### Report of NIST to the CCRI Section II Activities 2007-2008 Prepared by Lisa R. Karam, Chief, Ionizing Radiation Division Presented by Lisa R. Karam, Ionizing Radiation Division March 2009

### **INTRODUCTION**

The Ionizing Radiation Division, one of six divisions within the Physics Laboratory at the National Institute of Standards and Technology (NIST), develops, maintains and disseminates the national standards for ionizing radiation and radioactivity. The Division fulfills its mission through activities in three technical groups: Radiation Interactions and Dosimetry (led by Stephen M. Seltzer), Neutron Interactions and Dosimetry (led by Muhammad Arif), and Radioactivity (led by Michael P. Unterweger). In addition to promoting the accurate and meaningful measurements of dosimetric quantities pertaining to ionizing radiation (x and gamma rays, electrons, and energetic, positively charged particles), the Division maintains the national measurement standards for the Système International (SI) unit for radiation dosimetry (the gray) and activity (the becquerel). It also provides measurement services, standards, and fundamental research in support of NIST's mission as it relates to neutron technology and neutron physics for industrial research and development, national defense, homeland security, electric and alternative power production, and radiation protection, and maintains and disseminates measurement standards for neutron dosimeters, neutron survey instruments, and neutron sources. Finally, the Division is responsible for developing metrological techniques to standardize new radionuclides for research, and for exploring radiation and nuclear applications in health care, worker protection, environmental protection, and national defense.

The Division provides critical measurements and standards for all aspects of ionizing radiation in industry, health care, the environment, homeland security and defense, working closely with the user communities in all of these fields to define and prioritize our research and programs in metrology. Since our self-declaration of conformance in 2006, the Division maintains compliance to the relevant requirements of ISO/IEC 17025 and ISO Guide 34 as part of the NIST quality system in our calibration services and production of our Standard Reference Materials (SRMs<sup>®</sup>). Direct interactions with our international colleagues in metrology (by active participation in implementation of the Mutual Recognition Arrangement through intercomparisons, submission of Calibration and Measurement Capabilities for evaluation, and international meetings) as well as with community members (such as through the Council on Ionizing Radiation Measurements and Standards, CIRMS) serve to provide a strong basis for near-term planning and our current programs. Each year, generally in late October, NIST hosts the CIRMS Annual Meeting, which brings together more than 100 participants who provide perspectives on developments and needs in ionizing radiation research, measurements and standards in health care, homeland security, environmental and personnel protection, and industrial applications. The focus of the 16<sup>th</sup> Annual Meeting on measurements and standards for radiation-based imaging used in industry, academia and government, was of interest across the field of ionizing radiation: from health care applications such as in positron emission (PET) and computed (CT) tomography imaging used in diagnosis and treatment (planning and evaluation) to industry (x-ray imaging as a non-destructive tool to assess product integrity) and homeland security (for non-destructive screening). With the efforts of the Division to support quantitative medical imaging, the topic was particularly timely. In 2008, the 17<sup>th</sup> Annual Meeting ("Radiation Measurements and Standards at the Molecular Level") provided an opportunity for the entire ionizing radiation user community to meet and discuss recent developments and new trends related to measurements at the molecular level, particularly in biodosimetry, and included a special panel on the National Academy of Sciences report regarding the use and security of radioactive cesium salts as related to the evolving homeland security aspects of radiation protection.

The Division maintains a strong focus on addressing the needs in standards and measurements to support health care, particularly in radiation and nuclear therapies. Building on a long history of providing standards for medical x rays and radionuclides used in nuclear medicine, we have been expanding our efforts to better support the extensive use of medical physics in the US today, providing confidence in key results needed for drug and device development and marketing, therapy planning and efficacy, and disease screening. In particular, to support more quantitative medical imaging, we have taken the initial steps in developing phantoms for more accurate calibration of PET and CT systems, including standardizing <sup>68</sup>Ge, for the first time, calibrated to National measurement standards, for PET instrumentation. The ability to calibrate diagnostic imaging tools traceably to national standards will lead to a more quantitative approach and increased accuracy in treatment planning, increased patient safety, and greater confidence in results from clinical trials.

The Division continued to be actively involved in several international efforts over the last two years. In addition to our active participation in the three sections of the Consultative Committee on Ionizing Radiation (CCRI), we have participated in the EURAMAT key comparison (among approximately 26 countries) of air-kerma and absorbed dose to water from <sup>60</sup>Co gamma-ray beams, serving as the host laboratory for participants in the Sistema Interamericano de Metrología (SIM) regional metrological organization (including Canada, Brazil, and Argentina). The first primary standardization of <sup>210</sup>Pb performed by NIST, recently reported by Laureano-Perez, *et al.* [*Applied Radiation Isotopes* 65, *1368-1380* (2007)], led to the development and dissemination of SRM 4337. A direct measurement comparison of this SRM with a UK national <sup>210</sup>Pb standard showed very good agreement, within measurement uncertainties, using five different methods.

Several facilities in the Division, used by a variety of internal and external users from industry, government, and academia as well as international colleagues, have expanded in continued support of research efforts in industrial and medical dosimetry, homeland security, and radiation-hardness and materials-effects studies. Significant progress has been made toward the development of the High-Energy Computed Tomography imaging system (with applications in homeland security), the Clinac accelerator (which will eventually serve as a high-energy calibration laboratory based on primary dosimetry standards for radiation therapy applications), and neutron interferometry (with measurements of 100 nm vertical coherence length for a single crystal neutron interferometer *via* path separation). To minimize operator exposures and expedite the standards-transfer process, a new, automated ionization chamber has been developed to measure up to 100 radioactive samples with minimal sample handling while maintaining the high precision and reproducibility of the manual instrument; the new instrument has successfully been used to measure the half-lives of <sup>82</sup>Sr and <sup>99</sup>Tc<sup>m</sup>.

This document describes some of the significant activities and accomplishments of the Radioactivity Group in 2007 and 2008. Contact information for the primary lead on each of these projects is provided and you are invited to contact the NIST staff for more details. In addition, please visit our website (http://physics.nist.gov/ird) for more information on these and other activities in the Division.

*Strategic Element:* Develop and provide standards for radioactivity based on the SI unit, the becquerel, for homeland security, environmental, medical, and radiation protection applications.

# **Standards in Radioactivity**

The Radioactivity Group develops and improves the metrological techniques used for the standardization of radionuclides, and carries out a wide range of programs in low-level standards for environmental measurements and monitoring, standards for nuclear medicine, standards and testing criteria for radiological instrumentation used for security, and radionuclide metrology. Its mission is to develop, maintain, and disseminate radioactivity standards, develop and apply radioactivity measurement techniques, and engage in research to meet the requirements for new standards. Our participation in international comparison exercises has kept us abreast of efforts of other laboratories and helped us to maintain our own capabilities.

We continue to lead the national effort, in collaboration with the Department of Homeland Security, to develop standards and protocols for radiation instrumentation for early and emergency responders. We have developed an accreditation program with NVLAP for instrument testing. We are also spearheading the development of ANSI standards and testing protocols for spectroscopic portal monitors, neutron detectors, x-ray and high energy gamma-ray interrogation methods, x-ray imaging, data formats for instrumentation data output, and training standards for responders.

The Group continues to lead an internationally-recognized program for standards in nuclear medicine, providing the national standards for radionuclides used in 13 million diagnostic procedures and 200,000 therapeutic nuclear medicine procedures annually in the US. Primary standards of two radionuclides, <sup>223</sup>Ra and <sup>68</sup>Ge have been developed. A new initiative, aimed at establishing standards and measurement support to improve accuracy and consistency in quantitative Positron Emission Tomography/X-ray Computed Tomography (PET/CT) and Single-Photon Emission Computed Tomography (SPECT) imaging, is well underway.

The Group's environmental program leads the community in low-level and natural matrix material measurements and standardization, and continues to be heavily involved in the world-wide measurement of environmental-level radionuclide dispersal and contamination through a large number of international intercomparisons and traceability programs and SRMs. A Radioanalytical Emergency Procedures Manual Database has been developed to assist organizations preparing for emergency response.

Revitalization of our basic metrology capabilities has involved extensive work in many areas. In addition to introducing the TDCR method into routine use at NIST, work is also progressing on the construction of a second-generation TDCR system. The principle method of primary standardization at NIST is live-timed  $4\pi\beta$ - $\gamma$  anticoincidence counting. During the past two years, NIST researchers have adapted this method to perform primary measurements on a variety of radionuclides. A new automated ionization chamber has been developed at NIST to measure up to 100 samples with programmable sample queuing, sample handling and measurement parameters.

# **Radionuclide Metrology and Standards**

## **International Comparison of Co-57**

As part of a program aimed at expanding the existing International Atomic Energy Agency (IAEA)/World Health Organization (WHO) Secondary Standard Dosimetry Standard Laboratory (SSDL) Network to include proficiency testing and calibrations for radioactivity, the IAEA, in collaboration with NIST and other institutions, recently organized a comparison of <sup>57</sup>Co as a surrogate test for the measurement of <sup>99</sup>Tc<sup>m</sup>. The participating laboratories were metrology institutes and other organizations interested in becoming members of the expanded network and represented Brazil, Cuba, the Czech Republic, India, Iran, the Republic of Korea, Romania, and Turkey. Using solutions prepared and calibrated by a commercial company (QSA Global, Braunschweig, Germany), the participants measured the activity concentration of the solutions with either primary methods or radionuclide activity calibrators in a pre-calibrated geometry. The individual laboratory results were transferred to NIST for evaluation in September 2008 and the initial analysis should be finished before the end of 2008. This activity has been registered as a CCRI (II) Supplemental Comparison and a publication describing the comparison and its results will be presented at the meeting of the International Committee on Radionuclide Metrology (ICRM) in Bratislava in 2009.

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# Standardization of <sup>223</sup>Ra

Prostate cancer is one of the most common forms of cancer among men in the United States, with an estimated 220,000 new cases diagnosed each year, and is the second leading cause of cancer death in men. In aggressive cases, the cancer can spread to the skeleton, resulting in extremely painful metastatic bone cancer. A Norwegian company, Algeta, ASA, is developing a novel radiopharmaceutical that uses <sup>223</sup>Ra as the radiolabel in order to not only relieve the pain of the bone cancer, but also to possibly help treat the primary cancer itself. The benefit of <sup>223</sup>Ra over other medically-used radionuclides is that it decays in a chain of 4  $\alpha$ -particle emitters and 2  $\beta$  emitters for each decay of the <sup>223</sup>Ra parent. This results in a very high localized dose to the tumor, with minimal dose to surrounding healthy tissue. Algeta wishes to begin human trials in the United States with this new drug, but a NIST-traceable radioactivity measurement standard needs to be developed in order to ensure that dosages are accurately dispensed before approval is granted by the Food and Drug and Administration.

Using two different techniques to detect the emitted  $\alpha$ - and  $\beta$ - particles, researchers in the Radioactivity Group have been able to calibrate solutions containing therapeutic levels of the drug (10 MBq) for Algeta with a combined standard uncertainty of about 0.8%. Agreement between the two techniques, namely liquid scintillation counting with <sup>3</sup>H efficiency tracing and  $\alpha$ -particle counting using a  $2\pi$  proportional counter, was better than 0.12% and well within the respective experimental uncertainties. As part of this study, a calibration factor for the NIST " $4\pi$ " $\gamma$  secondary standard ionization chamber was developed that will enable future measurements of this radionuclide to be made quickly with approximately the same level of uncertainty. Work is continuing on the development of secondary standards of <sup>223</sup>Ra in clinically relevant geometries that will allow NIST traceable measurements of dosages to be made in hospitals and in Algeta's manufacturing facilities.

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# Development of a primary standard for <sup>68</sup>Ge

The number of Positron Emission Tomography (PET) procedures using <sup>18</sup>F continues to rise at a rate of about



20 % per year. The ability to obtain quantitative data about the status of a disease depends on the ability to calibrate all the instrumentation used to make radioactivity measurements in the clinic, including the PET scanners, to the same standard. Although NIST has standardized <sup>18</sup>F and has performed a number of calibrations for the NIH PET Center and PETNET Solutions (a distributor of  $^{18}$ F regional radiopharmaceuticals), the short half-life (1.83 h) makes it impossible to widely distribute SRMs of this radionuclide. The

Results of absolute activity measurements of the <sup>68</sup>Ge master solution using  $4\pi\beta$ - $\gamma$  livetimed anticoincidence counting (LTAC), the Triple-to-Double Coincidence Ratio (TDCR) method and the CIEMAT/NIST method of <sup>3</sup>H efficiency tracing (CNET). The uncertainty bars represent expanded (k=2) uncertainties.

Radioactivity Group, in collaboration with RadQual, LLC, has developed a new standard for <sup>68</sup>Ge (in equilibrium with its <sup>68</sup>Ga decay daughter), which is being proposed as a long-lived substitute for <sup>18</sup>F for calibration purposes. Germanium-68 decays with a half-life of 270.95 d to <sup>68</sup>Ga, which has a very similar decay scheme to that of <sup>18</sup>F.

A master solution of <sup>68</sup>Ge was gravimetrically diluted and the resulting solution was analyzed for radioactivity content by 3 liquid scintillation-based techniques:  $4\pi\beta$ - $\gamma$  anticoincidence (AC), Triple-to-Double Coincidence Ratio (TDCR) method, and <sup>3</sup>H-standard efficiency tracing with the CIEMAT/NIST method (CNET). The anticoincidence technique was able to determine the activity concentration of the solution with a combined standard uncertainty of less than 0.3 %. The other two methods gave activity concentration values with respective differences from the reference value of +1.2% and -1.5%, which were still within the experimental uncertainties. As part of the project, the calibration was transferred to the NIST  $4\pi\gamma$  secondary standard ionization chamber, which will allow for future calibrations to be performed on a routine basis, and calibration factors for commercial re-entrant ionization chambers ("dose calibrators") were developed to facilitate clinical measurements.

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## Liquid Scintillation with the Triple-to-Double Coincidence Ratio Method

The NIST TDCR spectrometer was used to provide confirmatory measurements of <sup>90</sup>Sr, <sup>99</sup>Tc, and <sup>229</sup>Th in support of the Standard Reference Material program, as well as being one of the primary methods used in the standardization of <sup>68</sup>Ge for the Nuclear Medicine Standards Project. Additional experiments were made on a solution of <sup>241</sup>Pu with good agreement observed between TDCR results from the NIST system and several measurements made on the TDCR systems at the Labortoire National Henri Becquerel (LNHB), but with significant (~7 %) differences between all the TDCR results and those obtained from CIEMAT/NIST efficiency tracing and <sup>241</sup>Am in-growth. An interlaboratory comparison of <sup>241</sup>Pu is planned for early 2009 between the National Physics Laboratory (UK), the Physikalisch - Technische Bundesanstalt (Germany), LNHB, and NIST that will hopefully provide insight into the origin of the discrepancy.

In addition to introducing the TDCR method into routine use at NIST, work is also progressing on the construction of a second-generation TDCR system. This new spectrometer was designed to include the possibility of using the Compton spectrum of electrons produced in the scintillator from a standard external source to independently determine the form of the quench function for that cocktail. Design modifications to

the sample chamber will also hopefully result in a detection efficiency that is significantly higher than the existing instrument. Finally, the electronics of this new system will be based primarily on Field Programmable Gate Array (FPGA) technology in place of the LNHB-designed MAC3 coincidence unit, which is unfortunately no longer available.

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### Comparison of TDCR data analysis methodologies and uncertainty assessments

In order to gain insight into the differences in measurement results arising from the use of different TDCR



analysis programs, a data analysis comparison was organized by NIST and the Liquid Scintillation Counting Working Group of the ICRM. In this activity, a single set of counting data for <sup>99</sup>Tc, acquired on the NIST TDCR system as part of the certification of a 99Tc SRM, was sent to 9 participating laboratories. Each laboratory was asked to analyze the counting data using whatever programs and methodologies they would normally use. The preliminary results, normalized to the activity concentration of the <sup>99</sup>Tc solution as certified by NIST are shown to the left.

Although the analysis of the results is still being carried out, two important trends have emerged. The first is that the shape factor used for the beta spectrum of <sup>99</sup>Tc plays a crucial role in the correct determination of the activity using the TDCR method for this radionuclide. The other is the fact that even for a relative high-energy beta emitter such as <sup>99</sup>Tc ( $E_{\beta}$ ,max = 293.5 keV), failure to account for the effect of phototube asymmetry can lead to errors of up to 0.6 % in the calculated efficiency.

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### Primary Standardization of Radionuclides by Anticoincidence Counting

The standardization of radionuclides by primary methods is the foundation for all the standards and services



Coating a thin-film source with gold for an anticoincidence measurement.

primary includes is the foundation for an the standards and services provided by the NIST radioactivity group. These include: SRM's, special calibrations, and proficiency testing. The principle method of primary standardization at NIST is live-timed  $4\pi\beta$ - $\gamma$  anticoincidence counting. During the past two years, NIST researchers have adapted this method to perform primary measurements on a variety of radionuclides including alpha, beta and positron/EC emitters: namely, <sup>241</sup>Am, <sup>57</sup>Co, <sup>68</sup>Ge/<sup>68</sup>Ga, <sup>229</sup>Th and <sup>99</sup>Tc. In 2008, the anticoincidence system was expanded to detect charged particles using either a liquid scintillation detector or a proportional counter. This upgrade involved developments in source preparation and detector performance. With this new flexibility, NIST will be able to access a wide range of particle energies and source configurations. Work is underway to utilize this capability for the first intercontinental comparison of <sup>99</sup>Tc<sup>m</sup> primary standards.

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# Automated Multi-Sample Ionization Chamber for Radioactivity Standards

NIST radioactivity standards for gamma-ray emitting radionuclides are presently maintained as calibration



ting radionuclides are presently maintained as calibration factors for a single, manually operated, ionization chamber instrument. A new automated ionization chamber has been developed at NIST to measure up to 100 samples with programmable sample queuing, sample handling and measurement parameters while maintaining the high precision and reproducibility of the manual instrument. Routine use of this new instrument will reduce radiation exposure to operators, expedite the standards-transfer process and improve quality assurance. Measurement reproducibility and the stability of calibration factors over a one-year period have been verified. Calibration factors are being transferred to the new instrument, and it has

successfully been used to measure the half-lives of <sup>82</sup>Sr and <sup>99</sup>Tc<sup>m</sup>.

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# Discrepancies in the <sup>241</sup>Pu Standardization of SRM 4340B

All of the plutonium isotopes, which have a very high biotoxicity, are man-made and result from various nuclear activities. The isotope <sup>241</sup>Pu, as a very low-energy pure-beta-emitting radionuclide with a half-life of



about 14 years, is the most difficult of the plutonium isotopes to detect and measure. As a result, radioactivity standards of <sup>241</sup>Pu are of great interest for many environmental monitoring and radioecological studies. A new solution standard of <sup>241</sup>Pu was developed and disseminated as Standard Reference Material 4340B. A previous <sup>241</sup>Pu standard (SRM 4340A) was issued in 1996. The certified massic activity in the new standard is wholly consistent with the certified activity in SRM 4340A. However, an unexpected and inexplicable discrepancy between measurement methods was uncovered. The certified massic activity in the new SRM 4340B was based on liquid scintillation (LS) measurements using the CIEMAT/NIST <sup>3</sup>Hstandard efficiency tracing method (CNET), but was

independently confirmed by agreement with <sup>241</sup>Am ingrowth measurements that have been performed over a period of 31 years on a linked <sup>241</sup>Pu solution. Two independent determinations (one by NIST and another by the Laboratoire National Henri Becquerel in France) on gravimetrically linked solutions, based on LS measurements using a triple-to-double-coincidence ratio (TDCR) counting method, are in excellent agreement to within about 0.2 % but are seriously discrepant with the LS CNET-based certified value by about 8 %. The discrepancy is difficult to understand considering that the CNET and TDCR results used the same nuclear data (beta spectra and decay scheme) and are based on the identical underlying models, although the computational approaches are different. In an effort to further study and resolve this measurement discrepancy, an

international measurement comparison amongst a few select national metrology laboratories has been planned and will be conducted in the near future.

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## Wall Effect for Alpha Particles in Liquid Scintillation Counting

For many years, our laboratory assumed that the counting loss for  $\alpha$  particles due to a wall effect in LS counting was negligible. A report by a researcher at the Laboratoire National Henri Becquerel in France reported that these measurements required a correction of magnitude of about 0.2 %. We did not believe that this report could withstand critical scrutiny. Several experiments using <sup>241</sup>Am and <sup>210</sup>Po were performed to evaluate whether a significant wall effect exists. Trials were made that looked at the LS rates of standardized solutions as a function of large changes in the surface to volume ratio in a series of LS cocktails. In addition, experiments were conducted to look at the non-detection efficiency in  $4\pi\alpha(\text{LS})$ - $\gamma(\text{NaI})$  live-timed anticoincidence counting. Neither effort supported the existence of a 0.2 % wall effect loss, although the results were somewhat equivocal. Further, more definitive experiments are planned and are underway.

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# **Rigorous Evaluation of Primary**<sup>241</sup>**Am Standardizations**

Solution standards of <sup>241</sup>Am are needed by and produced for several of the Radioactivity Group's programs, including the SRM program, the NEI Nuclear Power Plant Program (PPP), environmental programs, and the Integrated Consortium of Laboratory Networks (ICLN) that includes various nuclear forensic laboratories. The certified massic activities for these <sup>241</sup>Am standards typically have relative expanded uncertainties (k = 2) of about 0.2 %. We recently completed a rigorous test of our capability to perform primary <sup>241</sup>Am standardizations.

This was achieved by comparing the results of four independent primary standardizations on three gravimetrically linked solution standards that were prepared for different programs. Three of the standardizations were based on  $4\pi\alpha$  LS spectrometry and the fourth used a more powerful  $4\pi\alpha$ (LS)- $\gamma$ (NaI) live-timed anticoincidence counting (LTAC) method. The <sup>241</sup>Am solution standards comprised those produced for SRM 4322C, for a set of ampoules used for proficiency testing by the NEI PPP, and a set of ampoules prepared for the ICLN. In addition, direct comparative measurements were made between the new SRM 4322C and the previous SRM 4322B issue. These were relative measurements based on  $\gamma$ -ray spectrometry with HPGe detectors and even more precise determinations using a  $4\pi\gamma$ (NaI) sandwich detector that consists of two large 20-cm scintillation crystals.

The certified values for the massic activity of the two SRMs agreed to within 0.13 % for the  $4\pi\gamma$ (NaI) measurements. The primary standardization of SRM 4322C by  $4\pi\alpha$  LS and by the  $4\pi\alpha$ (LS)- $\gamma$ (NaI) LTAC methods agreed to about 0.05 %; whereas the three  $4\pi\alpha$  LS standardizations on the three solution standards agreed to about 0.2 % compared to their gravimetric linkages.

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## **New Mass Comparator Is Operational**

The ability to perform accurate mass measurements in a range from a few milligrams up to several kilograms



New mass comparator and its ancillary class E1 weight set

is a vital component of many of the Radioactivity Group's programs. It is needed for the preparation of counting sources, for the production of large volumes of standardized solutions, and to ensure that the various sources and solutions are gravimetrically linked. Twentytwo different microbalances, analytical balances, and large capacity balances are maintained by the Group for this purpose. One of the principal workhorses, particularly for the production of SRMs, that has been used for at least the past 40 years, has been a Voland Jupiter 3000 balance. It has a capacity of 3 kg and readability of 10 µg (actually only 100 µg effectively) and uses internal dial weights and an optical scale. In recognition that this balance may be approaching the end of its useful lifetime and that the SRM program

would have been shut down if it wasn't available for use, a suitable replacement was sought, obtained, and installed. The replacement is a Mettler-Toledo AX12004 Mass Comparator, which is a high-precision mass comparator up to 12 kg and a 100  $\mu$ g readability, with a two-position turntable and a large draft shield to accommodate large volumes. The comparator uses internal balance weights and has an "electronic" EMF compensation range of about 100 g. Mass measurements are made by substitution weighing against a calibrated E1 weight set. Extensive tests evaluating the performance of the balance for our purposes have been completed, and protocols for its use to measure large volume solutions have been developed.

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# International agreement on <sup>210</sup>Pb radioactivity standards

Radionuclidic standards of <sup>210</sup>Pb are amongst the most important of those needed by the worldwide



environmental radioactivity measurement community. Its standardization, however, is exceedingly difficult because of its particular decay modes, and has rarely been attempted by the national metrology institutes of other countries. The first primary standardization of <sup>210</sup>Pb performed by NIST was recently reported on by Laureano-Perez, *et al.* [Applied Radiation Isotopes 65, 1368-1380 (2007)], and led to the development and dissemination of Standard Reference Material SRM 4337. The standardization was based on  $4\pi\alpha\beta$  liquid scintillation (LS) spectrometry with <sup>3</sup>H-standard efficiency tracing. Confirmatory measurements were also performed by three other methods. The expanded (k = 2) combined standard uncertainty was conservatively estimated to be 2.4 %, which is atypically large compared to most primary

radioactivity standardizations that are performed by NIST. This larger-than-usual uncertainty (and our desire to insure the standard's consistency with other national metrology laboratories) led us to perform a direct measurement comparison with a UK national standard of <sup>210</sup>Pb that was obtained from the National Physical Laboratory (NPL), and which was also known to have direct links to a primary standardization performed by

the Physikalisch-Technische Bundesanstalt (PTB) in Germany. The NIST and NPL solution standards were directly compared with five measurement methods, including by  $4\pi\alpha\beta$  LS counting, that was the most precise method, its value agreed to within 0.3 %, which was well within the 1.5 % propagated standard uncertainty assigned to both standards. The ratio of the measured activities for the two standards compared to their certified values were in very good agreement for all five methods within their measurement uncertainties. This finding suggests that the originally assigned uncertainty on the <sup>210</sup>Pb standard may have been overestimated.

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# Standardization of <sup>229</sup>Th: Comparison of Methods

Measurements of thorium isotopes in the naturally-occurring radioactive decay chains (i.e., <sup>235</sup>U, <sup>238</sup>U, and <sup>232</sup>Th) are exceedingly important for various applications related to the nuclear fuel cycle, nuclear materials waste storage and cleanup, radiobioassays, and geochemical studies. Thorium-229 is the most important long-



lived thorium isotope that is used as a monitoring tracer for these measurements. Hence, a standard for <sup>229</sup>Th is the most requested and of greatest importance to the national network of nuclear forensic laboratories. It is the parent of a very difficult-to-measure eight-member decay chain: Th-229  $\rightarrow$  Ra-225  $\rightarrow$  Ac-225  $\rightarrow$  $Fr-221 \rightarrow At-217 \rightarrow Bi-213 \rightarrow Po-213$  $\rightarrow$  Pb-209  $\rightarrow$  Bi-209 (stable). The chain is especially difficult to measure due to Po-213, whose half-life  $(4.2 \ \mu s)$  interferes with the resolving times of most counting This led us to consider instruments. doing the standardization by a variety of different methods, particularly by more than one primary method. As a result a <sup>229</sup>Th Standard Reference Material (SRM

4328C) was developed and disseminated by NIST. The certification for the standard was based on  $4\pi\alpha\beta(LS)$  -  $\gamma(NaI)$  live-timed anti-coincidence counting (LTAC) with confirmatory measurements by five other methods: (i)  $4\pi\alpha\beta$  liquid scintillation (LS CNET) spectrometry (with <sup>3</sup>H standard efficiency tracing for  $\beta$  efficiencies); (ii) an LS-based  $4\pi\alpha\beta$  triple-to-double coincidence ratio (TDCR) method; (iii)  $2\pi\alpha$  proportional counting (PC); (iv)  $2\pi\alpha$  spectrometry using Si surface barrier detector (a-SPECT), following chemical separation, with a <sup>230</sup>Th standard tracer; and (v) HPGe  $\gamma$ -ray spectrometry (g-SPECT). The expanded (k = 2) uncertainties for the five confirmatory methods were: (i) 1.2 %; (ii) 1.0 %; (iii) 2 %; (iv) 2 %; and (v) 3 %, respectively. All of the confirmatory measurements agreed with the certified value within their respective measurement uncertainties, except that for the TDCR result (-1.7 % difference). This discrepancy in methods has also been observed in <sup>241</sup>Pu which suggests that there is a need for further study on this matter. The results for methods (i) and (iv) agreed with the certified anti-coincidence value to better than 0.1 %.

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# **Investigation on** <sup>99</sup>Tc

Technetium-99 is a pure beta emitter with a half-life of  $2.11 \times 10^5$  years. It is the most significant long-lived fission product and one of the more significant components of nuclear waste. A <sup>99</sup>Tc standard is important for monitoring inside nuclear facilities and in the environment. In addition, since there is not a stable technetium isotope, <sup>99</sup>Tc is also used in chemical studies to characterize the radiopharmaceuticals developed for the widely used <sup>99</sup>Tc<sup>m</sup>. A new <sup>99</sup>Tc Standard Reference Material was developed and disseminated by NIST as SRM 4288B. The certified massic activity for <sup>99</sup>Tc was obtained by  $4\pi\beta$  liquid scintillation (LS) spectrometry. The LS detection efficiency was calculated using the TRACER code for the CIEMAT/NIST method with composition matched LS cocktails of a 3H standard as the efficiency detection monitor. Since previous calibration showed dependence on the <sup>99</sup>Tc beta spectrum, its shape factor was also studied. Analysis was performed assuming allowed, 2nd forbidden and experimental "partially forbidden" transitions. Confirmatory measurements were performed by: (i)  $4\pi\beta(LS) - \gamma(NaI)$  live-timed anti-coincidence (LTAC) counting; and (ii) an LS-based  $4\pi\beta$  triple-to-double coincidence ratio (TDCR) method. The certified value was in agreement with the confirmatory determinations to within -0.22 % and +0.06 %, respectively, for the LTAC and TDCR methods. The relative k = 2 expanded uncertainties on the two confirmatory determinations are approximately 0.7 % and 0.3 %, respectively. A previous solution of 99Tc (SRM 4288A) was calibrated in 1996 by  $4\pi\beta(LS)$ -  $\gamma$ (NaI) live-timed anti-coincidence (LTAC) counting and by  $4\pi\beta$  liquid scintillation (LS) spectrometry. The two standards were compared by LS measurements and were found to be in agreement to better than 0.6%.

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### A New Low-Energy Photon Spectrometry System

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A new low-energy photonic-emission spectrometry system (consisting of a high-resolution, windowless, liquid nitrogen cooled, Si(Li) detector, an especially-designed and fabricated vacuum chamber equipped with a gate valve for coupling it to the windowless detector, and a suitable calibration range in the chamber to handle a variety of source configurations and geometries) has been assembled. Extensive performance and evaluation tests for energy resolution, detection efficiency, and source positioning effects are currently underway to characterize the system for use with point sources. The development work was delayed for nearly a year because of the loss of laboratory space and the need to dismantle and reassemble the entire system in a new laboratory. The Radioactivity Group must rely on photonic-emission rate measurements for its SRM program and calibration services; for impurity analyses of virtually every radioactive source handled by the group; and for developing new primary standardizations and calibrations, particularly for applications in nuclear medicine. At present, the Group has very little capability (none in some energy ranges) for measuring low energy photons. For example, the group was recently hampered in its efforts to provide a <sup>210</sup>Pb standardization because it could not measure its 46-keV gamma ray to better than 8 %. Similarly, we were unable to perform simple x-ray measurements of <sup>55</sup>Fe, which would have been useful to confirm our submitted measurement result for the BIPM international comparison. Additionally, this present measurement limitation precludes the Group from providing measurements, calibrations, and standards from a whole range of nuclides that decay by electron capture, which are amongst the most difficult within the realm of radionuclidic standardization. One can easily identify tens of radionuclides that we must measure for calibrations and standards whose activity determinations would benefit from having a low-energy emission rate measurement. These include nuclides that have become increasingly important for nuclear medicine (e.g., <sup>103</sup>Pd, <sup>82</sup>Sr, <sup>68</sup>Ge, and <sup>223</sup>Ra), for environmental measurements (e.g., <sup>210</sup>Pb, <sup>226</sup>Ra, <sup>239</sup>Pu, as well as many other actinides), for the nuclear fuel cycle and nuclear materials waste storage and cleanup (<sup>99</sup>Tc, <sup>125</sup>I, and <sup>55</sup>Fe), and for homeland security (e.g., <sup>137</sup>Cs and special nuclear materials). This expanded measurement capability would be of great value for the performance of impurity analyses on both internal-to-NIST sources and for submitted sources; in the development of new primary standardizations for nuclides with applications in many areas (medicine, environment, nuclear fuel cycle, homeland security), particularly for those we are presently incapable of standardizing (and those requiring a confirmatory measurement method); for confirmations of primary

measurements; for calibration of our solution and natural matrix SRMs; and in extending our photon calibration services to much lower energies.

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### **Resonance Ionization Mass Spectrometry**

The resonance ionization mass spectroscopy (RIMS) facility at NIST is used for trace analysis applications. It



The resonance ionization mass spectrometry system (RIMS).

### scopy (RIMS) facility at NIST is used for trace analysis applications. It is optimized for precisely measuring isotopic ratios, while eliminating isobaric interferences that may hinder other methods. A quadrupole mass spectrometer (QMS) has been incorporated into the RIMS system and is being used to measure cesium isotopes, using CW diode and Ar-ion lasers for the resonant-excitation and ionization steps, respectively. The sensitivity of the new instrument has been demonstrated and work is ongoing to improve the overall efficiency. The next phase of the project will focus on adapting the RIMS system to the changing needs of the nuclear forensics field.

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# Absolute Characterization of Isotopes with Coincident Emission of Several Gammas by NaI(Tl) Detectors





A modification of the gamma-gamma coincident activity measurement method was used for the characterization of <sup>60</sup>Co. Two-dimensional analysis of coincident events allows one to calculate the fraction of Compton scattered gammas for each of two gammas emitted by 60Co and for each of two NaI(Tl) detectors. A modification of standard formulas that were used before for absolute calibration of <sup>125</sup>I (Eldridge formulas) was done. These modified Eldridge formulas were applied for absolute calibration of <sup>60</sup>Co samples with two NaI(Tl) detectors. A similar approach was applied to more complicated cases of three coincident gamma emission in <sup>108m</sup>Ag decay and for positron-gamma coincident emission in <sup>22</sup>Na and <sup>26</sup>Al

decays. The large number of experimental quantities, measured in coincident and non-coincident modes, allows determining both detector efficiencies and source activity.

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# **Homeland Security**

## Testing

The National Institute of Standard and Technology (NIST) is working together with the Department of Homeland Security (DHS) Domestic Nuclear Detection Office (DNDO) in testing radiation detection equipment for homeland security applications. These tests included instruments to be deployed in different venues and included Personal radiation detectors (PRDs) used by law enforcement and boat-mounted radiation detection systems used in maritime applications. These tests assist users with the procurement of instruments to meet their needs and provide manufacturers with information to improve existing technology. NIST has worked in the testing of the Advance Spectroscopic Portal (ASP) monitors and the development of a new Graduated Rad/Nuc Detector Evaluation and Reporting (GRaDER) Program. The GRaDER program will provide users with information about the performance of radiation instruments. The outcome of this evaluation will provide DHS/DNDO with a list of equipment that will meet performance requirements listed in relevant ANSI/IEEE standards.

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# Rapid screening of <sup>90</sup>Sr and <sup>63</sup>Ni in urine

Treatment of <sup>90</sup>Sr and <sup>63</sup>Ni-spiked, acidified urine samples with activated carbon has been shown to remove the quenching problem without adsorption loss of the radionuclide. The results to date offer a promising method for the development of a truly rapid urine screening procedure for <sup>90</sup>Sr and <sup>63</sup>Ni that, despite its non-selectivity, has excellent sensitivity with high throughput and minimal operator handling.

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# Testing of large NaI (Tl) detectors for DNDO SIMP program

The Sodium Iodide Manufacturing Program (SIMP) of the Domestic Nuclear Detection Office (DNDO) program for large NaI (Tl) detectors required a more accurate measurement of NaI (Tl) scintillation detector energy resolution and calibration. The five isotope method for calibration and energy resolution measurements was developed and implemented. This method, which accounts for the non-linearity of the calibration curve, allows for the control and reduction of uncertainty values in energy resolution measurements. The method also allows for the conversion of pulse height resolution measurements obtained by the standard method into energy resolution values more efficiently.

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# **Nuclear Medicine**

## Development of secondary standards for <sup>68</sup>Ge

Because of its ability to provide quantitative data about the in-vivo distribution of an injected radioactive tracer, Positron Emission Tomography (PET) is being increasingly used as a tool in evaluating drug effectiveness during both clinical trials and patient treatment. In order to be able to establish that an observed change in tracer distribution is due to changes in disease progression and not to components of uncertainty encountered in the measurement of the distribution, it is vital that the entire measurement system, from the

dose calibrator to the PET scanner itself, be calibrated to a common standard and monitored regularly. This is especially important in multi-center trials, in which the comparability of data between clinical sites can be



made only if all data are acquired with reference to a single standard. The National Institute of Standards and Technology (NIST) is developing a suite radioactivity standards that of will eventually allow for the first time measurements made with different scanners in different clinical sites over the course of a multi-year trial to be made in a way that is traceable to a single National standard. Up to this point, there has been no way to ensure that the activity calibration for the phantom was directly linked to the calibration of the dose calibrator. Moreover, there was no way to ensure that the calibrations between any two clinical sites were related, nor was there assurance that the calibration had not changed.

The strategy adopted to establish the necessary standards and measurement methodologies was as follows:

- 1. Develop a National *primary radioactivity standard* for <sup>68</sup>Ge as a long-lived surrogate for <sup>18</sup>F.
- 2. Develop a methodology for calibrating the <sup>68</sup>Ge content in an epoxy-based "mock" syringe *dose calibrator standard* (developed and marketed by RadQual, LLC) against the NIST <sup>68</sup>Ge primary standard.
- 3. Develop a methodology for calibrating the <sup>68</sup>Ge content in an epoxy-based *cylindrical phantom insert* (developed and constructed by NIST) against the NIST <sup>68</sup>Ge primary standard.
- 4. Using direct measurements, provide a *link between the* <sup>68</sup>*Ge calibration sources and equivalent sources containing* <sup>18</sup>*F*, all in a way that is traceable to the NIST National standards of those radionuclides.

For the secondary standards, a series of epoxy-based <sup>68</sup>Ge sources was gravimetrically prepared by combining a commercially-available epoxy with an appropriate amount of <sup>68</sup>GeCl<sub>4</sub> solution and mixing according to the



Epoxy-based <sup>68</sup>Ge "mock" syringe artifact standard developed by RadQual, LLC for the calibration of dose calibrators for <sup>18</sup>F. The epoxy is colored in this photograph only for visual detail.

manufacturer's instructions. Nominally 3 g of the <sup>68</sup>Ge-epoxy were gravimetrically dispensed into 8 "mock" syringe blanks. Because the batches of <sup>68</sup>Ge-epoxy couldnot be made homogeneous enough (to within 1 % between prepared sources), it was necessary to develop a way to calibrate each source individually using a Vinten 671 secondary standard ionization chamber. This chamber was calibrated by preparing a set of "mock" syringes using pre-calibrated <sup>68</sup>Ge solution and developing a calibration factor, which was obtained with a combined standard uncertainty of about 0.5 %. Possible differences in chamber response due to differences in composition and density between the <sup>68</sup>Ge-epoxy and the standardized solution were investigated by Monte Carlo simulations. The results from both Monte Carlo codes indicated a difference in energy deposited in the ionization chamber gas of 0.1 % between the <sup>68</sup>GeCl<sub>4</sub>

solution and the epoxy, with a relative standard uncertainty on each simulation of about 0.9 %. It was thus concluded that no measurable difference in activity determination for the epoxy-filled "mock" syringes would be expected by use of the calibration factor derived for the solution-filled "mock" syringes. Using the experimentally-determined calibration factor in combination with measurements made on the epoxy-filled "mock" syringes, the activity in each source was determined. Those activities were in turn used to derive empirical dial settings for Capintec and AtomLab dose calibrators.

Using an <sup>18</sup>F-fluorodeoxyglucose solution, a total of 4 solution-filled "syringes" and one 5 mL flame-sealed ampoule were gravimetrically prepared. The activity concentration of the solution was determined by measuring the ampoule in the Vinten chamber and applying a calibration factor that was derived in an earlier experiment. Using the measured activity concentration and the dispensed mass for each "syringe" source, the total activity of <sup>18</sup>F in each source was calculated and used to compare with the activity reading at the calibration settings derived for the <sup>68</sup>Ge-epoxy sources. From these data, it was possible to derive a relative response factor to relate the observed <sup>68</sup>Ge activity to an equivalent <sup>18</sup>F activity in these instruments. This response factor indicates that a 3 mL sample of <sup>18</sup>F in this "syringe" geometry will give a reading in these dose calibrators that is about 5 % higher than an equivalent volume of <sup>68</sup>Ge having the same activity. As a confirmation of the response factor, an additional set of Monte Carlo calculations was carried out for the <sup>18</sup>F in the Vinten chamber was calculated to be 1.053, including the differences in the positron branching ratios, with a standard uncertainty of about 0.9 %, which is in excellent agreement with the experimental value obtained above.

Work is continuing to expand the use of the calibration methodology developed for the "mock syringe" sources to include phantom inserts that can be distributed for use in calibrating PET scanners. This work is expected to be completed by the end of 2008.

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# Secondary Standards for <sup>223</sup>Ra

As a bone-seeking alpha emitter, <sup>223</sup>Ra is being evaluated as a radiopharmaceutical for the treatment of skeletal metastases. In clinical applications, activity measurements of the injected dosage are most often made with commercially available reentrant ionization chambers, often referred to as "dose calibrators". To achieve acceptable levels of accuracy, appropriate calibration factors, or "dial settings", must be employed. In the course of the primary standardization of <sup>223</sup>Ra, the Radioactivity Group at NIST found that calibration settings used in preliminary trials give average activity readings 5.7 % to 8.7 % higher than the NIST calibrated activity. A secondary standardization is currently being undertaken to empirically derive calibration settings for the most commonly used dose calibrators. Initial measurements in the 5 mL NIST ampoule geometry— the standard geometry for manufacturer-recommended calibration settings for the most commonly used dose calibrator with results for clinically relevant geometries. Because the characteristics (wall thickness, composition, etc.) of the sample container affect the extent to which source radiation is attenuated, accurate activity measurements require dial settings specific to a given geometry. Given the relatively low energy x rays and bremsstrahlung characteristic of <sup>223</sup>Ra decay, attenuation effects might be expected to be particularly acute.

Preliminary measurements find that the optimum calibration factors for a 20 mL dose vial are consistent with those for the 5 mL NIST ampoule to within experimental uncertainty. Furthermore, no volume correction was required over a range between 0.5 mL to 6.0 mL. Continuing efforts will extend the secondary standardization to several syringe geometries.

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# **Radiochemistry and Environmental Metrology**

**Russian Roulette with a Hot-Headed Phantom: Monte Carlo efficiency calculations of a voxelized BOMAB** 



A 3-dimensional depiction of the MCNP geometry. The radioactive voxelized BOMAB phantom is seated in front of a HPGe detector



voxelized BOMAB phantom. Each bottle is filled with a nitric acid solution spiked with Ga-67.

This work is a continuation of a long term project presented last year in which we seek to estimate the efficiency of gamma-ray systems using Monte Carlo computation. This computed efficiency is validated by making real measurements of our standard geometry, a Bottle Manikin Absorption (BOMAB) phantom. This phantom consists of 10 plastic bottles which assemble to represent the ICRP 23 reference man. Each bottle was filled with a nitric acid solution spiked with Ga-67. The radioactive BOMAB phantom was measured in a seated position at 8 counting distances from a HPGe detector and the experimental efficiency for our gamma-ray spectrometry system is determined. The same set of experiments is then modeled using the Monte Carlo N-Particle Transport Code (MCNP). The main task for the MCNP calculations is to create an accurate description of the measuring system which includes the detector, the radioactive phantom, and the environment. To this end, a realistic computational model of our BOMAB phantom was developed with the aid of tomographic medical imaging modalities.

Each of the plastic bottles which comprise the BOMAB phantom was individually CAT scanned at the National Naval Medical Center. Using the computer software Scan2MCNP (White Rock Science), the resulting tomograms underwent a process called

segmentation in which materials of interest are assigned to appropriate regions of the medical images according to their density. The segmented data was then written into an array of voxels (hexahedra-shaped volume elements) suitable for implementation in MCNP radiation transport calculations. The resulting computational phantom contains roughly 9 million voxels with dimensions of approximately 2.5mm x 2.5mm x 1.0mm.

Monoenergetic input files for the 5 photon energies of Ga-67 with greatest intensity were run at each of the counting distances. On a single computer, the average computation time per simulation required to achieve statistics of 2% is approximately 10 days. The time the user has to wait for results can be cut significantly by splitting the job amongst multiple computers. Agreement between the computationally determined and experimentally measured efficiencies has been achieved in the majority of cases. With further optimization of the input file, it is expected that results will improve.

The final scope of the project is to create a NIST standard human body phantom, to validate its theoretical efficiency based on a comparison of the Monte Carlo computation with the experimentally measured efficiency, and to calibrate existing phantoms using their respective CAT scan data.

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# **Optimization of the sequential extraction protocol**

Experiments are being carried out on the NIST lake sediment SRM4354 to develop a NIST method for simple but robust radionuclide sequential extraction. The goal of this work is to establish the NIST method for characterization of a new line of environmental radionuclide SRMs which will serve as benchmark reference materials. The new SRMs will be used by environmental assessment and risk management investigators working on environmental cleanup and accident/incident remediation efforts. Optimum extraction conditions are chosen based on radiochemical analysis for Pu and U performed by NIST and on stable elemental analysis performed by Environmental Protection Agency (EPA). Work has been carried out for the five fractions: I (exchangeable), II (bound to carbonates), III (metal oxides), IV (organics), and V (resistates) that shows some substantial differences from the sequential extraction parameters determined on previous trials using the Rocky Flats Soil – 1 (SRM 4353), i.e., optimized extraction time, reagent concentration, and extraction times. The results on the NIST lake sediment provided an important test of the robustness of the optimized extraction

conditions. The optimized sequential extraction protocol has been proposed and is now ready to be used for future radionuclide SRM characterizations.

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## Virtual Gamma-ray Imaging

This project seeks to estimate the efficiency of gamma-ray systems using high resolution gamma-ray imaging and Monte Carlo computation to quantify the radionuclide content in people either by occupational exposure or medical diagnostics. This computed efficiency is validated by making real measurements of our standard geometry, a Bottle Manikin Absorption (BOMAB) phantom. This phantom consists of 10 plastic bottles which assemble to represent the ICRP 23 reference man. Each bottle was filled with a nitric acid solution spiked with Ga-67. The radioactive BOMAB phantom was measured at 8 counting distances from an HPGe detector and the experimental efficiency for our gamma-ray spectrometry system is determined. A chair was specially designed to measure the BOMAB in a seated position. The same set of experiments is then modeled using the Monte Carlo N-Particle Transport Code (MCNP).

The main task for the MCNP calculations is to create an accurate description of the measuring system which includes the detector, the radioactive phantom, and the environment. To this end, a realistic computational model of our BOMAB phantom was developed with the aid of tomographic medical imaging modalities. Each of the plastic bottles which comprise the BOMAB phantom was individually CAT scanned at the National Naval Medical Center. Using the computer software Scan2MCNP (White Rock Science), the resulting tomograms underwent a process called segmentation in which materials of interest are assigned to appropriate regions of the medical images according to their density. The segmented data was then written into an array of voxels (hexahedra-shaped volume elements) suitable for implementation in MCNP radiation transport calculations. The resulting computational phantom contains roughly 9 million voxels with dimensions of approximately 2.5mm x 2.5mm x 1.0mm.



2"x 4" x 16" detector with 3.5" PMT

Monoenergetic input files for the 5 photon energies of Ga-67 with greatest intensity were run at each of the counting distances. On a single computer, the average computation time per simulation required to achieve statistics of 2% is approximately 10 days. The time the user has to wait for results can be cut significantly by splitting the job amongst

multiple computers. Agreement (1 to 4%;  $u_c$  of 1 to 3%, k=1) between the computationally determined and experimentally measured efficiencies has been achieved. With further optimization of the input file, it is expected that results will improve.

Currently, we are calibrating the HPGe detector at different energies by using selected BOMAB phantoms borrowed from the DOE Phantom Library. To date, gamma measurements have been performed on BOMAB phantoms PL-106 ( $^{152}$ Eu), PL-108 ( $^{60}$ Co +  $^{137}$ Cs), and PL-109 (blank phantom) at different distances from the detector. These phantoms have slightly different geometries and masses. Monte Carlo computations for these phantoms based on their tomographic data will follow in the future.

The next part of the project is to create a NIST standard human body phantom, to validate its theoretical efficiency based on a comparison of the Monte Carlo computation with the experimentally measured efficiency, and to calibrate existing phantoms using their respective CAT scan data.

The project will be extended to also evaluate the effect of human variation on the detection characterization of the Virtual Gamma-Ray Range. NIST IRB approval has been granted to carry out this part of the project with NIST volunteers that have received nuclear medicine diagnostic radio nuclides. This part of the project will determine the whole-body detector response of the Radioactivity Group's Virtual Gamma-ray Range system to provide support in the event of an emergency radiological incident. The project will evaluate the system's response to a variety of body geometries, and relate the system's response to handheld *in vivo* gamma-ray

detectors readings that would be used during radiological emergency response. Physical parameters of the volunteers such as height, weight will be needed to assess the correlation with counting response that is expected to have uncertainties as large as 50 %. Homeland Security and nuclear weapon occupational exposure need to assess the exposure of the population of concern to internal contamination received to determine treatment and long-term follow-up.

All of these efforts will be followed on by extending the computational tools to more sophisticated and realistic phantoms, and eventually the ultimate details of the human body.

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### Comparison on Radionuclide Content of SRM 4355A (Peruvian Soil-I)

Nuclear weapons testing discharge from manufacturing and reprocessing facilities, and waste dumping by a number of technologically advanced countries have led to significant world-wide contamination with radioactivity. Nuclear waste storage facilities will inevitably deteriorate and may release further large quantities of radionuclides into the environment. For more than 20 years, countries and their agencies that monitor discharge sites and storage facilities have relied on the NIST SRM 4355 Peruvian Soil reference material. Its low fallout contamination makes it an ideal soil blank for measurements associated with terrestrial pathways-to-man studies. Presently, SRM 4355 is out of stock, and a new batch of the Peruvian soil is currently under development as the future NIST SRM 4355A. Both environmental radioanalytical laboratories and nuclear forensics communities will benefit from this SRM. The former must assess their laboratory contamination and measurement detection limits by measurement of blank sample material. The Peruvian Soil is so low in anthropogenic radionuclides that it is a suitable virtual blank. On the other hand, the nuclear forensics laboratories have high sensitivity instruments that are capable of quantitative isotopic measurements at the level of the Peruvian Soil. Consequently, the Peruvian Soil will provide the nuclear forensics community with the calibration, quality control, and testing material needed for methods development, attribution and legal defensibility. Analyses on three "raw" (before pulverization) Peruvian Soil - II samples were completed for <sup>239+240</sup>Pu content by dissolution of 10 g ash samples, two anion-exchangecolumn (8M HNO<sub>3</sub>) purifications, electrodeposition, and alpha spectrometry. The results show an upper limit of plutonium content in the Peruvian Soil - II to be on the order of 10 :Bq/g. Approximately 2100 bottles (100 g each) of pulverized, blended, and bottled Peruvian Soil - II were radiation-sterilized at Neutron Products, Inc. (Dickerson, MD) in preparation for distribution prior to inter-laboratory analyses for future issuance as natural matrix SRM 4355A. The NIST measurement design, <sup>242</sup>Pu tracer and bottles of Peruvian Soil have been delivered to National Laboratories (ORNL, SRNL, LANL, LLNL, PNNL, and NBL). Measurement results from the National Laboratories are expected within 3 months.

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### **Characterization of In Vivo Radioactivity Reference Phantoms**

Measurements of the Radioactivity Group whole-body counting system has been performed based on gamma spectrometry measurements of the Pacific Northwest National Laboratory (PNNL) Phantom Library. Efficiency assessment for Bottle Manikin Absorption (BOMAB) Phantoms are to be carried out for <sup>137</sup>Cs, <sup>60</sup>Co, and <sup>152</sup>Eu gamma emitters.

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# **Characterizing Anthropogenic and Natural Hot Particles**

Acid resistant hot particles have been isolated from the NIST Columbia River Sediment (SRM 4350B), Rocky Flats Soil (SRM 4353), and Ocean Sediment (SRM 4357), and confirmed by Fujii plate luminescence. The chemical compositions of these hot particles were determined by X-ray fluorescence and identified by x-ray diffraction as zircon. Massic activity measurements are to be completed summer 2009.

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Inn, K.G.W., "Metrology Infrastructure for Radioanalytical Emergency Response," Radioactivity Group Seminar, March 2007.

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