibration sources on hand to verify the operational capabilities of the system. The following radioactive sources (with appropriate activities)

[2] Copies may be obtained from the Institute of Electrical and Electronics Engineers, Inc., 345 East 47th Street, New York, New York 10017.

[3] Although the quality control and preshipment testing procedures of the commercial vendors of detectors and associated electronics have improved and are quite dependable, some user verification of the specifications claimed by the manufacturer is strongly recommended.

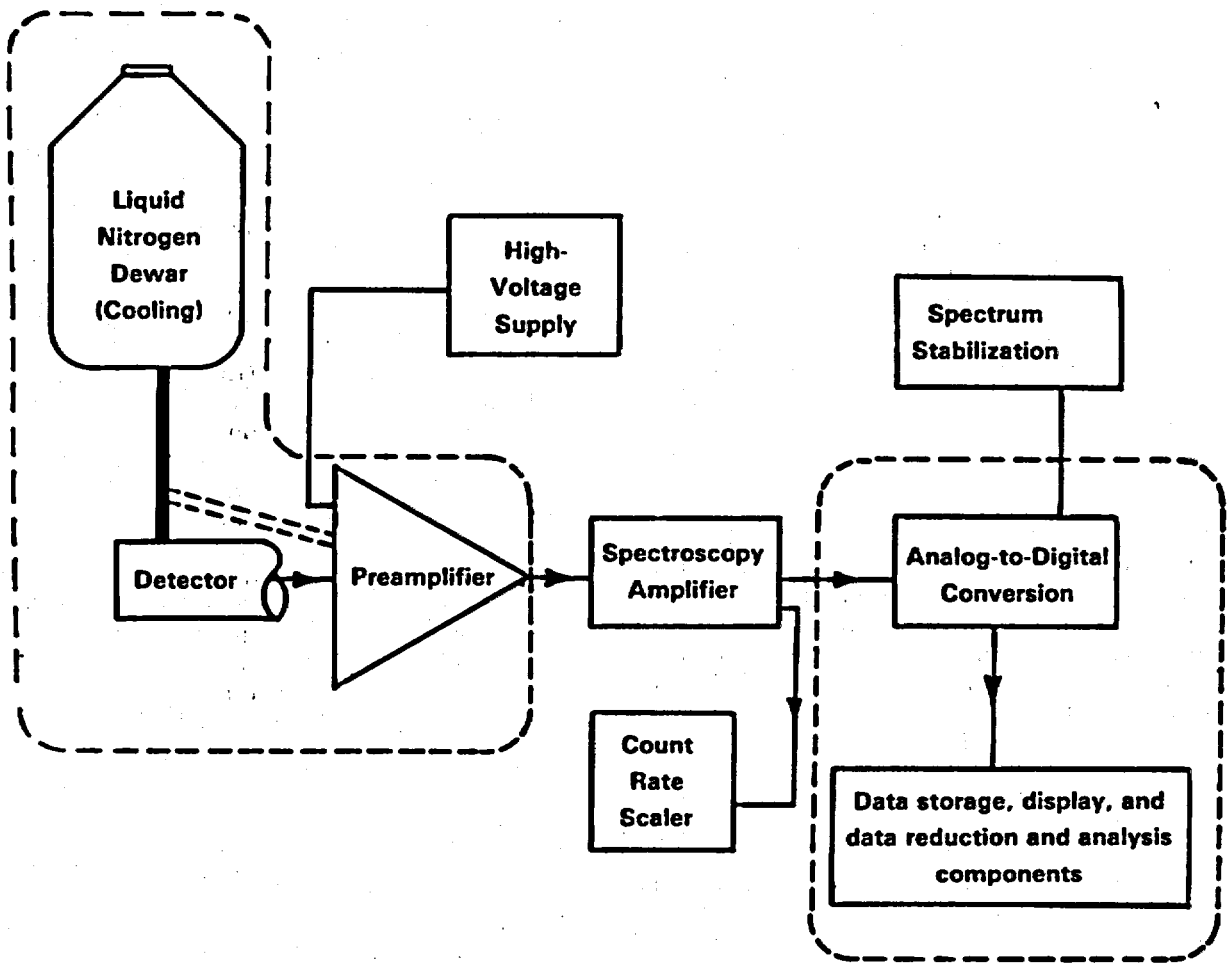

FIGURE 1

A block diagram of a typical setup of a high-resolution gamma ray spectroscopy system. The dashed boxes indicate which sets of modules are usually packaged as one component in commercially available systems. Liquid nitrogen cooling of the detector is required for proper operation of the system, but the field-effect transistor (FET) in the preamplifier input stage may or may not be cooled, depending upon the type of detector used and the energy resolution desired. A scaler is shown connected to the main amplifier, a common method of monitoring the total system count rate. For long-term data acquisition, spectrum stabilization is recommended, and the method is indicated here by a stabilizer module in communication with the analog-to-digital converter (ADC).

will provide sufficient counting rates to verify the energy resolution specifications of the manufacturer and to carry out any other performance tests desired by the user:

^{60}Co 10-30 μCi, Gamma ray energies: 1173, 1332 keV
^{57}Co 1-10 μCi, Gamma ray energies: 14, 122, 136 keV

C. REGULATORY POSITION

Ge(Li) or HPGe gamma ray spectroscopy data acquisition systems meeting the general guidelines outlined briefly below are considered more than adequate for use in SNM assay requiring resolution better than that obtainable with NaI detectors. The potential user should select the detector and associated electronics that meet the needs of the particular assay task required, with careful consideration of all factors that could affect the quality of the assay.

1. DETECTOR PERFORMANCE

Excellent performance, routinely available in coaxial germanium detectors, may be represented by energy resolutions (FWHM)[4] of approximately 1.7 keV at 1332 keV (^{60}Co) and approximately 0.7 keV at 122 keV (^{57}Co) for detectors with efficiencies up to 20 percent.[5] The full width at 0.1 maximum (FWTM) for such detectors is typically up to 1.9 times the FWHM. For these higher efficiency detectors, "peak-to-Compton ratios" are usually quoted in the range of 25 to 40. These ratios are strong functions of resolution, efficiency, and exact detector crystal geometry, and no typical values can be given without knowledge of all of these parameters. Coaxial detectors with this kind of resolution will usually have cooled field-effect transistor (FET) preamplifiers and an energy-rate capability of approximately 50,000 MeV/sec.[6] Room temperature preamplifiers have somewhat worse resolution but have rate capabilities on the order of 150,000 MeV/sec.

The resolution of planar detectors is a stronger function of the crystal size and shape than that of coaxial detectors, so representative resolutions cannot be given over a range of sizes. As an example from the middle of the range of sizes usually offered, an excellent 2 cm^3 planar detector (i.e., 2 cm^2 front face area x 1 cm thick) would have a resolution of approximately 0.5 keV at 122 keV (^{57}Co) and 0.21 keV at 5.9 keV (Mn X-ray from ^{55}Fe decay). Planar detectors will always have LN-cooled FET preamplifiers in order to achieve the excellent resolution of these systems. The preamplifier feedback loop may be either pulsed optical or resistive,[7] and the system will have fairly modest rate capabilities in the range of 5000 MeV/sec.[6] It is important to decouple the detector from noisy mechanical environments to avoid microphonic pickup.

2. ELECTRONICS PERFORMANCE

For ease of use, maintenance, and replacement of the components in a high-resolution gamma ray spectroscopy system, the electronic components should be standard nuclear instrument modules (NIM) (Ref. 1), with the possible exception of the pulse-height analysis (i.e., multichannel analyzer) components. Pulse signals should be transmitted from module to module in shielded coaxial cable to minimize the effects of possible electronic noise from nearby machinery at the measurement site. The cables should have a characteristic impedance that matches the terminations used in the NIM modules (generally 93 ohms).

The system power supplies (detector high voltage, preamplifier, and NIM bin) should be capable of operating the system within the operating specifications when supplied with 115 volts (±10 percent) at 50 to 65 hertz (at constant room temperature). The power supplied for the detection system should be stabilized against voltage shifts in order to maintain resolution. The output voltage of the detector bias supply is determined by the detector requirements; 5 kilovolts is sufficient for most applications.

The main amplifier, commonly referred to as the spectroscopy amplifier, should have variable gain and pulse-shaping controls for maximum setup flexibility. Most high-quality amplifiers are equipped with baseline restoration and pole-zero cancellation circuits (Ref. 2), which greatly improve the resolution that can be achieved on a routine basis. Baseline restoration is essential for assay situations in which count rates in excess of several kilohertz are anticipated. Pulse pileup suppression is also a useful feature, if available; it may be found in some spectroscopy amplifiers and even in separate NIM modules designed for that purpose.

Electronic components should be obtained with state-of-the-art linearity and temperature sensitivity. Maintenance of long-term gain stability may require the use of a spectrum stabilizer. Centroid variations of a stabilization peak of less than one channel in a 4096-channel spectrum are achievable with commercially available stabilizer modules. Stabilization peaks can be provided either by a pulser or by a radioactive source. Generally, a radioactive source is preferred because it contributes less distortion to the gamma ray spectrum and has a stable (although decaying) emission rate. Furthermore, stabilization peaks from natural sources may be obtained from existing peaks in the assay spectrum itself, which simplifies the assay

[4] The full width of the gamma ray photopeak at half of its maximum height (FWHM) is defined in ANSI/IEEE Std 301-1976.

[5] 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 1332-keV gamma rays (^{60}Co) at a source-to-detector distance of 25 cm. The detailed procedures for determining the efficiency in accordance with this definition are presented in Section 5.2 of ANSI/IEEE Std 301-1976.

[6] Counting rate capabilities, expressed in MeV/sec, denote the maximum charge-to-voltage conversion rate of which the preamplifier is capable. For ^{60}Co, a 50,000-MeV/sec rate capability corresponds to a pulse counting rate limitation of approximately 80,000 counts/sec. For ^{57}Co, a 5000-MeV/sec rate capability also corresponds to a pulse rate limitation of approximately 80,000 counts/sec. Of course, nuclear material assays should be performed at count rates well below these limiting values in order to minimize rate-related losses from pulse pileup and dead time.

[7] Feedback methods for charge-sensitive preamplifiers are discussed thoroughly in Chapter 5 of Reference 2.

setup. Dead-time and pileup corrections may also be performed using a pulser or a separate radioactive source fixed to the detector. The latter method is preferred for the reasons stated above.

3. SYSTEM SELECTION AND USE

The detailed requirements and constraints of a particular measurement situation will cause wide variation in the optimum choice of systems, even within a fairly well-defined application. For example, a requirement for high throughput may dictate higher efficiency detectors and highly automated data acquisition electronics. Anticipated interferences from uranium, thorium, or fission products may make the best possible system resolution the most important consideration. Severe operating environments may make the use of digital stabilization highly desirable. Constraints of space and location could dictate an unusually small LN dewar with automatic filling capacity. The list of such considerations in a given situation can be long, and each situation should be considered carefully and individually in order to achieve a system that can acquire the required measurement data.

Beyond the choice of data acquisition systems, many other factors influence the successful use of gamma ray spectroscopy in quantitative assay measurements. Some of these are:

a. **Gamma Ray Signatures:** The energies and intensities of the relevant gamma rays place fundamental restrictions on the sensitivity, precision, and accuracy of any assay. The range of gamma ray energies of interest also determines the type of gamma ray detector appropriate for optimum efficiency.

b. **Full-Energy Peak Area Determination:** The procedure for extracting this fundamental information from the spectral data will be determined by the complexity of the gamma ray spectra as well as the intensity and complexity of the gamma ray background at energies near the peaks of interest.

c. **Gamma Ray Attenuation by the Samples and Surrounding Materials:** Corrections for this effect are essential for accurate assays. The importance of this correction will increase as the gamma ray energies of interest decrease and the absorptive power of the sample and surrounding materials increases.

All of this emphasizes that by far the most important factor in choosing an appropriate data acquisition system, in implementing proper assay procedures, and in supervising the assay operations is a highly competent person, preferably experienced in gamma ray spectroscopy and its application to assay measurements of special nuclear materials. Such a person, with the assistance of the existing literature and of others in the gamma ray field, will be able to consider a particular application in detail and choose an appropriate detector and electronics to create a data acquisition system that is well suited to the required assay task.

REFERENCES

1. L. Costrell, "Standard Nuclear Instrument Modules," U.S. Atomic Energy Commission, TID-20893, Revision 3, 1969.

2. P. W. Nicholson, *Nuclear Electronics*, John Wiley and Sons, New York, 1974.

BIBLIOGRAPHY

Adams, F., and R. Dams, *Applied Gamma-Ray Spectroscopy*, Pergamon Press, New York, 1970.

This work provides a comprehensive coverage of background material pertinent to the gamma ray spectroscopist. Considerable information is provided on both NaI and Ge detectors.

Augustson, R. H., and T. D. Reilly, "Fundamentals of Passive Nondestructive Assay of Fissionable Material," Los Alamos Scientific Laboratory, LA-5651-M, 1974.

This manual contains helpful introductory descriptions of NDA applications of gamma ray spectroscopy, as well as some discussion of gamma ray detection systems.

Hajnal, F., and C. Klusek, "Semi-Empirical Efficiency Equations for Ge(Li) Detectors," *Nuclear Instruments and Methods*, Vol. 122, p. 559, 1974.

Hansen, J., J. McGeorge, and R. Fink, "Efficiency Calibration of Semiconductor Detectors in the X-Ray Region," *Nuclear Instruments and Methods*, Vol. 112, p. 239, 1973.

Hansen, J., et al., "Accurate Efficiency Calibration and Properties of Semiconductor Detectors for Low-Energy Photons," *Nuclear Instruments and Methods*, Vol. 106, p. 365, 1973.

Knoll, G. F., *Radiation Detection and Measurement*, John Wiley and Sons, New York, 1979.

This book provides extensive discussion of all types of radiation detection systems, including high-resolution gamma ray spectroscopy systems. In particular, Section D deals exclusively with solid state detectors, and Section F is devoted to detector electronics and pulse processing.

Kull, L. A., "An Introduction to Ge(Li) and NaI Gamma-Ray Detectors for Safeguards Applications," Argonne National Laboratory, ANL-AECA-103, 1974.

P. W. Nicholson, *Nuclear Electronics*, John Wiley and Sons, New York, 1974.

This is an extensive treatise on electronics systems associated with high-resolution detectors. Detailed descriptions are given of detector preamplifiers, pulse shaping, rate-related losses, pulse-height analysis, and spectral resolution.

Reilly, T. D., and J. L. Parker, "Guide to Gamma-Ray Assay for Nuclear Material Accountability," Los Alamos Scientific Laboratory, LA-5794-M, 1975.

This report briefly covers the principles involved in using gamma ray spectroscopy in the quantitative assay of SNM and attempts to describe both capabilities and limitations of gamma ray assay techniques. The report also includes a description of procedures for determining plutonium isotopic ratios.

Rogers, D. R., "Handbook of Nuclear Safeguards Measurement Methods," Nuclear Regulatory Commission, NUREG/CR-2078, 1983.

Chapter 5, "Passive Nondestructive Assay Methods," contains descriptions of many applications of high-resolution gamma ray spectroscopy, as well as many references to original papers and reports.

Roney, W., and W. Seale, "Gamma-Ray Intensity Standards for Calibrating Ge(Li) Detectors for the Energy Range 200-1700 keV," *Nuclear Instruments and Methods*, Vol. 171, p. 389, 1980.

Sher, R., and S. Untermeyer, *The Detection of Fissionable Materials by Nondestructive Means*, American Nuclear Society Monograph, 1980.

This relatively short book summarizes the principles of most nondestructive assay methods and briefly describes many typical applications, including those of high-resolution gamma ray spectroscopy. Chapters 3 and 5 are of particular interest since they deal, respectively, with nuclear detection methods and passive NDA techniques. The book also contains many references to original papers and reports.

VALUE/IMPACT STATEMENT

1. PROPOSED ACTION

1.1 Description

Licensees authorized to possess at any one time more than one effective kilogram of special nuclear material (SNM) are required in § 70.51 of 10 CFR Part 70 to establish and maintain a system of control and accountability so that the standard error of any inventory difference 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 are essential for the maintenance of these standards.

Many types of nondestructive assay (NDA) measurements on SNM can involve, or even require, a high-resolution gamma ray spectroscopy system. The proposed action is to provide some general guidelines in the selection of such systems and to point out useful resources for more detailed information on their assembly, optimization, and use in material protection measurements.

1.2 Need for Proposed Action

Regulatory Guide 5.9, which provides guidance in this area, has not been updated since 1974 and does not contain a list of pertinent information currently available in the literature.

1.3 Value/Impact of Proposed Action

1.3.1 NRC Operations

The experience and improvements in detector technology that have occurred since the guide was issued will be made available for the regulatory process. Using these updated techniques should have no adverse impact.

1.3.2 Other Government Agencies

Not applicable.

1.3.3 Industry

Since industry is already applying the more recent detector technology discussed in the guide, updating these techniques should have no adverse impact.

1.3.4 Public

No adverse impact on the public can be foreseen.

1.4 Decision on Proposed Action

The guide should be revised to reflect improvements in techniques, to bring the guide into conformity with current practice, and to provide a list of pertinent information currently available.

2. TECHNICAL APPROACH

Not applicable.

3. PROCEDURAL APPROACH

Of the alternative procedures considered, revision of the existing regulatory guide was selected as the most advantageous and cost effective.

4. STATUTORY CONSIDERATIONS

4.1 NRC Authority

Authority for the proposed action is derived from the Atomic Energy Act of 1954, as amended, and the Energy Reorganization Act of 1974, as amended, and implemented through the Commission's regulations.

4.2 Need for NEPA Assessment

The proposed action is not a major action that may significantly affect the quality of the human environment and does not require an environmental impact statement.

5. RELATIONSHIP TO OTHER EXISTING OR PROPOSED REGULATIONS OR POLICIES

The proposed action is one of a series of revisions of existing regulatory guides on nondestructive assay techniques.

6. SUMMARY AND CONCLUSIONS

Regulatory Guide 5.9 should be revised to bring it up to date.

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