

## **Two-year Report to the BIPM 28 February, 2001**

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### **Scientific and Technical Activities**

***Cryogenic Calorimetry for Absolute Radioactivity Measurements.*** Several decades ago the NIST Radioactivity Group was a world leader in calorimetric measurements of radioactivity, although this capability was gradually lost over time. An initiative is currently underway to re-establish this basic measurement capability. To this end, a dual-compensated cryogenic calorimeter operating at 8 K was obtained from Science Research Laboratory (SRL) as part of a SBIR program. Extensive efforts are underway to get the calorimeter operational for use in absolute activity measurements of nuclides that decay by pure beta-particle emission and orbital electron capture. To date, all of the component parts have been assembled and preliminarily tested. This included acquisition and installation of a turbomolecular high-vacuum pumping station for the cryostat's vacuum chamber, plumbing of the mechanical refrigerator which utilizes a closed-cycle He compressor, start-up tests of the temperature control and power measurement circuitry and computer interface. The extant calorimeter, and as initially delivered by SRL, has never exhibited sufficient sensitivity or baseline stability to allow measurements in the required  $\mu\text{W}$  power range. Collaboration with SRL is underway to investigate unresolved problems with the instrument, and to possibly incorporate any needed design changes. (R. Collé)

***Evaluations and Upgrade of the  $^{222}\text{Rn}$  Pulse-Ionization-Chamber System.*** The national standard for radon measurements is embodied in a primary radon measurement system that has been maintained for nearly sixty years to accurately measure radon ( $^{222}\text{Rn}$ ) against international and national radium ( $^{226}\text{Ra}$ ) standards. All of the radon measurements made at NIST and the radon transfer standards and calibration services provided by NIST are directly related to this national radon standard. This primary radon measurement system consists of pulse ionization chambers and ancillary gas handling and gas purification equipment. As noted last year, efforts have been underway to re-evaluate the performance of the system and to upgrade some of its data acquisition components. Work has been completed on establishing revised gas-handling protocols, and these have been incorporated into routine quality control checks. A complete new set of data analysis and calculational procedures, including software revisions, for five specific measurement applications have been outlined, and are being incorporated. The upgrade of the data acquisition hardware and analysis software is also near completion. Final testing of the upgraded system will be initiated shortly. (R. Collé, M.P. Unterweger, P. Hodge)

***Upgrade of Gamma-ray Spectroscopy Facilities.*** The Radioactivity Group is upgrading its gamma-ray spectroscopy facilities in order to allow them to keep pace

with the growing demand for source analysis using this technique. The non-networked, UNIX-based data acquisition system currently in use is being converted to PC-based systems, which are on the NIST network. Some of the analysis software is being ported to the Windows environment, while other programs will be replaced by commercially available versions currently being evaluated. The detectors themselves are being moved to a larger laboratory to minimize interferences between detectors and to allow for the installation of additional detectors. These changes and subsequent recalibrations are being carried out in such a way as to not disrupt normal operations. (B.E. Zimmerman, L.R. Karam, J.T. Cessna, L. Pibida. M. Millican)

**Renovation of High-Level Radiochemistry Laboratory.** Calibrations of radionuclides performed by the Radioactivity Group have traditionally used samples that had activity concentrations on the order of  $\text{kBq}\cdot\text{g}^{-1}$  or lower. However, during the past few years, an increasing number of requests have been made by the nuclear medicine community for calibrations of samples with activity concentrations 3 orders of magnitude higher or more. This presents a number of logistical and safety concerns, which are being addressed by the renovation of the High-Level Radiochemistry Laboratory. In response to this problem, we undertook replacement of one of the existing fume hoods in order to construct a "hot cell", which, in conjunction with a remote manipulator arm, will be used to dispense and prepare sources for calibration. Other changes include the acquisition and installation of a new digital balance and shielding system, which will reduce the radiation exposure to workers preparing gravimetric sources. Two of the NIST re-entrant ionization chambers have also been interfaced to a PC computer that will control the chambers and acquire the data without the need for a person to be present in the room during the measurements. (B.E. Zimmerman)

**Phosphor Plate Imaging for Standards Development.** The storage photostimulable phosphor (SPP) imaging plate detection system, with pixel sizes as small as  $25\ \mu\text{m} \times 25\ \mu\text{m}$ , was originally developed for diagnostic radiography and they are sensitive to all types of ionizing radiation. Their usage ranges from the field of bio-medical research to material science and now is being applied for environmental radiation monitoring purposes. A typical SPP plate stores a radiation image as a distribution of F-centers in a thin coating of photostimulable phosphor ( $\text{BaFBr:Eu}^{2+}$ ). The latent image is read out by measuring the intensity of fluorescence stimulated by scanning a He-Ne laser beam over the surface of the plate for a few minutes. We have been conducting measurements using the owned Fuji BAS 2000 SPP system to determine its applicable measurement limits for various types of radioactivity and to suggest operation procedures tailored to different types of radiation and radioactivity levels. These measurements are designed to realize the potential of this SPP system as a radiation imaging device, a quantitative measurement instrument and a research tool to design counters for ultra low level radioactivity measurement required by the electronic and aerospace industry. (Y.T. Cheng, L.R. Karam, M.P. Unterweger)

**Calibrations of Large-Area Beta Sources.** Studies are continuing on the effects of beta-backscattering to develop a systematic method for determining the effective source thickness needed for relating the measured rate to activity. These determinations will be used to calibrate surface monitoring equipment in terms of radioactivity rather than emission rate. Nuclear Regulatory requirements are that the surface contamination be measured in radioactivity quantities when determining the necessary steps needed for remediation of nuclear sites. An accurate determination can be cost-effective in deciding what, if any, “cleanup” is required. This will be incorporated into an ANSI standard for the use of large-area beta sources. (M.P. Unterweger, P. Hodge).

**Counting Yields for Beta and Alpha Particle Sources.** Using the results of Monte Carlo calculations, the counting yields of beta- and alpha-particle sources have been determined and tabulated. The counting yield is defined as the fraction of the emitted particles that emerge from the source and are counted by a 2B detector, and takes into account backscattering and self-absorption in the source. The results apply to sources consisting of a thin layer of radioactive material placed on top of, or distributed in a top layer, of a thick metal backing. (M.J. Berger, L.R. Karam, M.P. Unterweger, J.M.R. Hutchinson)

**Liquid Scintillation Cocktail Composition and Mismatch Effects.** Liquid scintillation (LS) spectrometry with  $^3\text{H}$ -standard efficiency tracing is one of the Radioactivity Group's principal and powerful measurement tools and is being applied to many newly-standardized, important nuclides such as those used in nuclear medicine. In the past few years, a variety of subtle and perplexing LS cocktail composition effects have been observed in various calibrations. Most of these effects appear to arise because of the existence of different chemical species in the various cocktails (e.g., between the  $^3\text{H}$  standard and the nuclide being calibrated). As a result, efforts to understand these effects through broad, systematic evaluations are ongoing. (R. Collé, B.E. Zimmerman, J.T. Cessna)

**Characterization of Commercial Re-entrant Ionization Chambers as Secondary Standards for Nuclear Medicine.** One of the properties of most nuclides used in nuclear medicine that make them so attractive for such applications, namely the short half-life, unfortunately has the drawback of making it impractical to distribute solution standards as Standard Reference Materials (SRMs). Regulatory agencies such as the NRC and FDA, however, still require that users and producers of radiopharmaceuticals containing these radionuclides demonstrate the ability to accurately measure the amount of contained radioactivity in a sample prior to administration. One way that NIST is attempting to solve this problem is through the development of calibration factors for commonly used, commercially available, re-entrant ionization chambers, or “dose calibrators”, that can be used as secondary standards in the clinic. In order to be used with a large degree of confidence, however, it is necessary to fully characterize the uncertainties involved with such measurements. As part of a continuing project, we have developed a measurement

model to allow for the determination of uncertainties involved with the actual derivation of the calibration factor and have recently expanded the program to include the uncertainty due to variation between different individual chambers and variations among similar chambers from the same manufacturer. We plan to conduct one such study, using  $^{166}\text{Ho}$ , to study the variability in measurements among chambers from Capintec (4 chambers), AtomLab (2 chambers), and Vinten (1 chamber). All future experiments involving the derivation of calibration factors will include measurements on chambers from each of these manufacturers. (B. E. Zimmerman, J. T. Cessna, M. A. Millican)

**Reevaluation of the Half-Life of Tritium.** A remeasurement of the half-life of tritium has also been done and a reevaluation of the half-life using all reported values has been published. An accurate value for the half-life is very important in extending the useful lifetime of the tritium standards. (L.L. Lucas, M.P. Unterweger)

**Gas Counting of  $^{37}\text{Ar}$ .** A sample of  $^{37}\text{Ar}$  from Max-Planck-Institut für Kernphysik, Heidelberg, Germany, was measured and used to calibrate the gain settings for the internal gas counters. (M.P. Unterweger)

**Calibration of  $^{68}\text{Ge}/^{68}\text{Ga}$  Source.** The radionuclide  $^{69}\text{Ge}$  and its positron-emitting daughter,  $^{68}\text{Ga}$ , are often used as long-lived (270 d) sources to calibrate detector systems for positrons as well as the 511-keV radiation that results from their annihilation. A NIST Physics Laboratory Researcher in Boulder was using such a commercially available source to calibrate his instrumentation and observed a factor of 3 discrepancy between his expected yields based on the activity certificate from the manufacturer and his experimental data. Although the Radioactivity Group has not yet standardized these radionuclides, we were able to perform an activity measurement on the source using high-purity germanium gamma-ray spectrometry. The expanded uncertainty was 7.6 % and was mainly due to uncertainties in fitting the 511-keV annihilation peak and the uncertainty in the decay data. Despite the relatively large uncertainty, we were able to determine that the manufacturer's certified value was not the source of discrepancy, although the manufacturer had understated their uncertainties by a factor of about 5. Further discussions with the NIST collaborator led to changes in his experimental setup that removed most of the discrepancy. (B. E. Zimmerman, L. Pibida, L. R. Karam)

**Holmium-166m.**  $^{166\text{m}}\text{Ho}$  is a long-lived (the half-life is 1200 years) radionuclide that emits a large number of gamma rays with energies from 80 keV to more than 1400 keV. The large number of gamma rays, the wide energy range, and the long half life make  $^{166\text{m}}\text{Ho}$  a very desirable gamma-ray source for determining the detection efficiency of germanium detectors and for monitoring their long-term stability. High-purity stable  $^{165}\text{Ho}$  was neutron irradiated to produce  $^{166\text{m}}\text{Ho}$ , which is now being calibrated in terms of activity. The gamma-ray emission probabilities and their uncertainties are also being evaluated. SRM 4274,  $^{166\text{m}}\text{Ho}$  solution, will be issued in FY 2001. (L.L. Lucas, B.E. Zimmerman, L.R. Karam)

***Deadtime Effects in Liquid Scintillation Counting of the  $^{222}\text{Rn}$  Subseries.***

Experiments are underway, in collaboration with the Henri Becquerel National Laboratory (HBNL) in Saclay, France (formerly LPRI), to evaluate the magnitude of the deadtime loss effect for measurement of the 165- $\mu\text{s}$   $^{214}\text{Po}$  nuclide (a member of the  $^{222}\text{Rn}$  subseries) in commercial liquid scintillation (LS) spectrometers. At Saclay, using their triple-to-double-coincidence (TDCR) LS spectrometer, we recently measured the counting loss of  $^{222}\text{Rn}$  (in radioactive equilibrium with its four decay products) as a function of an imposed extended deadtime, over a range of  $\tau = 8 \mu\text{s}$  to 50  $\mu\text{s}$ , and made determinations of the deadtime distributions. The apparent losses for  $^{214}\text{Po}$  ranged from less than 4 % at  $\tau < 10 \mu\text{s}$  to about 21 % at  $\tau = 50 \mu\text{s}$  which correspond to overall  $^{222}\text{Rn}$  subseries efficiency losses of less than 1 % to about 4 %. Ancillary experiments demonstrated that the observed deadtime loss effect was not an artifact of the TDCR spectrometer. Future experiments that have been designed include extending these results to commercial LS spectrometers (for all three principal spectrometer manufacturers), and intercomparisons of the NIST radon-in-water standard generator and the NIST radon LS calibration with the HBNL cryogenic radon standard and their TDCR LS spectrometer. This work comprises one of the last remaining pieces of the decade-long effort to establish liquid-scintillation-based calibrations for the  $^{222}\text{Rn}$  subseries and for  $^{226}\text{Ra}$  in the presence of the  $^{210}\text{Pb}$  subseries. (R. Collé)

***Development of Radioactivity Standards for Alpha-Emitting Radiotherapy Nuclides.***

There has been a great deal of interest during the past few years in the development of radioimmunotherapy (RIT) agents using radionuclides that emit  $\alpha$ -particles. These agents are particularly attractive because  $\alpha$ -particles impart a very high radiation dose over a short linear range, typically 2 to 4 cell diameters. When coupled to tumor-specific antibodies, the  $\alpha$ -emitting radiopharmaceutical is able to deliver a high dose to the tumor, while sparing healthy tissue. This type of therapy would be especially suited for single-cell diseases such as myelogenous leukemia. Human trials using  $\alpha$ -emitters are currently underway at Memorial Sloan-Kettering Cancer Center (MSKCC), (using  $^{213}\text{Bi}$  to treat patients with acute myelogenous leukemia) and at Duke University (investigating the efficacy of  $^{211}\text{At}$ -labeled antibodies to treat glioma). The results to date appear promising. Many questions remain, however, regarding the accuracy with which the radioactivity of radiopharmaceuticals containing these radionuclides is being measured. The de facto standard instrument for measuring radioactive solutions in hospitals and clinics is the re-entrant ionization chamber, or "dose calibrator". Correct use of these instruments requires that the correct instrument calibration factor, or "dial setting" be applied for the specific nuclide or nuclides (when multiple daughters are found in equilibrium with their parent nuclides) in the exact geometry for which the dial setting was determined. Currently, there is no such dial setting available for either  $^{211}\text{At}$  or  $^{213}\text{Bi}$  for the most popular commercially available dose calibrator, manufactured by Capintec. There is a dial setting available for  $^{212}\text{Bi}$ , but it is valid only when the solution is contained in a 5 mL thin-walled glass ampoule, which is rarely encountered in a clinical setting. Differences in glass thickness or composition

have been found to lead to observed changes of up to 5 % or more in activity measurements made with these devices. Thus, correct dial settings for each of the nuclides presently being investigated need to be accurately determined for clinically useful geometries. The only way this can be accomplished is by using accurately calibrated sources. We have begun experiments aimed at developing a standard for  $^{211}\text{At}$  in collaboration with the Positron Emission Tomography Group at NIH. Initial studies suggest some degree of chemical volatility, which could preclude the use of solid, uncovered sources for calibration. Moreover, variability in measurement results due to differences in the responses of the liquid scintillation spectrometers indicates the need to address the issue of optical effects that can lead to inconsistent results. (B. E. Zimmerman, J. T. Cessna, M. A. Millican)

***Cocktail Composition Effects in 4pb Liquid Scintillation Spectrometry in the Standardization of Radionuclides Used in Nuclear Medicine.*** Liquid scintillation (LS) counting continues to be the method preferred by most metrology laboratories for performing quantitative assays of solutions containing  $\beta$ -emitting radionuclides. This is primarily due to the inherently high detection efficiency and ease of sample preparation. Under many circumstances, there is little need to be concerned about the composition of the analyte solution, as most changes in detection efficiency arising from chemical effects can be accounted for by efficiency tracing against an established standard. However, there can be cases in which the solution composition can affect the efficiency in ways that cannot be accounted for using efficiency tracing techniques. Studies carried out in this laboratory on nuclides such as  $^{55}\text{Fe}$ ,  $^{63}\text{Ni}$ , and  $^{90}\text{Y}$  suggest that the concentration of nonradioactive ions, the presence of chelators, the choice of commercial scintillant, and the pH of the analyte solution can play crucial roles in the chemical stability of the LS cocktail. We have been carrying out experiments aimed at identifying composition variables that influence LS cocktail stability and subsequent assay results for solutions containing the rare-earth radionuclide  $^{177}\text{Lu}$ . Liquid scintillation cocktails were prepared in commercially available scintillation fluids with three solutions of  $^{177}\text{Lu}$ : one with a high ionic concentration (0.06 g of nonradioactive  $\text{Lu}^{+3}$  "carrier" ions per mL of 1 mol  $\cdot$  L $^{-1}$  HCl), another having minimal ionic concentration (0.3  $\mu\text{g} \cdot$  mL $^{-1}$   $\text{Lu}^{+3}$  in 0.01 mol  $\cdot$  L $^{-1}$  HCl), and a third having an ionic concentration similar to the second solution, but with the  $^{177}\text{Lu}$  bound to a peptide. In the case of the high concentration samples, the total ionic concentration in the cocktail was varied by the addition of distilled water or 1 mol  $\cdot$  L $^{-1}$  HCl. For the low-ion, unbound samples, the effect of chelation was explored by the addition of 0.1 mol  $\cdot$  L $^{-1}$  tetrasodium ethylenediaminetetraacetic acid (EDTA). In order to monitor chemical effects, cocktails containing a tritiated water standard were prepared so as to have nominally identical compositions as the  $^{177}\text{Lu}$  cocktails. The results indicate that the cocktails having high ionic concentrations exhibit relatively good stability over a long period of time in each of the commercial scintillants investigated. In contrast, the low-ion samples exhibited a marked instability in scintillants design for high sample loading, giving decay-corrected activity values that dropped by about 0.8 % per day with no change in quench indicating parameter or tritium efficiency. In cocktails designed for much smaller

sample loading, however, the samples were stable. Chelation was shown to be effective in maintaining stability in the cocktails studied. In none of the cases studied was there any effect on the tritium samples. Further experiments are planned to elucidate the mechanism of these effects.

Studies have also been carried to identify issues dealing with the use of commercial scintillants to count somewhat strongly alkaline solutions, specifically those containing  $^{188}\text{W}$  in equilibrium with  $^{188}\text{Re}$ , in which the solution is prepared with nominally 1 N NaOH. Unfortunately, information as to the ability of these scintillants to handle alkaline samples with moderate to high ion concentration is not widely available from the manufacturers. A total of four commercial scintillants were studied: Packard Ultima Gold AB, Packard Hionic Fluor, Packard Insta-Gel XF, and Wallac OptiPhase Hi-Safe III. The water fractions of the cocktails were adjusted by the addition of between 0.02 g and 1 g of water or 1.0 N NaOH to nominally 10 mL of scintillant, with the exception of the Insta-gel cocktails, which contained 7 mL of scintillant and 5 mL of either water or 1 N NaOH. The quenching range in the cocktails was altered by the addition between 1 and 12 drops of a solution of nitromethane diluted with ethanol. In addition to the  $^{188}\text{W}$  cocktails, an additional series of samples were prepared with tritiated water in such a way as to be chemically identical with the  $^{188}\text{W}$  cocktails. All cocktails were counted in each of three spectrometers for 10 cycles each. The results indicate that the Insta-Gel cocktails prepared with NaOH as the primary aqueous component decomposed, resulting in a white precipitate, but that those prepared with water as the aqueous component were stable. In addition, Ultima Gold AB was shown to be unstable in the presence of NaOH and gave widely varying results, depending upon the aqueous (NaOH) fraction of the cocktail. The Hionic Fluor and Hi-Safe III cocktails were all stable over time and gave good results, despite the fact there was an observed difference of about 0.4 % in the averages of the activities determined for each cocktail. The Insta-Gel cocktails prepared with water gave activity values that were in excellent agreement with those obtained with HiSafe III. (B. E. Zimmerman, J. T. Cessna, M. A. Millican)

### ***Quantitative Destructive Assay Technique for Radioactive Coronary Stents.***

Nearly one half million coronary angioplasty procedures are performed every year in the United States. In up to 50 percent of these cases, the patient will experience restenosis, or re-closing, of the artery walls. One method used to prevent this phenomenon is the permanent placement of a metal stent that expands to form scaffolding to hold the artery walls open. Unfortunately this has not proven effective in every case and a re-examination of the site will find scar tissue growing through the stent. Another method employed is intravascular brachytherapy, where the area is subjected to a dose of radiation, at a level known to inhibit proliferative cells, such as scar tissue. Recently a Belmont, California company, IsoStent, has combined these two methods by developing a stainless steel stent containing the beta emitter phosphorus-32. In order for correct dosimetry calculations to be performed, and as a check of the manufacturing process, it is necessary for the activity contained in a stent to be accurately measured. However given the nature of the sample and radionuclide it is not possible to measure the activity directly in an intact stent. For

this reason the Radioactivity Group has developed quantitative destructive assay techniques for this type of source.

A series of 36 stents in varying lengths and activity levels were supplied to the Group. The stents were first intercompared in a NaI well crystal  $\gamma$ -detector, looking at the bremsstrahlung emitted. A subset of the stents were then slowly digested in a small volume of carrier solution, with the addition of a few drops of concentrated hydrofluoric acid. All components were gravimetrically added to ascertain the final composition and total mass of the digestion solution. Aliquots of this solution were then liquid-scintillation counted using the CIEMAT/NIST method of efficiency tracing against carefully matched tritium samples, prepared with identical digestions of blank stents. The resulting activity concentration was multiplied by the total mass of the digestion solution to give the total activity contained in the stent. This subset of stent activities were then used to derive calibration factors for the NaI system, which were in turn used to derive the total activity contained in the remaining undigested stents. Several checks were performed throughout the experiment to ensure there was no activity lost in any of the transfers or containers. The expanded ( $k=2$ ) uncertainties on the activities were on the order of 1.1 percent – 2.1 percent for the digested stents and 1.5 percent – 2.6 percent for the undigested stents. (J.T. Cessna)

#### ***Calibration of Pure-Beta-Emitting Intravascular Brachytherapy Sources.***

Considerable work has continued on providing NIST-based activity calibrations for manufacturers of intravascular brachytherapy sources. These sources are intended for use in the prophylactic inhibition of restenosis following balloon angioplasty in heart-disease patients. Their use has an ultimate potential of possibly serving over 600,000 patients per year in the US alone, with an economic value in excess of a billion dollars. In the past year, three sets of nondestructive ionization-chamber-based calibrations were performed for  $^{32}\text{P}$  "hot-wall" angioplasty-balloon catheter sources that are manufactured by Radiance Medical Systems Inc. (Irvine, CA). These calibration measurements utilized previously established ionization-chamber calibration factors that were in turn derived from destructive assays. The results of the calibrations were used by Radiance for internal quality control and to transfer the NIST calibrations to two other calibration laboratories, *viz.*, the Radiation Calibration Laboratory at the University of Wisconsin-Madison and the Physikalisch - Technische Bundesanstalt (PTB) in Braunschweig, Germany. Independent destructive assays of the  $^{32}\text{P}$  content of balloon catheters by PTB were in agreement with the NIST to within less than 1 %. Ionization-chamber-based calibrations were also performed on the Novoste Corp. (Norcross, GA) sources. These are stainless-steel-encapsulated, ceramic-based  $^{90}\text{Sr}$ - $^{90}\text{Y}$  sources whose calibration factors were also derived from earlier destructive radionuclidic assays by NIST. (R. Collé)

***Development of Radioactivity Standards for New Brachytherapy Device for Brain Cancer Therapy.*** Proxima Therapeutics, Inc. (Alpharetta, GA), is developing a new radiation treatment technique to prevent the recurrence of aggressive brain tumors after surgical resection. The American Cancer Society projects 18,000 U.S.



patients will be diagnosed each year with malignant brain tumors and nearly all experience tumor re-growth after initial treatment. Most tumors recur within a short period of time and more than 80 percent are located within two centimeters of the original cancer site. This new treatment modality involves the implantation of a small balloon device, named the "GliaSite", in the cavity that remains after the tumor is removed. The balloon is subsequently filled with a solution containing radioactive  $^{125}\text{I}$ , marketed under the trade name "Iotrex". Treatment planning for brachytherapy of resected brain tumors with the GliaSite and Iotrex requires dosimetry estimates based on accurate  $^{125}\text{I}$  activity measurements using commercially available dose calibrators. The Iotrex is shipped to the hospital in 2 mL conical vials and assayed/administered in 5 mL syringes. A correct calibration factor or "dial setting" is required for each source geometry and dose calibrator model that may be used. The low photon energies of  $^{125}\text{I}$  (<35 keV) make its assays sensitive not only to the type and construction of the measurement container, but also to the filling volume. The radioactivity Group has experimentally analyzed the response of Capintec CRC-12, CRC-35R, and Vinten 671 dose calibrators to NIST-calibrated solutions of Iotrex. Measurements were conducted in 5 mL BD plastic syringes over a volume range of 1 mL to 3 mL and 2 mL glass conical v-vials over the volume range 0.2 mL to 2 mL. New calibration factors and dial settings were determined from these data. The results indicate use of the dose calibrator manufacturer's recommended dial setting produces errors in the activity assay of up to +16% and -55% for the syringes and vials, respectively, depending on the filling volume. For syringes holding 1.0 to 3.0 mL of Iotrex, the experimentally determined dial setting for the CRC-12 ( $497\pm 8$ ) was shown to have an expanded ( $k=2$ ) uncertainty of 1.6 %. Further experiments were carried out that characterized the measurement uncertainty components of repeatability and reproducibility and were found to be about 0.3 % and 0.6 %, respectively. The use of these new dial settings and correction factors significantly improves the accuracy of Iotrex assays. Furthermore, these data constitute an important part of the documentation that must be submitted by Proxima in order to receive FDA approval for the device. (B. E. Zimmerman, J. T. Cessna)

**Development of  $^{166}\text{Ho}$  Standard.** Due to the decay scheme of  $^{166}\text{Ho}$ , the same properties that make it desirable as a therapeutic radionuclide in turn make it necessary to take care when measuring the radionuclide with an ionization chamber. This is true of both the NIST primary ionization chamber "A" and the commercial ionization chambers, or "dose calibrators," used in both the manufacturing process and eventually the clinic.

A solution of  $^{166}\text{Ho}$ , as a chloride in  $0.3 \text{ mol XL}^{-1} \text{ HCl}$ , was supplied by MURR. This solution was used to prepare several gravimetrically related series of samples for a variety of measurements. A series of 3 NIST 5-mL ampoules was prepared for measurement on the NIST chamber "A", an NPL ionization chamber, the commercial dose calibrators residing at NIST, and a commercial dose calibrator belonging to I<sup>3</sup>. While one of these ampoules was kept intact for subsequent half-life measurements on chamber "A," another was divided and diluted into 3 20-mL dose vials, each

containing a total volume of 16 mL. These dose vials were first measured on the dose calibrators and the NPL chamber and shipped to the customers for measurement in their own dose calibrators. The final ampoule was divided into two series of sources for chamber "A" and a dilution to be used as a master for liquid-scintillation measurements. The two series of sources for chamber "A" were designed to vary in one series the acid concentration of the solution and in the other the carrier concentration. Liquid-scintillation (LS) measurements were performed on the final dilution using the CIEMET/NIST method of tritium-efficiency tracing. The results of these LS measurements were used to produce an equation describing the effect of acid concentration and carrier concentration on the calibration factor for NIST chamber "A." Finally, sources were prepared at two levels of this dilution scheme for measurement of impurities on High-Purity Germanium detectors.

The complexity of this dilution and measurement scheme required the assistance and cooperation of a large number of members of the Radioactivity Group, Research Associates from the Nuclear Energy Institute, and the customers themselves. More work is ongoing to extend the dissemination of the national standard to the complexed form of the radiopharmaceutical. (J. T. Cessna, B. E. Zimmerman, M. P. Unterweger, L. R. Karam, M. A. Millican)

**Development of Radioactivity Standard for  $^{188}\text{W}/^{188}\text{Re}$ .** The availability of the  $^{188}\text{W}/^{188}\text{Re}$  generator has increased interest in using  $^{188}\text{Re}$  for a wide variety of nuclear medicine applications. The daughter,  $^{188}\text{Re}$ , is milked from the generator and is currently under investigation for radioimmunotherapy, radiation synovectomy, intravascular brachytherapy, and as a bone palliation agent. Wires containing  $^{188}\text{W}$  are currently being studied for possible applications in intravascular brachytherapy. Because of its short ( $t_{1/2}=17$  h) half-life, it is impractical to produce a Standard Reference Material (SRM) of  $^{188}\text{Re}$  to distribute to the large number of users worldwide. Rather, by producing a standard of  $^{188}\text{W}$  in secular equilibrium with  $^{188}\text{Re}$ , users can calibrate their instrumentation for  $^{188}\text{Re}$  with a source that has a relatively long ( $t_{1/2}=69$  d) half-life. Using liquid scintillation counting, we have succeeded in calibrating solutions of  $^{188}\text{W}$  in equilibrium with  $^{188}\text{Re}$  with an expanded uncertainty ( $k=2$ ) of 0.79 %. This calibration was transferred to the NIST "4 $\pi$ "  $\gamma$  ionization chamber to facilitate subsequent calibrations. Calibration factors were also derived for the Capintec CRC-12 and CRC-35R dose calibrators maintained at NIST in the standard 5 mL ampoule geometry. (B. E. Zimmerman, J. T. Cessna)

**Application of SPP to Personnel Radioactivity Monitoring.** Accidental exposure to radiation is, by definition, unplanned, and normal dosimetric procedures (such as the wearing of radiation "badges") used to assess radiation exposure are not applicable. Monitoring often requires retrospective analyses of available samples from the victims or their clothing in order to evaluate individual exposure. Phosphor plate imaging is being applied in the development of methods to map the distribution of radioactivity in dental tissue as a possible assessment tool for the accidental ingestion of radioactive materials.

Internally deposited radionuclides, such as  $^{90}\text{Sr}$  (half-life 28.79 years), when competing with calcium, are preferentially deposited in stable (with low turnover) tissues of the body, particularly the teeth and bones. Fuji plate imaging potentially can be used to map radionuclide distribution either in excised teeth or *in situ* (i.e., in the mouth itself). In order to obtain reliable and reproducible results, we have been calibrating the Fuji imaging system with well defined  $^{90}\text{Sr}/^{90}\text{Y}$  point sources to give a first approximation of a surface deposition on tooth. We are continuing the development of a calibration curve in the range of apparent activity anticipated in tooth samples (from 0.2 to 0.5 Bq). In addition, the minimum detection level, influence from background radiation, and reproducibility of the technique over time (i.e., quantification of the image when fading due to exposure time becomes important) are being investigated. Our next step will be to “imbed” the radionuclide (by placing various layers of dental-mimic material between point sources and the imaging plate) to mirror the depth distribution within a tooth so as to account for absorption, attenuation and back-scatter which can all confound quantitative results. (L.R. Karam, M.P. Unterweger)

**Application of SPP to Cell-Radiolabelling Efficiency.** In collaboration with members of CSTL and as part of a contract effort for the Air Force, we have studied the application of SPP to monitor the efficiency of bacterial cell labelling with radioactive isotopes. We have measured the activity of  $^{32}\text{P}$ -labelled *bacillus* on air sampling filters with the SPP system. Using this imaging plate technology, we were able to obtain results 2 orders of magnitude more sensitive than with previously used methods. (L.R. Karam, C. O’Connell [CSTL])

**Resonance Ionization Mass Spectrometry.** RIMS has been evaluated for measuring  $^{135}\text{Cs} / ^{137}\text{Cs}$  isotopic ratios. Spectroscopic measurements of  $6s\ ^2S_{1/2}(F = 4) \rightarrow 6p\ ^2P_{3/2}(F = 5)$  transition frequency shifts for  $^{135}\text{Cs}$  and  $^{137}\text{Cs}$  confirmed existing values and demonstrated that it is possible to perform such measurements on sub-picogram samples. Optical isotopic selectivity of  $\sim 10^3$  for both  $^{135}\text{Cs}$  and  $^{137}\text{Cs}$  against stable  $^{133}\text{Cs}$  was observed and, when combined with a quadrupole mass spectrometer, overall selectivity of greater than  $10^9$  was demonstrated. This selectivity appears to be limited by neutral particles generated during the atomization of the samples and could possibly be improved by using a non-axial geometry that prevents direct line-of-sight transport between the atomization source and the ion detector region. Because of the inherent elemental selectivity of the resonance ionization process, no interference could be observed from barium isobars in the RIMS measurements. Overall detection efficiency for the RIMS process was found to be  $1 - 2 \times 10^{-6}$ , limited in approximately equal parts by the efficiency of converting Cs salts in the atomization source to neutral gaseous Cs atoms and the efficiency of ionizing the Cs atoms entering the laser beams. Test measurements on samples containing as much as  $4 \times 10^8$  excess of  $^{133}\text{Cs}$  were performed and demonstrated detection limits of  $1 - 2 \times 10^8$  atoms. While this only corresponds to  $\sim 100$  mBq for  $^{137}\text{Cs}$ , the activity equivalent for  $^{135}\text{Cs}$  is  $\sim 10^{-3}$  mBq, which could not be detected by normal decay counting methods.

Further, both radioisotopes can be measured simultaneously by the same method, and this method is independent of the nuclear decay properties. Isotope ratios for  $^{135}\text{Cs} / ^{137}\text{Cs}$  were compared using both RIMS and conventional TIMS and found to be in excellent agreement. The isotope ratio measurements were able to precisely date a standard sample whose isotopic composition had been accurately measured two decades previously; however, measurements on another standard with unknown  $^{135}\text{Cs}$  content yielded an anomalous ratio that illustrates problems that may arise because of the neutron-flux dependent fission yield of  $^{135}\text{Cs}$ . However the neutron-flux dependence for the fission yield of  $^{135}\text{Cs}$  can provide information about the origin of the samples studied. (L. Pibida, L.R. Karam, J.M.R. Hutchinson)

***Energy Deposition and Radiation Quality of Radon and Radon Daughters.*** We have created a quantitative description at the micrometer and nanometer level of the physical interactions of the alpha particles from radon and its daughters with the cells at carcinogenic risk in the lung and bronchial epithelium. This information is basic to a detailed understanding of the mechanisms of the biological effectiveness of radon and provides input information to biophysical and biochemical models of radiation interaction (collaboration with Dr. Werner Hofmann, University of Salzburg, Austria). The results of these calculations should improve our understanding of the interaction of radon and radon daughter alpha particles with the relevant tissues through comparison with experimental biophysical, biochemical and biological information. (L.R. Karam, R.S. Caswell)

***Status of Low-level Radiochemical Analysis in the US.*** The NIST Radiochemistry Intercomparison Program has four years of performance evaluation data from fifteen commercial, university, advocacy, National, and Federal Agency laboratories across the US for  $^{90}\text{Sr}$ ,  $^{238}\text{U}$ ,  $^{238}\text{Pu}$ ,  $^{240}\text{Pu}$ ,  $^{241}\text{Am}$ , at 0.03-0.3 Bq per sample in water, air-filters, soil, synthetic urine, and synthetic feces. A systematic evaluation of the data provides a glimpse at the status of low-level radiochemical analyses in the U.S. The relative mean difference from the NIST massic activity values across all matrices and radionuclides ranged from -1 to -6 percent with standard deviation of the means from 2 to 7 percent. Although there is an improvement in laboratory performance since the inception of NRIP, analysis of variance indicated that the analytical methods used was a factor in the difference of measured  $^{241}\text{Am}$  and  $^{90}\text{Sr}$  values from the NIST massic activity values, whereas the test matrix was a determining factor for  $^{238}\text{U}$  analyses. Furthermore, about 90 percent of traceability evaluations were acceptable when compared to the ANSI N42.22 criteria for performance evaluations. The majority of the failures to pass the ANSI N42.22 criteria was due to unrealistically small estimates of measurement uncertainties. The Radioactivity Group has provided workshops to assist the NRIP participants with estimating their measurement uncertainties. (Z.Y. Wu, C. McMahon, Z. Lin, K.G.W. Inn)

***Radioanalytical Traceability.*** Over the past several years, NIST has been working with the American National Standards Institute's nuclear instrumentation N42 and N13 committees to establish widely accepted criteria for radioanalytical traceability. Three standards have been published: 1) ANSI N42.23 (Measurement and Associated

Instrumentation Quality Assurance for Radioassay Laboratories), 2) ANSI N42.22 (Traceability of Radioactive Sources to the National Institute of Standards and Technology and Associated Instrument Quality Control), and 3) ANSI N13.30 (Performance Criteria for Radiobioassay). Each of these standards were developed through consensus participation among industrial, commercial, utility, federal, state, national laboratory representatives to strengthen the credibility of national radioanalytical programs. ANSI N42.23 envisions the accreditation of a small group of program-specific reference laboratories that participate directly in a traceability-testing program with NIST, technical document reviews and on-site assessments. These reference laboratories would act as intermediaries in the establishment of the traceability testing link to the service laboratories through Performance Testing (PT) programs. One current driving force to establish reference laboratories is the privatization of the EPA Drinking Water Crosscheck Program that will require the NVLAP accreditation of PT Providers for drinking water laboratories. The traceability testing criteria for radiobioassay PT reference laboratories defined by ANSI N13.30 are similar. A third sector that will be establishing traceability testing for its reference laboratories is DOE-EM for its environmental remediation work. The ANSI N42.22 standard provides additional and more specific criteria for source manufacturers, including those reference laboratories that will be producing PT materials. In addition to the criteria for quality assurance, facilities and equipment, and certificates, ANSI N42.22 provides very specific criteria for the acceptance of testing results:

$$|V_R - V_N| \leq 3 \times (\sigma_N^2 + \sigma_R^2)^{0.5}$$

where the absolute value of the bias between the reported value,  $V_R$ , and the NIST value,  $V_N$ , shall be less than or equal to three times the total propagated NIST uncertainty,  $\sigma_N$ , and the reported uncertainty,  $\sigma_R$ .

Furthermore, the right hand side of the equation defines the “traceability limit” to which a source manufacturer could claim traceability. It is anticipated that traceability testing for the reference laboratories supporting these four national sectors will be initiated by the end of 2001. (Z.C. Lin, K.G.W. Inn, Z.Y. Wu, C. McMahon)

***Second Intercomparison Study for the Determination of  $\mu\text{Bq}$  Levels of  $^{239}\text{Pu}$  in Synthetic Urine by Mass Spectrometric and Fission Track Analysis.*** The Department of Energy, Office of International Health Programs (EH-63), is in the process of assisting Marshall Islanders to resettle their islands after five decades. The DOE and the resettled residents require assurances that the radiation dose to residents will not exceed recognized international standards or recommendations. One of the remaining radionuclides that could contribute to internal radiation dose from inhalation and ingestion intake pathways is Pu. The uptake of Pu is estimated from the excretion of Pu in the urine of an individual. The analytical technique must

have sufficient sensitivity to quantify Pu isotopes at or below a level of 20  $\mu\text{Bq}/\text{kg}$ .{PRIVE }

The goal of this project was to evaluate the state-of-the-art (accuracy, precision and minimum detection amount) for Pu in artificial urine measurements by inductively coupled plasma, thermal ionization mass spectrometry and fission track analysis in the range of 18-278  $\mu\text{Bq}/\text{sample}$  (approximately 3 million atoms of plutonium per 200 g sample). In completion of the first intercomparison study in 1997, all mass spectrometric and fission track analysis have shown the capabilities of making quantitative measurements of  $^{239}\text{Pu}$  from urine down to the 18  $\mu\text{Bq}/\text{sample}$  level. However, the reliability of the measurement were limited by some potential technical difficulties, such as data transcription, sample tracking, estimation of limits of detection, statistical control over analytical processes, and control of analytical background, for some of the participating laboratories and showed room for improvement. A workshop was held for planning the second intercomparison study in Los Alamos National Laboratory on September 28, 2000. The participants discussed the issues in analytes, concentration levels, final matrix, desired volume, interfering radionuclides, reservation, container type, data reporting, and acceptance criteria. The second intercomparison has been planned to include more blank samples and interfering isotopes of  $^{240}\text{Pu}$  and natural U for assessing the technologies under more realistic conditions. The intercomparison samples will be prepared in the NIST's clean room facilities and verified by gamma tracing techniques. The stability of Pu in the artificial urine during dilutions, conformational measurements, and storage will also be evaluated using  $^{139}\text{Ce}$  which possess similar chemical properties. (Z.C. Lin, K.G.W. Inn)

***NIST Radiochemistry Intercomparison Program (NRIP)***. The NIST Radiochemistry Intercomparison Program NRIP marks the successful completion of the fourth year of its measurement traceability program for low-level environmental radioactivity measurements. Four rounds of testing (five total matrices were offered: water, air-filters, soil, synthetic urine, and synthetic feces) were completed. The analyte list include  $^{90}\text{Sr}$ ,  $^{234}\text{U}$ ,  $^{238}\text{U}$ ,  $^{238}\text{Pu}$ ,  $^{240}\text{Pu}$ ,  $^{241}\text{Am}$ , at 0.03-0.3 Bq per sample. Participation in the program include: NM State Carlsbad Environmental Monitoring and Research Center, Evaluation Group, Martin Idaho Technologies Company, Institute of Nuclear Energy Research (Taiwan), Los Alamos National Laboratory, EPA Air and Radiation Environmental Laboratory, Oak Ridge National Laboratory's Bioassay Laboratory, Oak Ridge National Laboratory's Intercomparison Studies Program, Oak Ridge Institute for Science and Education, STL Richland Laboratory, Sandia National Laboratory, and Westinghouse Waste Isolation Pilot Plant. The program is vital for relating low-level radioanalytical measurements to the National Standards.

The program has been implemented to meet the demand for traceability as defined under ANSI N42.23. This voluntary standard defines a hierarchy of traceability with an unbroken linkage from service laboratories to NIST. Under the standard, the testing materials must be (1) of appropriate matrices (i.e., matrix categories commonly analyzed by the laboratory); and (2) of appropriate (ambient) activity

concentration ranges for these matrices. Currently NRIP is the only traceability program providing performance evaluation materials at environmental levels. The acceptance criteria as defined in ANSI N42.22 "Traceability of Radioactive Sources to the National Institute of Standards and Technology and Associated Instrument Quality Control" has been adopted by the NRIP group. The ANSI N42.22 standard defines the requirements for traceability for radioactivity-source manufacturers. The NRIP group has adopted the performance testing criteria of the standard, which states that for traceability the difference between reported results and the NIST certified values must be less than three times the total propagated uncertainty for the measurement. The standard establishes further the quantity "three times the total propagated uncertainty" as the "Traceability Limit" the limit to which a source manufacturer may claim traceability to NIST. Following each test, participating laboratories are issued Reports of Traceability, which reflect both the relative bias (compared to the NIST values) and the Traceability Limit of the measurements as defined in ANSI N42.22.

The test matrices and analytes for each fiscal year are to be determined at the upcoming Annual Conference on Bioassay, Analytical and Environmental Radiochemistry. Continued growth in the number of participating laboratories is anticipated from the commercial national and international communities. (K. G.W. Inn, C. McMahon, Z. Y. Wu, Z. Lin)

***International Equivalence.*** As part of NIST's efforts in the international arena of measurement activities, the Radioactivity Group has prepared a listing of calibration capabilities for Appendix C (list of calibration and measurement capabilities, or CMC's) of the Mutual Recognition Arrangement and submitted it to our RMO. The final version of the Group's CMC's had nearly 400 entries covering a range of dozens of nuclides, geometries and measurement techniques and will be part of a much larger database which will include the CMC's from NMI's around the world. In addition, the gamma emitting  $^{177}\text{Lu}$  was submitted to the Système International de Référence (SIR). (L.R. Karam, L.L. Lucas, B.E. Zimmerman)

**INVITED TALKS****1999**

Bennett, Douglas, Wu, Zhongyu, Inn, Kenneth G.W., "A Comparison of Efficiency Measurements Made Using a HPGe Detector with Calculations from Monte Carlo Simulations," 45th Annual Conference on Bioassay, Analytical, and Environmental Radiochemistry, Gaithersburg, MD, (October 1999).

Inn, Kenneth G.W., "NIST Radionuclide Traceability: How Good Is Good Enough?," Carlsbad Environmental Monitoring and Research Center, Carlsbad, NM, (August, 1999).

Inn, Kenneth G.W., "Acceptance Criteria for Measurement Traceability," 12 International Conference on Radionuclide Metrology and Its Applications, Prague, Czech Republic, (June, 1999).

Inn, Kenneth G.W., "Radionuclide Traceability to NIST," Annual Health Physics Conference, Philadelphia, PA, (June, 1999).

Inn, Kenneth G.W., "Reference Method for Radionuclide Partitioning in Soils and Sediments," Workshop on Speciation of Radionuclides, Edinburgh, Scotland, (September, 1999).

Lin, Zhichao, Inn, Kenneth G.W., Wu, Zhongyu, "A Bone Ash Standard Reference Material for Low-level Actinides and Fission Radionuclides," Annual Health Physics Conference, Philadelphia, PA, (June, 1999).

Lin, Zhichao, Inn, Kenneth G.W. and Wu, Zhongyu, "An Accurate, Precise, and Rapid Method for Analysis of Low-level Sr-90 in the NIST Ashed Bone Standard Reference Material," 44th Annual Conference on Bioassay, Analytical, and Environmental Radiochemistry, Albuquerque, New Mexico, (November, 1998).

Wilcox, Eva and Inn, Kenneth G.W., "Optimization of Sonoluminescence Light Intensity," 45th Annual Conference on Bioassay, Analytical, and Environmental Radiochemistry, Gaithersburg, MD, (October 1999).

Wu, Zhongyu, Inn, Kenneth G. W., Lin, Zhichao and McMahon, Ciara, "PE Sample Verification Process for NRIP Program," presented on 45th Annual Conference on Bioassay, Analytical, and Environmental Radiochemistry, Gaithersburg, Maryland, (October, 1999).

Wu, Zhongyu, Inn, Kenneth G. W., Lin, Zhichao and McMahon, Ciara, "PE Sample Verification Process for NRIP Program," presented on Council on Ionizing Radiation Measurements and Standards, Gaithersburg, Maryland, (October, 1999).

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“Preparation and Verification of NIST Synthetic Fecal Standard for Analysis of Am-241, Pu-238, U-238, and Sr-90,” 44th Annual Conference on Bioassay, Analytical, and Environmental Radiochemistry, Albuquerque, New Mexico, (November, 1998).

Zimmerman, Brian E., “Radioassays of  $^{18}\text{F}$ FDG and Experimental Evaluation of Dose Calibrator Settings” (with G. J. Kubicek, J. T. Cessna, P. S. Plascjak, and W. C. Eckelman), poster presented at the 9<sup>th</sup> European Symposium on Radiopharmaceuticals and Radiopharmacy, Lillehammer, Norway (March, 1999).

Zimmerman, Brian E., “Current and Future Trends in Standards Development for Radionuclides of Use in Nuclear Medicine” (with B. M. Coursey, L. R. Karam, and D. B. Golas), given at the 9<sup>th</sup> European Symposium on Radiopharmaceuticals and Radiopharmacy, Lillehammer, Norway (March, 1999).

Zimmerman, Brian E., “Experimental Determinations of Commercial Dose Calibrator Settings for Nuclides Used in Nuclear Medicine” (with J. T. Cessna), given at the 12<sup>th</sup> Meeting of the International Committee on Radionuclide Metrology, Prague, Czech Republic (June, 1999).

Zimmerman, Brian E., “Development of a Radioactivity Measurement Standard for the Potential Radiotherapy Radionuclide  $^{177}\text{Lu}$ ” (with M. P. Unterweger and J. W. Brodack), poster presented at the 13<sup>th</sup> International Symposium on Radiopharmaceutical Chemistry, St. Louis, Mo (June, 1999).

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Cessna, J., “Destructive Assay of  $^{32}\text{P}$  Stents for Contained Activity,” Cardiovascular Radiation Therapy IV, Washington, DC (February, 2000).

Cessna, J., (with Karam, L.R. and Coursey, B.M.) “Experimentally-Determined Dose Calibrator Settings for an Yttrium-90 Radioimmunotherapy Agent,” American Chemical Society Spring Meeting, San Francisco, CA (March, 2000).

Cessna, J., “Measurement of Contained Activity in a Phosphorus-32 Stent by Destructive Assay,” European Association of Nuclear Medicine Congress, Paris, France (September, 2000).

Collé, R., “Activity Standardizations of Pure-Beta-Emitting Endovascular Brachytherapy Sources by Liquid-Scintillation-Spectrometry-Based Destruction Radionuclide Assays,” Henri Becquerel National Laboratory, Saclay, France (September, 2000).

Inn, K.G.W. (with McCurdy, D., Kuruvilla, L., Barss, N.M., Pietrzak, P., Kaplan, E., Inkret, W., Efurud, W., Rokop, D., Lewis, D., Gautier, P., Thomas Bell, R., III), “Intercomparison Study of Inductively Coupled Plasma Mass Spectrometry, Thermal Ionization Mass Spectrometry and Fission Track Analysis of  $\mu\text{Bq}$  Quantities of  $^{239}\text{Pu}$

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Karam, L.R., “NIST and NAMP: 2000 and Beyond,” NAMP 2K Annual Meeting, Rockville, MD (June, 2000).

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Pibida, L., “Laser Mass Spectrometry as a Sensitive Tool for Analysis of Rare Atomic Traces,” George Mason University, School of Computational Sciences, Colloquium of the Laboratory for Computer Design of Materials, George Mason University, Fairfax, VA (September, 2000).

Wu, Z. (with Inn, K.G.W., Lin, Z., and McMahan, C.) “Traceability of Performance Evaluation Materials,” presented on Methods and Applications of Radioanalytical Chemistry-MARC V, Kailua-Kona, Hawaii (April, 2000).

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