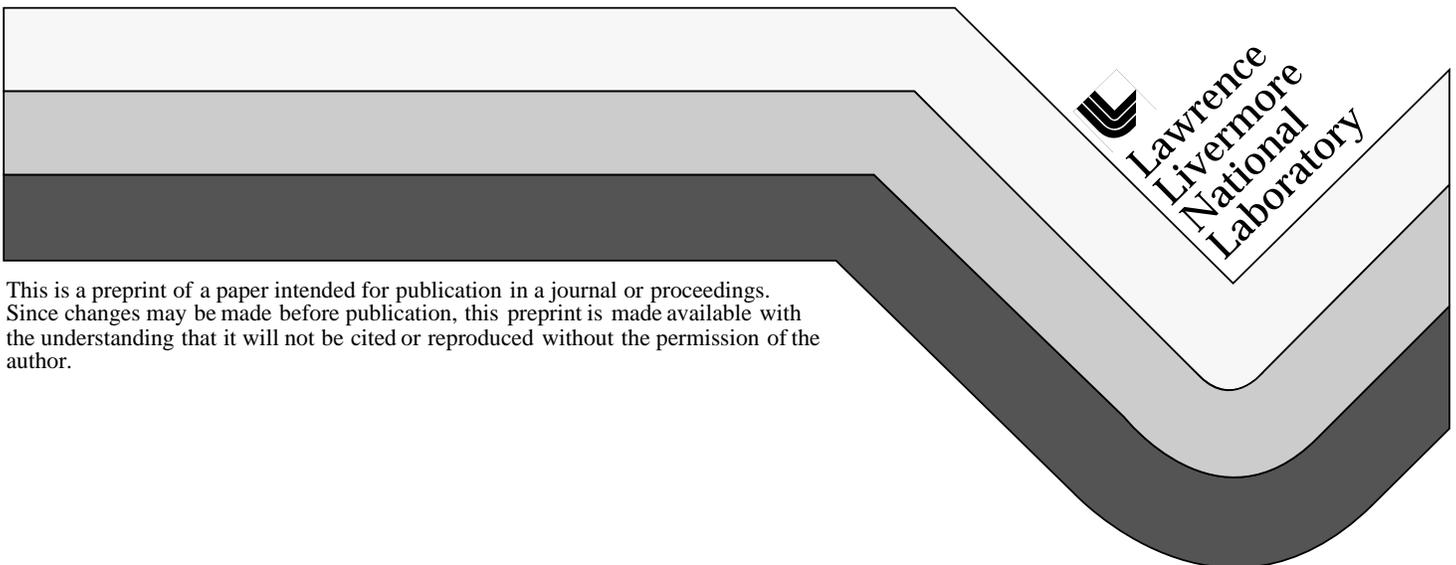


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This paper was prepared for submittal to the
European Safeguards and Research Development Association Workshop
on Quality Requirements on Non-Destructive Analysis Measurements
Ispra, Italy
November 17-19, 1998

October 1998



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Quality Management of Gamma-Ray Isotopic Measurements

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Abstract

Reliable knowledge of the quality or the accuracy and precision of a measurement is important for data evaluation and planning inspection strategies. The auditing of nuclear material accounts requires accepted performance information on the measurements used for the accounting data. It is also important to know what measurement equipment and conditions are necessary to achieve a certain level of quality in the measurement results. Information on the quality of gamma-ray isotopic measurements that can be obtained under various measurement conditions is obtained through inter-comparison exercises, laboratory or field measurement exercises, and tailored laboratory experiments. For these evaluations to be useful, the nuclear materials used in them must have well-characterized isotopic information. In this paper, we discuss measurement equipment and conditions required, reference material standards available and needed, and analysis modification procedures for managing the quality of gamma-ray isotopic measurements.

I. Introduction

To obtain gamma-ray isotopic measurements of good quality requires that the measured data be of good quality and that the data-analysis algorithms used treat the data properly. To obtain good quality data, one must use equipment and perform measurements that meet certain specifications. To ensure that the data-analysis algorithms treat the data properly requires extensive evaluation of the algorithms over a broad range of sample and measurement parameters. For evaluations of gamma-ray isotopic measurements to be useful, the isotopic information about the nuclear material in the samples should be well characterized

The role of nuclear reference materials (standards) in gamma-ray measurements of the isotopic composition of plutonium and uranium (except for the enrichment meter methods) is different than for standards used for other NDA methods. This difference is because the existing data-analysis algorithms do not require calibration with standards, but rely on known values of fundamental nuclear constants and use intrinsic calibration methods. This approach allows the measurement of the isotopic composition of plutonium and uranium in samples of arbitrary size, shape, and composition, and in a measurement geometry that is not required to be reproducible.

However, standards have an important role in the implementation and characterization of this measurement method. Isotopic standards are needed to determine and check measurement performance to provide assurance that the measurement technique is performing as required and to characterize measurement biases or quality.

In the following sections, we discuss the basis for these measurements that make them independent of calibrations, the requirements on the measurement equipment and conditions to ensure good quality data, and the reference material standards that are available for characterizing measurement quality. We describe procedures that should be used in modifying analysis algorithms to maintain their quality. We also describe a possible new approach using Monte Carlo simulations of gamma-ray spectra for characterizing the quality of gamma-ray data-analysis algorithms.

II. Measurement Principles

The basis for the measurement of the amount of any radioactive isotope by gamma-ray spectrometry is given by

$$A_{i,j} = X_i * \lambda_i * B_{i,j} * \epsilon_j, \quad (1)$$

where

$A_{i,j}$ = the measured area of a peak corresponding to a gamma ray of specific energy, E_j , emitted from isotope i ,
 X_i = the amount of the radioactive isotope, i , present in the sample,
 λ_i = decay constant of the isotope ($\lambda_i = \ln 2/T_{1/2}^i$ where $T_{1/2}^i$ is the half-life of isotope i)
 $B_{i,j}$ = the emission probability per decay of the gamma ray, j , being measured from isotope i , and
 ϵ_j = the overall detection or counting efficiency for a gamma ray with energy E_j . It includes detector efficiency, geometry, attenuation by materials between the sample and detector, and sample self-absorption.

The relative amounts of two isotopes may be determined by taking the ratio of Eq. 1 to a similar equation for the other isotope. The relative amounts of isotopes X_i and X_j is given by

$$\frac{X_i}{X_k} = \frac{A_j}{A_m} \cdot \frac{T_{1/2}^i}{T_{1/2}^k} \cdot \frac{B_k}{B_i} \cdot \frac{\epsilon_r}{\epsilon_j} \quad (2)$$

In Eq. 1 and 2, the gamma-ray peak areas, A , are measured and the half-lives, $T_{1/2}$, and the emission probabilities, B , are known nuclear data. In Eq. 2, the efficiency has been expressed in terms of the relative efficiency, ϵ_r . Geometry factors cancel and the relative efficiency includes detector efficiency, attenuation by materials between the sample and detector, and sample self-absorption. The use of an efficiency ratio removes the need for reproducible geometry and makes the measurement of isotopic ratios applicable to samples of arbitrary size, shape, and composition.

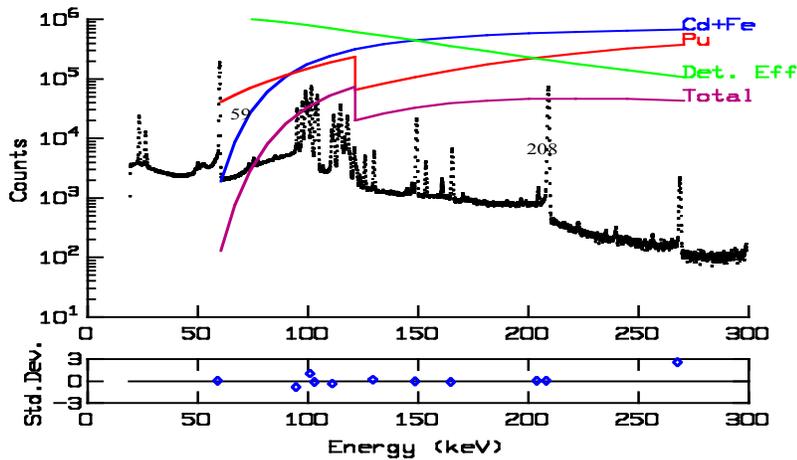
The relative efficiency may be determined as a function of energy by the variation of $A_{i,j}/B_{i,j}$ with energy for a series of gamma rays from isotope i . This variation in energy is the same for all isotopes in a sample (assuming no isotopic inhomogeneities). The data

from several isotopes may be normalized relative to each other to provide more data for determination of the relative efficiency curve.

This relative efficiency curve is unique to each detector/sample measurement combination and therefore must be obtained directly from the measured spectral data. The curve cannot be predetermined and thus is intrinsic to every measurement. Most gamma-ray analysis codes use a functional form for the efficiency curve that does not necessarily reflect the physical processes involved in detecting the gamma- and x-ray emissions. However, in MGA^{1,2} we use a functional form that describes the three principal interactions involved in the detection process. These are 1) the detector efficiency, 2) absorption by cadmium (or other) filters, and 3) self-attenuation by plutonium in the sample. The following equation is used to describe these processes:

$$A_j = \left(\sum_{k=1}^{k=3} (B_{j,k} \cdot X_k) \right) \cdot \exp(-\mu_j \cdot \mathbf{CD}) \cdot \left((1 - \exp(-\mu_j \cdot \mathbf{PU}) / \mu_j \cdot \mathbf{PU}) \right) \cdot \epsilon^0(1 - \mathbf{b} \cdot E - \mathbf{c} \cdot E^2) \quad (3)$$

where A_j are the areas of ten peaks in the low-energy region of each spectrum, due to ²³⁹Pu, ²⁴¹Pu-²³⁷U, and ²⁴¹Am and X_k are the unknown amounts of these isotopes. The absorption coefficients, μ_j , at each peak energy are known for Cd and Pu, but the thickness of the Cd filter and Pu sample thickness are usually treated as unknown variables. The final term in Eq. 3 describes the detector efficiency as a function of the energy, E , where the slope, b , and the curvature, c , are also usually treated as unknown variables. Equation 3 is very nonlinear in form and therefore the variables, shown in bold, must be solved by an iterative least-squares method. The characteristics of Eq. 3 are shown in Fig. 1. The resulting curve for the efficiency is used to determine the relative efficiency values at the peaks of interest.



□

Figure 1 Plot of the three principal processes that characterize the low-energy, intrinsic relative efficiency curve.

Once the relative efficiency has been determined by some method such as that described above, all the terms of Eq. 2 are known, giving a measurement of the ratio of isotope i to isotope k that is independent of calibration and standards.

III. Measurement Requirements

Good quality, measurement results begin with input data that satisfy certain conditions. These conditions are usually met by using equipment and performing measurements that meet certain requirements. In the following section, we discuss some of these requirements.

The gamma- and x-ray peaks in the measured gamma-ray spectral data must have good energy resolution and shapes that satisfy the requirements of the data-analysis algorithms, and be free of interferences from peaks not expected by the algorithms. The first two conditions are addressed by using an appropriate detector and pulse-processing electronics, while the latter conditions are satisfied by controlling the background and types of samples measured.

Good energy resolution is obtained by using a high-resolution, high-purity germanium detector. For MGA it is preferred that the detector can obtain an energy resolution less than 550 eV at 122 keV; however, spectra with energy resolutions in excess of 600 eV have been analyzed, but with reduced precision. If measurement times are to be kept quite short (less than 10 to 20 minutes), then the counting rate should be maximized even if there is some loss in resolution. In this case, the detector preamplifier should be configured with proper feedback resistance to handle high-count-rate measurements. Most detector suppliers are aware of these requirements for MGA. The detector should exhibit very few “slow” collection pulses. These ‘slow’ pulses produce distortions on the high-energy side of peaks and make it more difficult to accurately characterize the peak shape in the data analysis.

Another concern is the amount of damage a germanium detector receives as a result of energetic neutrons emitted by the sample. A high fluence of such neutrons causes damage sites in the detector that have a deleterious effect on the peak shapes. Germanium detector peak shapes should be monitored to identify and eliminate or reduce these effects.

Excellent commercial electronic hardware is available for amplifying and digitizing detector pulses; however, some attention should always be paid to their performance characteristics. The amplifier should include pulse-pileup-rejection and baseline restoration circuitry. We have observed several situations in recent years where high-energy gamma rays not visible in the spectrum (above 300 keV for MGA) were impacting the amplifier baseline and degrading the energy resolution, when baseline restoration capabilities were insufficient. It is important that the amplifier and ADC be very stable, because even small drifts of 0.01 to 0.02 in channel gain or zero level can cause peak-shape distortions and potentially affect the results. Gain and zero level stabilizers may be used to prevent such drifts; however, we have found in recent years

that they are usually not necessary for good amplifiers and ADCs. The amplifier/ADC system must also have good differential and integral linearity.

Nondestructive analysis implies that the measurements are made without altering the sample. However, to properly analyze such a sample, one should consider the isotopic, chemical, and physical characteristics of the sample that may have a bearing on the analysis.

A good analysis algorithm should accommodate any reasonable size sample. Obviously, the lower size limit is determined by the counting rate, the counting time, and the statistical precision required for the measurement. Although plutonium samples of less than 1 mg can be measured, a more practical lower limit is about 100 mg. There is no upper size limit. It should be realized though that the mean penetration of the measured radiations are very small and thus only the surface of a sample is actually measured. The size or more specifically the thickness (g/cm^2) of a sample affects the relative efficiencies with which the various radiations are detected. However, as the sample thickness is increased, the radiations of the added material become increasingly absorbed until a so-called "saturation" thickness is reached. When this condition is reached, the radiations from the back side of the sample are completely absorbed, and the detected number of gamma rays will not increase. The saturation thickness is energy dependent, but is approximately $5 \text{ g}/\text{cm}^2$ for 208-keV gamma rays. Variations of sample thickness less than this amount will significantly alter the relative efficiencies with which the lower-energy gamma rays are detected, hence changing the shape of the 'intrinsic' efficiency curve.

The common chemical forms of plutonium are metals, oxides, and solutions. The chemical form has little effect on the analysis of the spectrum with one notable exception. The alpha- and gamma-induced fluorescence of plutonium is related to the plutonium content or concentration in a sample. Consequently, there are dramatic differences in the observed x-ray intensities for metal, oxide, and solution samples. The qualitative differences can be used to distinguish between these forms.

Recently processed plutonium samples are usually low in ^{237}U and ^{241}Am . The ^{237}U increases in activity for about two months after processing, at which time it reaches equilibrium with the alpha-decay rate of its parent, ^{241}Pu . Any analysis algorithm that always assumes an established decay rate between these two isotopes cannot be used to measure freshly processed samples.

The ^{241}Am content in a sample increases with time. The main effect is the intensity and Compton distribution from its 59-keV peak which soon obscures the lower 38- to 51-keV peaks. Any effect that the increasing intensity of ^{241}Am peaks in the 100-keV regions may have needs further study.

Other radioactive materials may be present in plutonium samples as decay products, as contaminants from previous processes, or as a result of blending. Uranium is a common radioactive material that is blended with plutonium to form mixed-oxide (MOX) fuels.

MGA is capable of determining the relative abundances of the uranium and plutonium in such samples.

Other radioactive materials that may be encountered are ^{237}Np - ^{233}Pa , ^{243}Am - ^{239}Np , and low levels of some fission products such as ^{95}Zr - ^{95}Nb and ^{137}Cs . The fission products are of little concern except if analyzing high-energy regions. ^{237}Np - ^{233}Pa is nearly always present in aged plutonium, because it is a decay product of ^{241}Am . The presence of ^{237}Np does not create significant interference problems, but it should be noted that neptunium is not always removed completely during the chemical processing of nuclear fuels; therefore, its 86-keV peak can sometimes be observed in spectra.

The principal constraint on sample packaging is that the radiations must be able to penetrate the containers so that they can be detected. The lowest-energy peak usually used by MGA is the 59-keV peak. Although MGA can perform an analysis without it, this peak is useful in determining the cadmium absorber thickness, which is used to determine the “intrinsic” efficiency curve. Ideally, the cadmium absorber thickness used should attenuate the 59-keV peak’s intensity to approximately the same height as that of the peaks found in the 100-keV region.

If the 100-keV radiations cannot be observed, because the sample container walls are too thick or the container is lined with lead to reduce exposures, then a new analysis algorithm called MGAHI³ may be used. It uses gamma-ray peaks from 200- to 700-keV to determine the plutonium and ^{241}Am isotopic abundances. Although the accuracy and precision will not be as good as that provided by MGA from the 100-keV region, it does allow isotopic measurements to be performed on samples under these conditions.

The analysis algorithms can assist the user in measuring good quality data. The MGA++ executive³ includes an acquisition pre-check that can determine from a very brief data collection the quality of the data and diagnose basic problems with data acquisition. It also includes an analysis pre-check that examines the spectral data and suggests the most appropriate analysis method.

IV. Standards

Important tools for determining the quality of data-analysis methods are isotopic standards in which the isotopic composition has been determined with accuracy and precision that is greater than can be obtained by gamma-ray spectrometry. The more precise and accurate measurements of the isotopic composition are usually done by mass spectrometry. The characteristics of available plutonium isotopic standards are described below.

In addition to standards that have been prepared for nondestructive isotopic analysis measurements, we describe some standards prepared for mass spectrometry that may also be used for evaluation of gamma-ray isotopic analysis measurements. These mass-spectrometry standards also serve an important role in making other isotopic working standards traceable to the national measurement system, because these working standards are usually characterized by mass spectrometry.

CBNM Nuclear Reference Material 271

This standards set was prepared by the CBNM (now called the Institute of Reference Materials and Measurements (IRMM) in Geel, Belgium). The set consists of four, sealed stainless steel containers each containing a sintered pellet of about 6.6 grams of PuO₂. Each contains a different isotopic composition with their ²³⁹Pu abundance being nominally 93%, 84%, 70%, and 64%. They are certified for ²³⁸Pu-²⁴²Pu and ²⁴¹Am isotopic fractions.

EC Certified Nuclear Reference Material 171/NBL CRM 969

This set of low-enriched uranium standards was issued in 1985. They were prepared and certified jointly by CBNM and the New Brunswick laboratory (NBL). They were prepared primarily for enrichment-meter calibration purposes, but may also be used for nondestructive gamma-ray analysis measurements with codes like MGAU, U235, and FRAM. The set consists of five sealed aluminum alloy cans each containing 200 grams of U₃O₈ with a different enrichment. The ²³⁵U enrichments are nominally 0.31%, 0.71%, 1.94%, 2.95%, and 4.46%.

NBL Certified Nuclear Reference Material 146

This is a new set of uranium gamma-ray spectrometry standards from NBL. It consists of three sealed aluminum can each containing approximately 230 grams of compressed U₃O₈ powder with ²³⁵U enrichments of nominally 20%, 52% and 93%.

NBL Certified Nuclear Reference Material 122

This is a destructive analysis standard for assay, but its isotopic composition is certified. The standard is one gram of PuO₂ packaged in a glass vial. The ²⁴⁰Pu abundance is nominally 11%.

NBL Certified Nuclear Reference Materials 136, 137, 138

These standards were formerly National Bureau of Standards (NBS) 946, 947 and 948. They are the primary US standards available for the calibration of mass spectrometers. Each standard contains about 0.25 grams of plutonium as sulfate. The nominal ²⁴⁰Pu abundances are 12 % for CRM 136, 18% for CRM 137 and 8% for CRM 138. These standards have also been used for evaluation of gamma-ray isotopic analysis methods. These standards are old; and their ²⁴¹Am contents have not been certified and there appears to be a bias in the ²³⁸Pu content of CRM 138.

Plutonium Isotopic Determination Intercomparison Exercise (PIDIE) Standards

A set of seven plutonium standards each containing about 0.4 grams of plutonium with nominal ²⁴⁰Pu isotopic percentages of 6.0, 10.0, 14.1, 19.7, 21.1, 23.8, and 25.5. These standards are not certified, but have been measured by several analytical laboratories and were measured extensively in the intercomparison exercise.

Calorimetry Exchange Standard

The DOE supports a program to track the results of calorimetry and plutonium isotopic measurements by gamma-ray spectrometry throughout the complex. Seven DOE laboratories participate in the program. Each laboratory measures the isotopic composition and total power from a standard PuO₂ sample and reports the results, which are tabulated and distributed. The sample contains 400 grams of plutonium as PuO₂ with a nominal 6% ²⁴⁰Pu content.

PERLA Standards

A set of plutonium oxide and MOX standards with various burnups and range of masses exist at the PERformance Laboratory (PERLA)⁴. These standards are as well characterized as CRM standards. However, only one set exists, but is available for use at PERLA.

V. Use of Standards

Developers of data-analysis algorithms to determine plutonium or uranium isotopic ratios from gamma-ray spectral data need to know whether their analysis methods determine the isotopic ratios without bias. Therefore, they are interested in knowing whether the branching intensities and their peak-area and relative detection efficiency analysis methods are free of bias over the range of applicable parameters that may be encountered in measurements.

The measurement parameters that will vary in a sample are the isotopic abundances, sample size, shape, age, and composition. Parameters external to a sample that will vary are the container wall thickness and composition, sample-detector geometry, detector resolution and peak shape, background, and other gamma-ray interferences.

The standards described above provide a wide variation of isotopic abundances, but provide little variation in sample size, shape, age, and composition for a given isotopic abundance. The PERLA standards do provide variation in size for a given isotopic abundance, but their sizes are greater than the saturation thickness for 200-keV gamma rays so there is little variation in the intrinsic efficiency curve for these sample sizes. To effectively test the intrinsic efficiency analysis of MGA, standards with the same isotopic abundance and with a range of thicknesses less than 5 g/cm² are needed. It would be useful to have a standard in both metal and oxide form to evaluate efficiency analysis algorithms.

A set of standards that may be useful would have the same initial plutonium isotopic abundances, but with different ages from the time of separation from ²⁴¹Am. They would be used to determine whether an analysis algorithm shows any bias in the isotopic analysis as the sample ages. This can also be accomplished by measuring the same sample over a long period of time, but unfortunately, most analysis algorithms have changed with time making it difficult to determine the cause of any observed differences. Monte Carlo simulation of gamma-ray spectra as described in the next section may be useful for this purpose.

Variation in parameters external to a sample, e.g. sample-container wall thickness and composition, may be accomplished by measuring standards placed in various sample containers and in other measurement conditions.

VI. Monte-Carlo Simulations

Isotopic standards are difficult to make and certify, and therefore, are also costly. However, computer simulation can be a cost-effective and convenient tool to create spectra representative of physical standards. These simulations, while not substituting completely for physical standards, can help in determining the quality of isotopic analysis algorithms.

At the Lawrence Livermore National Laboratory (LLNL), we have developed a method⁵ that uses Monte Carlo simulation of photon transport to synthesize radioactive gamma-ray standards. This gamma-ray simulation method has three general components. The first part is the source term, which describes the gamma rays emitted from a radioactive sample with certain isotopic mixture. The computer code GAMGEN⁶, developed at LLNL, is used to calculate the associated gamma-ray intensities from the selected isotopes and their daughters. The code MCNP⁷ from Los Alamos National Laboratory is used to simulate the energy response of the source-detector combination. The third part is a post-processing code called POSTGL, which was written at LLNL, that imposes a realistic detector response function on a simulated spectrum. The detector response function includes Gaussian broadening with low-energy tailing components which simulates effects due to a non-ideal detector. The incorporation of the Lorentzian lineshape of X-rays due to the lifetime of atomic transitions is also included.

This method has been able to synthesize plutonium spectra with sufficient enough detail so that the isotopic composition may be determined from them using the MGA code. This simulation method provides us the capability to test an analysis algorithm on measurement and sample parameters that are difficult to create under laboratory conditions.

VII. Quality Management of Method Enhancements

Current methods for gamma-ray isotopic analysis may not be adequate in locations where fuel cycle or weapons complex reconfiguration or consolidation is occurring or when dealing with legacy materials. When samples or sampling environments produce analyses that are outside the performance envelop of the method, then the method must be enhanced. Alternatively a different method can be validated and used, but this produces measurement control issues.

The general requirements of the enhancement process are: the analysis method will be at least as reliable as before the enhancement, the method will adequately treat the current problem driving the enhancement, and the enhanced method will be available quickly.

All three requirements are addressed, in part, by the software development process. An adequate *software improvement request* (SIR) and *software problem report* (SPR) process needs to be implemented so that what needs to be done can be determined. Defining SIR and SPR forms that collect sufficient information to define the problem or enhancement and making it easy for the user to submit requests facilitate this process. (We maintain a support e-mail address advertised in the on-line help.)

Requirement analysis will refine these reports into proper requirements. Designing for maintainability is a function of architecture. We have selected an architecture (Figure. 2) that maintains the system independence of the physics modules. We explicitly separate calculation and interpretation from input and output. This produces a system where the physics modules can be maintained by scientists; not programmers.

For the actual implementation of enhancements it is important to adhere to software development standards and guidelines to ease maintenance and enforce a uniform look and feel to the code.

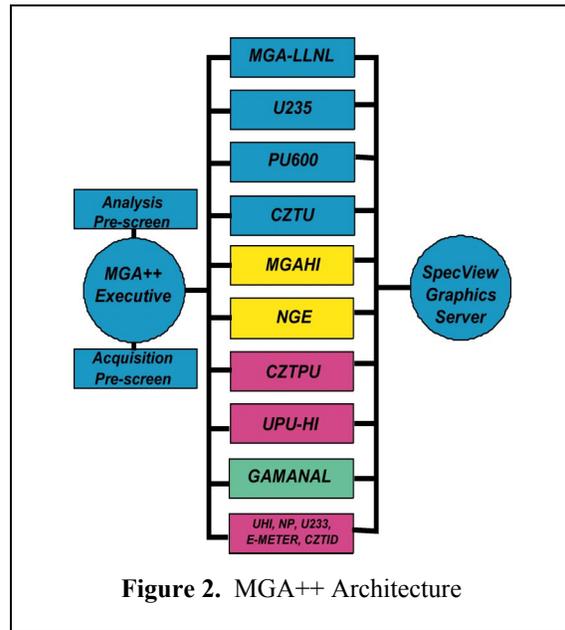


Figure 2. MGA++ Architecture

Quality and ease of maintenance are both enhanced through software configuration management. This is enforced through a Software Configuration Management Plan and appropriate software tools. A complication results when developers (in our case a National Laboratory) commercially licenses software. We must coordinate with the commercial vendor. Establishing a new baseline version at both the developer and licensee whenever software is transferred will ease coordination.

For measurement software the most important aspect to maintaining quality is verification and validation (or testing). This aspect is implemented and enforced through a verification and validation (V&V) plan. The individuals responsible for testing should not be the ones who implemented the changes. The performance testing of the enhanced software should be against a regression test suite. This suite should include not only a variety of spectra taken of standards for which the code has previously been used, but also a set of spectra that represent previously solved measurement problems and other spectra that represent the ‘edges’ of the performance envelope of the program. This regression suite grows with each enhancement.

VIII. Conclusions

There are several important elements in managing the quality of gamma-ray isotopic measurements. First, the input data must meet the requirements of the analysis algorithms. The analysis algorithms should check the data to determine whether its

requirements are met, and if not, reject the input data or flag the output results. The best approach is to check the input data early in a data acquisition and determine whether it meets the requirements of the analysis algorithm. Second, the data-analysis algorithms need to be tested on a broad range of isotopic standards and under as many measurement conditions as possible. In addition, a set of reference spectra or test suite of spectra should be used in modifying or enhancing an analysis algorithm to verify and validate that the changes have not impacted the quality of the analysis. Monte Carlo simulations offer a new tool for synthesizing spectra for testing analysis algorithms under measurement and sample conditions that are difficult to create in the laboratory.

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This work was performed under the auspices of the U.S. Department of Energy by Lawrence Livermore National Laboratory under contract No. W-7405-Eng-48.