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International Committee for Radionuclide Metrology
EC - JRC Institute for Reference Materials and Measurements

LABORATORY National Institute of Standards and Technology

NAMES R. Collé and B.E. Zimmerman

ACTIVITY Liquid Scintillation Cocktail Composition and Mismatch Effects

IN

PROGRESS We have initiated a very broad, systematic evaluation of liquid scintillation (LS) cocktail composition effects in the assay and standardization of low-energy β^- -emitters and low Z (atomic number) nuclides that decay by electron capture (EC). Progress to date has not only identified a co-dependent relationship between the aqueous fraction in a cocktail and the ionic content loading, but has also resulted in a new scheme for classifying cocktail compositions -- namely the important distinction between "cocktail stability" and what we have termed "cocktail tractability".

PUBLICATIONS B.E. Zimmerman and R. Collé, Cocktail Volume Effects in $4\pi\beta$ Liquid Scintillation Spectrometry with ^3H -Standard Efficiency Tracing for Low-Energy β^- -Emitting Radionuclides, *Appl. Radiat. Isot.*, in press (1996).

R. Collé, Systematic Effects of Total Cocktail Mass (Volume) and H_2O Fraction on $4\pi\beta$ Liquid Scintillation Spectrometry of ^3H , *Appl. Radiat. Isot.*, in press (1996).

R. Collé, Cocktail Mismatch Effects in $4\pi\beta$ Liquid Scintillation Spectrometry: Implications Based on the Systematics of ^3H Detection Efficiency and Quench Indicating Parameter Variations with Total Cocktail Mass (Volume) and H_2O Fraction, *Appl. Radiat. Isot.*, in press (1996).

IN PREPARATION a series of three papers describing the effects of aqueous mass fraction and ionic content on the cocktail stability and cocktail tractability of ^{63}Ni , ^{36}Cl and ^3H cocktails

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ACTIVITY International Intercomparisons of ^{63}Ni and ^{55}Fe

RESULTS The NIST Radioactivity Group recently participated in an international measurement intercomparison for ^{63}Ni and ^{55}Fe , which was conducted amongst principal national radionuclidic metrology laboratories. The intercomparison was sponsored by EUROMET and organized by LPRI, and was primarily intended to evaluate the capabilities of liquid scintillation spectrometry techniques for assays of nuclides that decay by low-energy β^- emission (like ^{63}Ni) and by low-Z (atomic number) electron capture (like ^{55}Fe). Preliminary results from this intercomparison reveal an excellent agreement for ^{63}Ni between the NIST finding and those from other participating laboratories. The results for ^{55}Fe suggest that we need to conduct rigorous, systematic evaluations of our LS capabilities in assaying radionuclides that decay by low-Z electron capture.

IN PREPARATION R. Collé and B.E. Zimmerman, A Compendium on the NIST Radionuclidic Assays of the Massic Activity of ^{63}Ni and ^{55}Fe Solutions Used for an International Intercomparison of Liquid Scintillation Spectrometry Techniques (*J. Res. Natl. Inst. Stds. Tech.*)

OTHER RELATED PUBLICATIONS

R. Collé and B.E. Zimmerman, ^{63}Ni Half-Life: A New Experimental Determination and Critical Review, *Appl. Radiat. Isot.* **47**, 677-691 (1996).

R. Collé and B.E. Zimmerman, Nickel-63 Standardization: 1968-1995, *Radioact. Radiochem.* **7**, no.2, 12-27 (1996).

B.E. Zimmerman and R. Collé, Standardization of ^{63}Ni by $4\pi\beta$ Liquid Scintillation Spectrometry with ^3H -Standard Efficiency Tracing, *J. Res. Natl. Inst. Stds. Tech.*, in press (1997).

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LABORATORY	National Institute of Standards and Technology
NAMES	R. Collé
ACTIVITY	Performance efficacy and long-term stability of the NIST radon-in-water standard generator
RESULTS	<p>A ^{226}Ra-^{222}Rn generator that could be used as a transfer standard for radon-in-water measurement calibrations was previously developed and described. The generator utilized a novel ^{222}Rn emanation source that was comprised of a ^{226}Ra solution encapsulated in polyethylene. The long-term performance of this standard generator has now been investigated and evaluated. The evaluation included exhaustive and more reliable measurement uncertainty analyses for the generator's performance and routine operation. Modifications to the original protocol for operation of the generator has also resulted in improved precision in the ^{222}Rn activity concentration in a dispensed aliquant. The evaluation results indicate that the generator performance has remained stable, and that the calibration parameters are still well within their given uncertainty intervals for the originally determined canonical values. Over a period of approximately six years, experimentally determined values of the ^{222}Rn emanation fraction, the most critical parameter, have remained constant and invariant of conditions within statistical variations of about 0.3 percent (corresponding to a relative standard deviation of the mean). All evaluation measurements were performed by 4π-$\alpha\beta$ liquid scintillation spectrometry of gravimetrically-determined aliquants dispensed from the generator.</p>
PUBLICATION	R. Collé and Raj Kishore, An Update on the NIST Radon-In-Water Standard Generator: Its Performance Efficacy and Long-Term Stability, <i>Nucl. Instr. Meth. Phys. Res.</i> , in press (1997).
OTHER RELATED PUBLICATIONS	<p>J.M.R. Hutchinson, P.A. Mullen and R. Collé, The NBS Radon-In-Water Standard Generator <i>Nucl. Instr. Meth. Phys. Res.</i> A247, 385-389 (1986).</p> <p>J.M.R. Hutchinson, P.A. Mullen and R. Collé, Development of a Regenerative Radon-In-Water Radioactivity Standard, <i>Nucl. Instr. Meth. Phys. Res.</i> 223, 451-457 (1984).</p>
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ACTIVITY Final Results for the International Intercomparison of Marine-Atmospheric Radon Measurements

RESULTS In 1991-1992, NIST conducted an *in situ* calibration and intercomparison exercise for marine atmospheric radon measurements. The participating laboratories have been responsible for perhaps 95 percent of the available surface-level measurements gathered around the globe over the last decade. The results of this intercomparison exercise have been fully published in a series of articles. The intercomparison utilized a common standardized, *in situ*, reference basis (provided by NIST) that could be directly related to U.S. national, and internationally recognized, ^{226}Ra and ^{222}Rn standards, and evaluated the performance of all principal instruments that are used to measure radon activity concentrations for marine-atmospheric studies. The findings will assist various users in the global modelling community in applying the available and future radon measurement data bases in a more reliable and effective manner. The work went beyond serving the needs of just this particular intercomparison. It also demonstrated the broader utility of the developed procedures, i.e., the calibration protocol and the methodology for providing *in situ* standardized samples. Most environmental measurement intercomparisons of field instruments in actual use merely rely on evaluating the relative performance of the participants, or some comparison to the pooled results. This exercise demonstrated, for the very first time, the capability of providing a standardized reference basis even for such low-level, field-measurement intercomparisons. The developed methodologies could be adopted with slight modifications to cover other radon concentration ranges and other applications, and could be employed in many other types of radon environmental field-measurement intercomparisons.

PUBLICATIONS R. Collé, M.P. Unterweger, P.A. Hodge, J.M.R. Hutchinson, S. Whittlestone, G. Polian, B. Ardouin, J.G. Kay, J.P. Friend, B.W. Blomquist, W. Nadler, T.T. Dang, R.J. Larsen and A.R. Hutter, An international intercomparison of marine atmospheric radon-222 measurements in Bermuda, *J. Geophys. Res.* **100**, 16617-16638 (1995).

R. Collé, M.P. Unterweger, P.A. Hodge and J.M.R. Hutchinson, An International Marine-Atmospheric ^{222}Rn Measurement Intercomparison in Bermuda, Part I: NIST Calibration and Methodology for Standardized Sample Additions, *J. Res. Natl. Inst. Stds Tech.* **101**, 1-19 (1996).

R. Collé, M.P. Unterweger, J.M.R. Hutchinson, S. Whittlestone, G. Polian, B. Ardouin, J.G. Kay, J.P. Friend, B.W. Blomquist, W. Nadler, T.T. Dang, R.J. Larsen and A.R. Hutter, An International Marine-Atmospheric ^{222}Rn Measurement Intercomparison in Bermuda, Part II: Results for the Participating Laboratories, *J. Res. Natl. Inst. Stds Tech.* **101**, 21-46 (1996).

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ACTIVITY LS Spectrometry Intercomparison of NIST and LPRI Tritiated Water Standards

RESULTS Radioactivity standards of tritiated water ($^3\text{H}_2\text{O}$) -- disseminated by the Laboratoire Primaire des Rayonnements Ionisants (LPRI) and the National Institute of Standards and Technology (NIST), the national radionuclidic metrology and standardization laboratories of France and U.S.A., respectively -- have been intercompared by liquid scintillation (LS) spectrometry. The ratio of the certified massic activities for the two standards was compared to that obtained from direct measurements on matched sets of LS cocktails prepared from the standards. Seven experimental trials (involving a total of 21 counting sources for each standard) were performed for the comparison. The trials were performed under a wide range of experimental conditions, including use of two different LS spectrometers and three series of LS cocktail compositions (with systematically varied ^3H detection efficiencies). The results exhibited an apparent mean disagreement between standards of less than 0.4 % on a relative basis. For contrast, the relative combined standard uncertainty on the massic activity ratio for the two standards, as obtained from their respective certified uncertainty assessments, is about 0.7 %.

PUBLICATION B.E. Zimmerman and R. Collé, Comparison of the French and U.S. National ^3H (Tritiated H_2O) Standards by $4\pi\beta$ Liquid Scintillation Spectrometry, *Appl. Radiat. Isot.*, in press (1997).

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NAMES	R. Collé and F.J. Schima
ACTIVITY	Development of a radionuclidic spiking protocol for a mock "soil" γ -ray calibration standard.
RESULTS	<p>A generalized protocol for radionuclidic spiking of solid, granular matrices (e.g., soils and sands for gamma-ray spectrometry calibration sources in Marinelli-beaker configurations) has been devised and developed. The protocol has been found to be very quantitative (in terms of the retention of chemically stable spiking radionuclides and the recovery of the spiked matrix), verifiable (in terms of, for example, demonstrating source homogeneity or accounting for possible spike losses and efficacious (in terms of its suitability for producing spiked sources and ability to be easily reproduced). For this work, a spiked, mixed-radionuclide, mock "soil" calibration standard (in a 1-L Marinelli beaker configuration) was prepared and calibrated by high-resolution photonic-emission spectrometry. The matrix consisted of a well-characterized (in terms of densities, particle sizes, etc) and blended Ottawa sand. The matrix was spiked with ^{241}Am, ^{109}Cd, ^{57}Co, ^{139}Ce, ^{203}Hg, ^{113}Sn, ^{85}Sr, ^{137}Cs, ^{88}Y, and ^{60}Co. Source homogeneity and possible spike losses were evaluated for every spiking radionuclide. Measurements on aliquants (30 g out of 1850 g of spiked matrix) taken as a function of blending time (40 min to 400 min) indicated source homogeneity to within several percent. The only observed spike loss was for ^{203}Hg which is due to the chemical instability and volatility of deposited mercury compounds.</p>
PUBLICATION	R. Collé and F.J. Schima, A Quantitative, Verifiable, and Efficacious Protocol for Spiking Solid, Granular Matrices with Radionuclidic Solutions, <i>Radioact. Radiochem.</i> 7, no. 3, 32-48 (1996).
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ACTIVITY Calibrations for Tropospheric Radon Measurements

IN

PROGRESS As part of a collaboration with the Atmospheric Sciences Research Center (ASRC) of the State University of New York at Albany (Western Field Office, Moffett Field, CA), efforts are underway to calibrate and evaluate the performance (stability, linearity, transfer and detection efficiencies, etc.) of airborne instrumentation used to measure radon (^{222}Rn) concentrations in the troposphere. Atmospheric radon measurements, both at surface levels and at varying altitudes, are widely used by the metrology and atmospheric sciences communities in applications ranging from its use as an indicator of the presence of continental air masses over the oceans to its use as a tracer for identifying air of recent tropospheric origin in the lower stratosphere. It has also become a major tool in the development and verification of global circulation and chemical transport models. These are the complex models used to assess questions such as ozone depletion and the possible effect of supersonic and subsonic aircraft on the chemical state of the atmosphere. Recently, modelling groups from all over the world have begun to examine some of the significant discrepancies in the various models, and have identified the need for reliable radon measurement results. The present collaboration with ASRC will assure and enhance the quality of a significant database required by the modelers -- namely free tropospheric profiles as a function of altitude. The on-going ASRC tropospheric measurements will represent an order-of-magnitude increase in the size of the database previously available in this region, and the NIST-traceable calibration will undoubtedly improve their quality by nearly as much. Major breakthroughs in evaluating the transport component of the models are anticipated as a result of significant improvements in the quality of this radon measurement database.

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ACTIVITY Chemical Digestion and Radionuclidic Assay of Encapsulated ^{32}P Interstitial Brachytherapy Sources

IN

PROGRESS Efforts are underway to relate the ^{32}P activity content of one type of interstitial brachytherapy source to measurements of the source's absorbed dose spatial distribution. The sources (of proprietary design) are thin clad cylinders (having a diameter of less than 0.5 mm and lengths up to a few cm), called "seeds", that are used to treat the wall of blood vessels during angioplasty to prevent restenosis (reclosing due to scar tissue formation in the healing process). For this work, a radiochemical procedure was developed to chemically digest the seed's encapsulating jacket for the release of ^{32}P into a solution which could subsequently be assayed for ^{32}P (and ^{33}P impurity) by $4\pi\beta$ liquid scintillation (LS) spectrometry. The procedure had to be devised to ensure both a quantitative release of the ^{32}P from the seed and the quantitative retention of the ^{32}P in the solution. The radiochemical assays will also allow us to develop calibration factors for non-destructive measurements of the seed's external radiations (e.g., with combination NaI(Tl) and re-entrant ionization chamber "dose calibrators").

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