QUALITY ASSURANCE IN MEASUREMENT OF RADON IN WATER BY LIQUID SCINTILLATION COUNTING

Uttam Saha, Public Service Associate & Program Coordinator, Agricultural and Environmental Services Laboratories, The University of Georgia, Athens, GA
Leticia Sonon, Senior Public Service Associate & Director, Agricultural and Environmental Services Laboratories, The University of Georgia, Athens, GA
Pamela R. Turner, Associate Professor and Extension Housing Specialist, Department of Financial Planning, Housing and Consumer Economics, The University of Georgia, Athens, GA
Dana Lynch, Public Service Associate and Family & Consumer Science Extension Agent, Monroe County, The University of Georgia, Forsyth, GA
Gabrielle Dean, Former Radon Educator, Department of Financial Planning, Housing and Consumer Economics, The University of Georgia, Athens, GA
sahau@uga.edu

Abstract

Our papers in 2016 and 2017 Symposia discussed optimum sampling and analysis methods for radon in water**.** This paper discusses quality assurance. The counting efficiencies of multiple liquid radium standards purchased from a commercial manufacturer produced inconsistent and unacceptable counting efficiencies; thus, their use in the analysis appeared questionable. Duplicate analysis of radon in 220 well water samples mostly yielded relative percentage deviation (RPD) ≤15 and seldom >15. However, >15 RPD in duplicate analysis was associated with the presence of an air bubble in one of the duplicate samples. Repeated analyses of two radon proficiency-test samples, regenerated at 40- to 60-day intervals over a period of two years, consistently yielded acceptable precision (based on the duplicate analyses) and accuracy (closeness to the theoretical radon concentration). Thus, a proficiency testing for radon in water is a valid and valuable option, and it should be part of programs that analyze radon in water.

Introduction

Carcinogenic effects of ingested radon via radiotoxicity is well established (Wrenn et al., 1985). Once ingested with water, radon gas diffuses into the stomach wall and irradiates the stomach wall tissues and can cause stomach cancer (Hopke et al., 2000). Inhaled radon from indoor air is known to cause lung cancer (Darby et al., 2005). Radon in household water supply poses both inhalation and ingestion risks. Most risk from radon in water comes from radon released into the air when water is used for showering, laundering, and other household purposes. According to United States Environmental Protection Agency (USEPA) (2012), the risk of lung cancer from inhaled radon from air is much larger than the risk of stomach cancer from ingesting water containing radon in it. As a rough rule of thumb, household water containing 10,000 pCi/L of radon has a potential toenrich indoor air radon concentration by about 1 pCi/L. Based on a National Academy of Sciences report (NAS, 1999) on radon in drinking water, USEPA estimates that radon in drinking water from public water supplies (PWS) causes about 168 cancer deaths per year nationwide, 89% from lung cancer caused by breathing in radon released from water, and 11% percent from stomach cancer caused by ingesting drinking radon-containing water. Public health consequences of radon in private household water wells are yet to be estimated. However, it may be more severe than the radon from PWS because private household well water systems have less opportunities to lose radon than PWS.

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Several investigations reported the presence radionuclides from three naturally occurring decay series (headed by ^{238}U, ^{232}Th, and ^{235}U) in some ground water and surface water in Georgia (Cline et al., 1983; Hess et al., 1985; Zapecza and Szabo, 1988; Coker and Olive, 1989). Albertson (2003) reported activities of gross alpha, ^{226}radium, and combined ^{226}radium plus ^{228}radium exceeding the Environmental Protection Agency's (USEPA) corresponding drinking water Maximum Contaminant Levels (MCL) in community water systems in the Piedmont, Blue Ridge, and parts of the Coastal Plain physiographic provinces of Georgia. Elevated uranium concentrations were also detected in drinking water in the Piedmont and Blue Ridge physiographic provinces (Albertson, 2003). Coker and Olive (1989) tested 90 wells in Georgia for radon and other radionuclides and concluded that groundwater from the granite and gneiss aquifers in the Piedmont contained the highest average concentrations of naturally occurring radionuclides. Stone et al. (2002) found elevated levels of radium in drinking water in the "piedmont and coastal plain sandhills" and elevated uranium in water in the "piedmont (and Blue Ridge) region" of South Carolina. Thus, radioactivity levels in household waters is a potential public health problem at least in the Piedmont-Blue Ridge regions in the southeastern United States, which merits monitoring, public education, and mitigation. The cooperative extension systems of the land-grant universities can play a major role in this regard.

In 2015, the Agricultural and Environmental Services Laboratories and The College of Family and Consumer Sciences of the University of Georgia launched a new Radon in Household Water Testing and Education program. In the papers published in the proceedings of 2016 and 2017 International Radon Symposia, we reported original research findings comparing different sampling methods (Direct Fill vs. Submerged Bottle), sample preparations (Simultaneous Drawing vs. Separate Drawing), scintillators (Mineral Oil vs. OPTI-FLUOR), and liquid scintillation counting assays (0-2000 keV vs. 130-700 keV) for analyzing radon in water four "Household Well Water" samples and two "Proficiency Test (PT)" samples (Saha et al. 2016; 2017). The "130-700 keV" assay had significantly higher radon recovery than the "0-2000 keV" assay. The Direct Fill sampling produced significantly lower results than the "Submerged Bottle" sampling. "Simultaneous Drawing" of both scintillator and water sample yielded higher radon recovery than "Separate Drawing". "Mineral Oil" scintillator provided higher radon activity than "OPTI-FLUOR". However, in eight consecutive measurements of the PT samples at 60 days (full ingrowth) interval, "Mineral Oil" always overestimated the radon activity compared to the predicted/ assigned value, whereas "OPTI-FLUOR" invariably produced results close to the predicted/assigned value (Saha et al. 2016; 2017).

We know the fact that all analytical methods should embrace a well-defined quality-assurance/quality-control (QA/QC) milestones. Because of its gaseous state, developing an appropriate practical QA/QC protocol for testing radon in water needs special considerations. Such protocols should allow the evaluation of both precision and accuracy. However, there is no dependable recommendations in this regard for testing radon in water. In this study, we evaluated precision of measuring radon in the duplicate water samples from private well waters and accuracy of multiple liquid radium standards purchased from a commercial manufacturer and two reusable radon proficiency-test samples by repeated measurement over a period of two years.

Methodology

Liquid Scintillation Counting (LSC) Assay

When ^{222}Rn goes through radioactive decay, it can produce the progenies ^{218}Po, ^{214}Po, ^{214}Pb and ^{214}Bi, all of which are relatively short-lived. A secular equilibrium of ^{222}Rn with these four progenies is reached after approximately 3.5 hours when all five of the radionuclides exist at the same level of activity. Radioactive decay of these five radionuclides release alpha and beta particles each of a known kinetic energy. When they are placed in liquid scintillation cocktail, scintillators and solvent of the cocktail convert this kinetic energy into light photons that are amplified by a photomultiplier tube (PMT) and are detected as PMT pulse with an amplitude or pulse height proportional to the energy of the decay particle that induced the response. The number of pulses, which is commonly known as "counts", induced at the PMT is proportional to the amount of radioactivity interacting in the cocktail.

An analog-to-digital converter transforms the analog PMT pulse to a digital value and assigns it to a channel in a multichannel analyzer (MCA). Alpha particles generated by radioactive decay have kinetic energies between 4 and 8 MeV in most cases. But, much of the energy emitted by alpha particles is left out from conversion to scintillation light photons. Because of such low scintillation light output, a 6.0 MeV alpha particle (for example) produces PMT pulses equivalent to those produced by a 600 keV beta particle. As such, all alpha particles appear in an LSC energy range of approximately 200 to 800 keV, the same energy range over which many beta particles are detected (Packard 1992; Kessler 1989).

Conventionally, during LSC the alpha and beta induced PMT pulses are collected in a single MCA. Because of the lower alpha scintillation yield, the alpha and beta spectra overlap in the MCA and cannot be effectively separated. Figure (1) shows a typical combined alpha-beta particle spectrum for a radon sample analyzed on a liquid scintillation analyzer. The two large peaks correspond to alpha particles. The right peak is from the 7.6 MeV alpha particle of ^{214}Po; the left is due to both the 5.0 and 6.0 MeV alpha particles emitted from ^{222}Rn and ^{218}Po, respectively. The broad, low height peak is the beta spectrum of ^{214}Pb and ^{214}Bi.

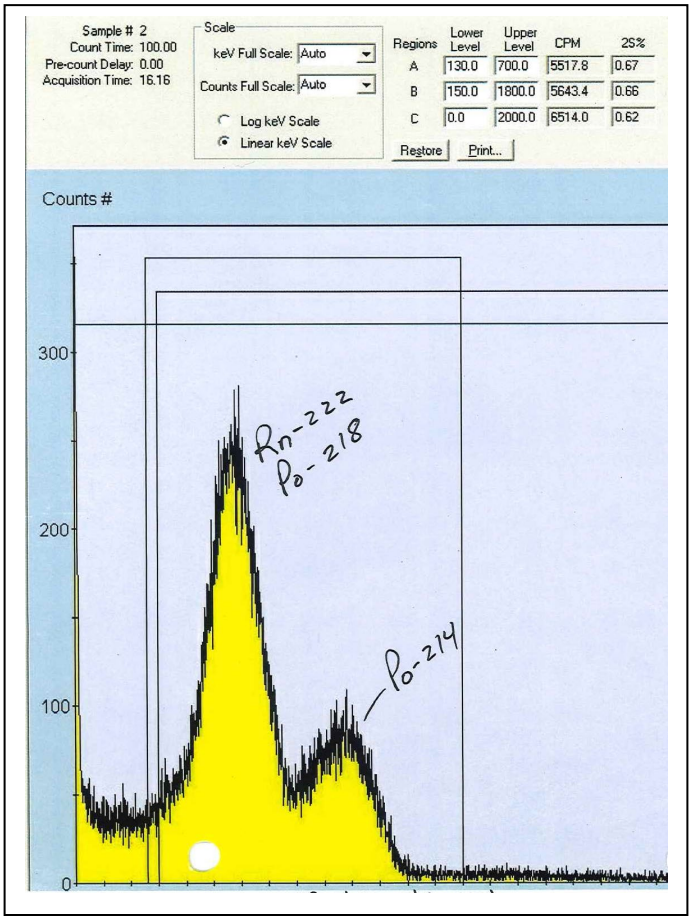

Figure (1): A common LSC spectrum for ^{222}Rn and progenies. The right peak is due to ^{214}Po alpha particle with 7.6 MeV while the larger peak is due to the alpha particles from combined ^{222}Rn (5.5 MeV) and ^{218}Po (6.0 MeV). The remainder of the signal is the beta particle spectrum from ^{214}Pb and ^{214}Bi.

Thus, in this study Liquid scintillation counting was done using a Tricarb 2910 Liquid Scintillation Counter (PerkinElmer, Waltham, MA) for counting radon in various sample types with the LSC assay presented in Table (1) below.

Table (1): The two different LSC assays compared in this study.

Region	Lower Limit (keV)	Upper Limit (keV)
A	130	700
B	150	1800
C	0	2000

As obvious in Table (1), this assay is limited within the region of interest (ROI) for ^{222}Rn from 130 to 700 keV, excluding the counts below 130 keV (which is indeed from “Bremsstrahlung” radiation). Cutting out the low-energy (below 130 keV) betas also reduces the quenching and background.

Household Water Samples for Evaluating the Precision

Over the three-year period of 2015-18, our laboratory received 220 household well water samples for testing radon through voluntary submission by the homeowners. All these 220 samples were submitted in duplicate. Duplicate analysis results of these 220 samples were used to evaluate the precision based Relative Percentage Difference (RPD) calculated as:

RPD = (|sample result - duplicate result| ×100) ÷ [(sample result + duplicate result) ÷ 2]

We evaluated the precision of radon testing using 15% and 20% RPD thresholds.

Ra-226 Standards from a Commercial Vendors

The ^{222}Rn gas is a progeny produced by the alpha decay of ^{226}Ra. In a ^{226}Ra solution, ^{222}Rn with a 3.83-day half-life, reaches a secular equilibrium with its 1600-year half-life parent (^{226}Ra) in approximately 28 days, where both ^{222}Rn and ^{226}Ra co-exist with same magnitude of activities. As such, a standard ^{226}Ra solution can be used as a ^{222}Rn standard in the analysis of radon in water by LSC.

We evaluated the accuracy of LSC assay of radon using two types of ^{226}Ra standards procured from Eckert & Ziegler Analytics (Atlanta, GA).

The primary ^{226}Ra standard had 15 mL of liquid in a 20 mL flame sealed liquid scintillation vial. The 15 mL of liquid contained 7.5 mL radium chloride in 7.5 mL OPTI-FLUOR (PerkinElmer, Waltham, MA) cocktail with a total activity of 3.932 Bq. According to the vendor, "this standard radionuclide source was prepared gravimetrically from a master solution calibrated by Eckert & Ziegler Analytics, using a germanium gamma spectrometer system. Radionuclide purity and calibration were checked by germanium gamma-ray spectrometry, liquid scintillation counting, and/or alpha spectrometry, as applicable. The half-life was 5.844±0.05 days and reference date was April 24, 2015 according to Eckert & Ziegler Analytics (EZA). Eckert & Ziegler Analytics (EZA) maintains traceability to the National Institute of Standards and Technology through a Measurements Assurance Program as described in USNRC Regulatory Guide 4.15, Revision 2, July 2007, and compliance with ANSI N42.22-1995: Traceability of Radioactive Sources to NIST." The expiration date stamped as April 2018, meaning this standard should have been useful for 3 years after the reference date.

The secondary ^{226}Ra standard was prepared from a NIST traceable 40 mL ^{226}Ra liquid primary standard in a 0.1 M HCl media supplied by the vendor in a 50 mL flame sealed ampule. The recommended density of this solution was 1.0 g/mL. This ^{226}Ra primary standard solution had a total activity of 16.01 Bq. From this primary standard solution, a secondary standard solution was prepared as follows:

An empty, clean 20 mL liquid scintillation vial was placed on a balance and the balance was tared. Then a 7.5 mL aliquot of the primary ^{226}Ra standard solution was gravimetrically transfer into the vial and the weight of the ^{226}Ra solution in the liquid scintillation vial was recorded, this weight was used to determine the actual total activity of the secondary liquid scintillation standard. Then 7.5 mL of OPTI-FLUOR cocktail was added to the liquid scintillation vial containing the ^{226}Ra solution, the vial was sealed tightly, and the content was shaken to mix well. This cocktail was kept away from light to increase the longevity of the secondary liquid scintillation standard. The secondary liquid scintillation standard was shaken vigorously for one minute before every count. This ^{226}Ra secondary standard solution had a total activity of around 3 Bq (depending on the actual weight of the primary ^{226}Ra taken).

The remaining ^{226}Ra primary standard solution was stored in a screw-top glass bottle with the highest quality seal to prevent evaporation. Further protection from evaporation was achieved by wrapping the top of the bottle and cap with parafilm, placed in an amber secondary container, and stored in a light-free

refrigerator.

The counting efficiency of the primary and secondary ^{226}Ra standard was evaluated over a long period of time with an expected efficiency (CPM per DPM) of 2.0.

Proficiency Test Samples

We used two ^{222}Rn in water "proficiency test" or "standard" samples, labelled as "Standard-15" and "Standard-17", obtained from the Laboratory of Inorganic & Nuclear Chemistry, Wadsworth Center, New York State Department of Health, Albany, NY. The "Standard-15" and "Standard-17" are reusable radon-in-water standards as they were prepared using a ^{226}Ra-loaded filter sandwiched in polyethylene sheeting (Kitto et al., 2008). At full ingrowth (>30 days), the ^{222}Rn produced by the sandwiched ^{226}Ra sources in both "Standard-15" and "Standard-17" should be 4375 pCi/L at 100% emanation, but due to retardation by the polyethylene, produces only 3763 pCi/L at 86% emanation. These two standard samples were prepared in both OPTI-FLUOR and mineral oil cocktail with simultaneous and separate drawing of 8 mL and counted by two different LSC assays (Full Sepectrum: 0-2000 keV and ROI: 130-700 keV) after every 40-60 day in-growing over a period of two and half years. The preparation methods of these samples have been described elsewhere (Saha et al., 2016; Saha et al., 2017). The results of these measurements were evaluated using acceptance window of 3763 ± 940 (25%) pCi/L.

Results and Discussion

Precision of Duplicate Measurements

Figure (2) shows the Relative Percentage Difference (RPD) for the duplicate sampling and measurements of 220 household well water samples. The results show that >90% of the duplicate measurements had RPD less than 20% and about 87% had RPD less than 15%. However, the duplicate measurements that yielded RPD greater than 15 were from the cases where one of the two sample bottles had air bubbles in it or both had air bubbles of unequal sizes. The finding depicted in Figure (2) is robust because this is based on a fairly large number of duplicate samples collected by the 220 homeowners and analyzed by 5 analysts (randomly) in our laboratory over a period of three years (2015-18). Thus, our results demonstrate that liquid scintillation analysis of radon in water could be considered precise if RPD of duplicate measurements is less than 15% or 20%. The 15-20% RPD accounts for the errors in sampling and sample preparation. A practically feasible threshold RPD value to evaluate precision of analyzing radon in water is yet to be formalized. Our findings could be useful in making this much-needed recommendation of a threshold RPD value for evaluating precision of analyzing radon in water.

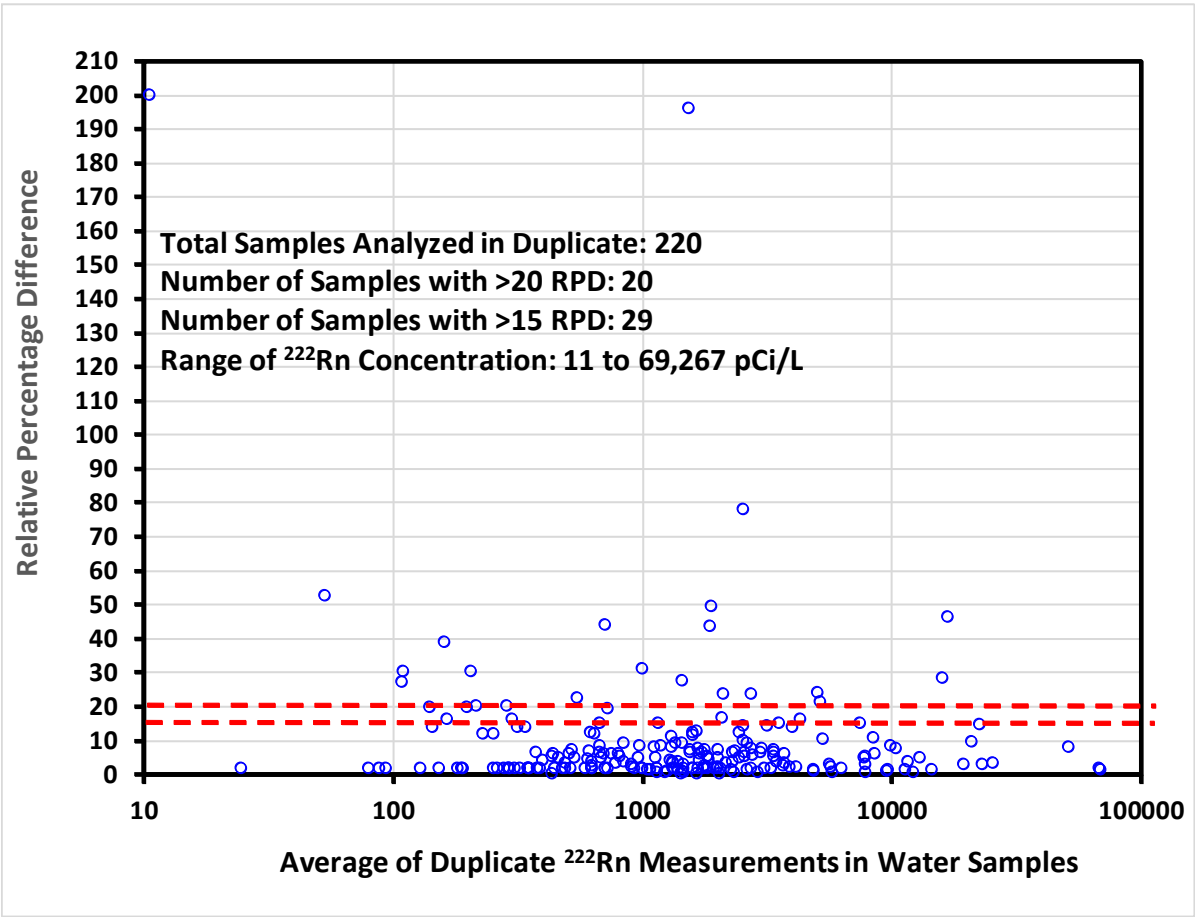

Figure (2): Relative Percentage Difference (RPD) of duplicate sampling and measurement radon carried out on 220 household well water samples.

Note: The samples were collected by the 220 homeowners and analyzed by 5 different analysts (randomly) on different over a period of three years (2015-18).

Efficiency and Longevity of ^{226}Ra Primary Standard (B#45715)

As depicted in Figure (3), the ^{226}Ra primary standard, prepared by mixing 1:1 volume ratio of radium chloride solution:OPTI-FLUOR, initially gave expected counting efficiency (CPM ÷ DPM) of around 2.0 and this satisfactory performance lasted for a while. However, the standard started to give unacceptable counting efficiency from October 20, 2016, which was long before its expiry date of April 2018 given by the vendor. This finding reminds the users and vendors of this kind of ^{226}Ra primary standard regarding the assigned expiration date (by the vendor).

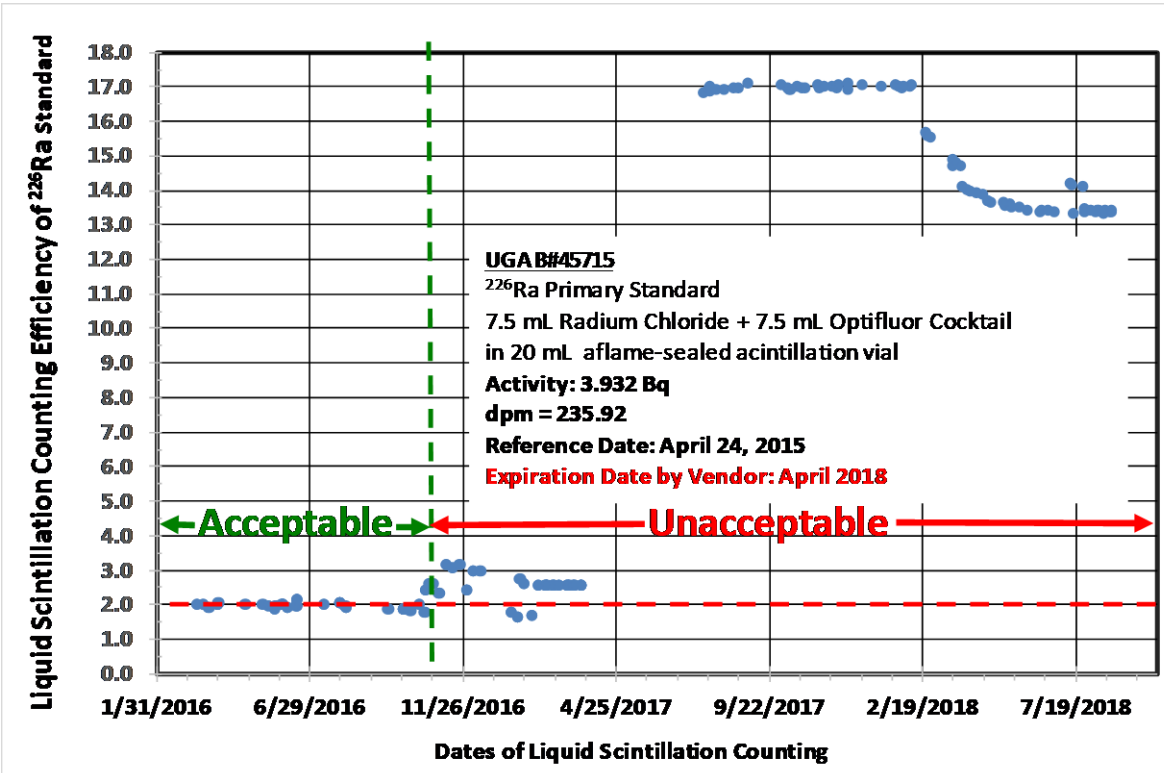

Figure (3): Liquid Scintillation Counting Efficiency of a ^{226}Ra primary standard over time.

Efficiency and Longevity of ^{226}Ra Secondary Standards

After being reported about the deterioration of the primary standard (B#45715) way before its assigned expiry date, the vendor provided another 40 mL ^{226}Ra primary standard in a 50 mL flame sealed ampule. This was indeed a NIST traceable RaCl solution in 0.1 M HCl media with a total activity of 16.01 Bq (SRS# 107682). As suggested by the vendor, we prepared the secondary working standard by gravimetrically transferring 7.5 mL of this primary standard solution to a vial containing 7.5 mL of OPTI-FLUOR and mixing the content by vigorous shaking. It was first prepared on October 16, 2018 and have been on counting it with the routine samples. However, it never gave a satisfactory counting efficiency (Figure (4)). Having such negative observations, we prepared a second secondary standard from the same source in the same way on May 11, 2018 and has been on counting with the routine samples. As depicted in Figure (5), the counting efficiency of this second preparation was random and unacceptable for about two months after preparation. From July 7, 2018, the counting efficiency given by this second preparation was close to the expected value of 2.0. However, a sustained satisfactory counting efficiency

of this standard for a considerable length of time could be a reality or an illusion based on the experience with the standards received from the commercial vendor.

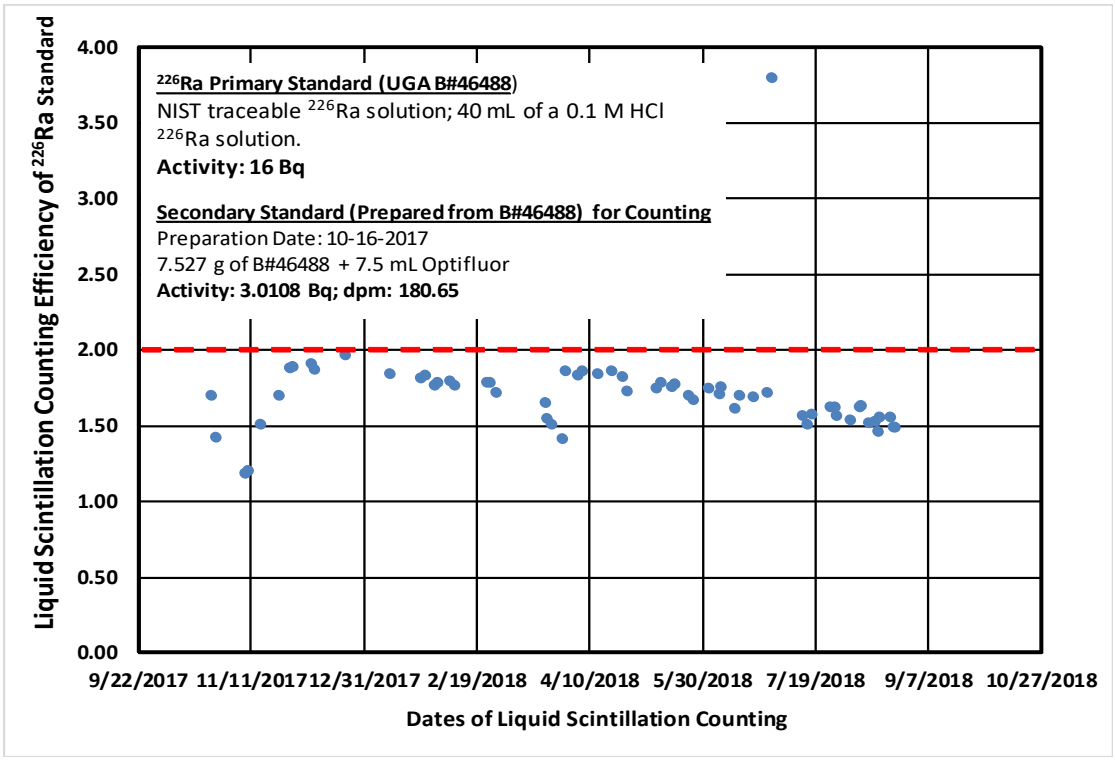

Figure (4): Liquid Scintillation Counting Efficiency of a ^{226}Ra secondary standard-1 over time.

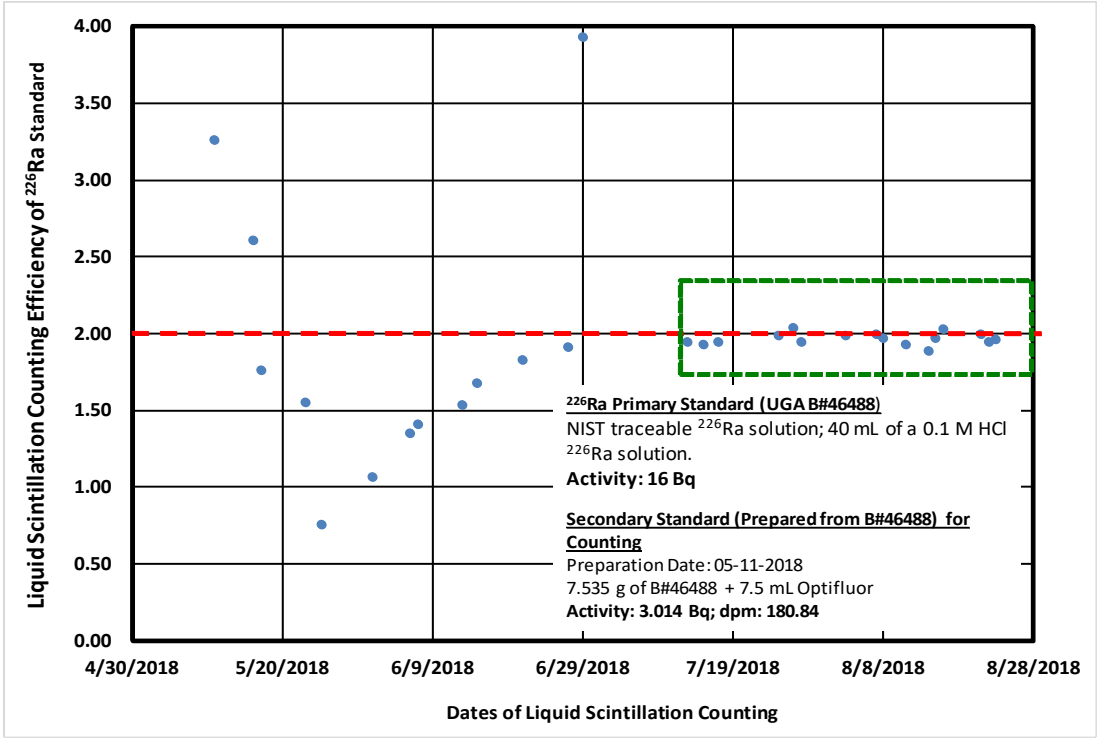

Figure (5): Liquid Scintillation Counting Efficiency of a ^{226}Ra secondary standard-2 over time.

Reusable Proficiency Test Samples

Laboratory of Inorganic & Nuclear Chemistry, Wadsworth Center, New York State Department of Health, Albany, NY provided us two reusable ^{222}Rn in water "proficiency test (PT)" or "standard" samples, labelled as "Standard-15" and "Standard-17". The assigned or known concentration of radon in both "Standard-15" and "Standard-17" is 3763 pCi/L with the lower and upper acceptance limits of 2822 (~75% of the known) and 4704 (~125% of the known) pCi/L, respectively (Kitto et al., 2008). Figure 6 plots the detailed results of the 13 consecutive radon measurements on these two reusable PT standards carried out at around 60-day intervals at the University of Georgia (UGA) laboratory using LSC assays and compare these results with those reported by other laboratories across the United States using various methods. The x-axis represents the various laboratories in the US. The y-axis is the measured radon. The data points corresponding to x-axis values 1-22 were reported by various other laboratories in the nation (Kitto et al., 2008b) and plotted in ascending order. The data points corresponding to x-axis values 23 and higher are the values obtained in the UGA laboratory by 4 different sample processing (mineral oil and OPTI-FLUOR in combination with simultaneous and separate drawings: 2 × 2 = 4). The blue-filled and red-filled circles are the results obtained from the cocktails prepared in mineral oil and analyzed the assay-2 (130 - 700 keV ROI) and assay-1 (0 – 2000 keV ROI), respectively. The blue-open and red-open circles are the results obtained from the cocktails prepared in OPTI-FLUOR and analyzed the assay-2 (130 -
700 keV ROI) and assay-1 (0 - 200 keV ROI), respectively. As depicted in Figure (6), all results generated by the assay-1 (0 - 2000 keV ROI) were lower than the lower limit of acceptance, which means they all failed. Among the results generated by assay-2 (130 - 700 keV), the data points for the samples prepared in "mineral oil" were mostly higher than the upper limit of acceptance, which means they also failed in most cases. In contrast, all results from OPTI-FLUOR are acceptable, and at least half of them were very close to the true value. Therefore, the assay-1 (0 - 2000 kev ROI) with both mineral oil and OPTI-FLUOR can grossly underestimate the actual radon concentration, and with assay-2 (130 - 700 keV ROI), mineral oil can over-estimate the radon concentration; both should be avoided. Thus, our results show that these samples can serve as a reusable PT standard when measured in OPTI-FLUOR with the assay-1 (130 – 700 keV ROI) and this should be a part of nationally coordinated proficiency program and should be adopted by the laboratories testing radon in water.

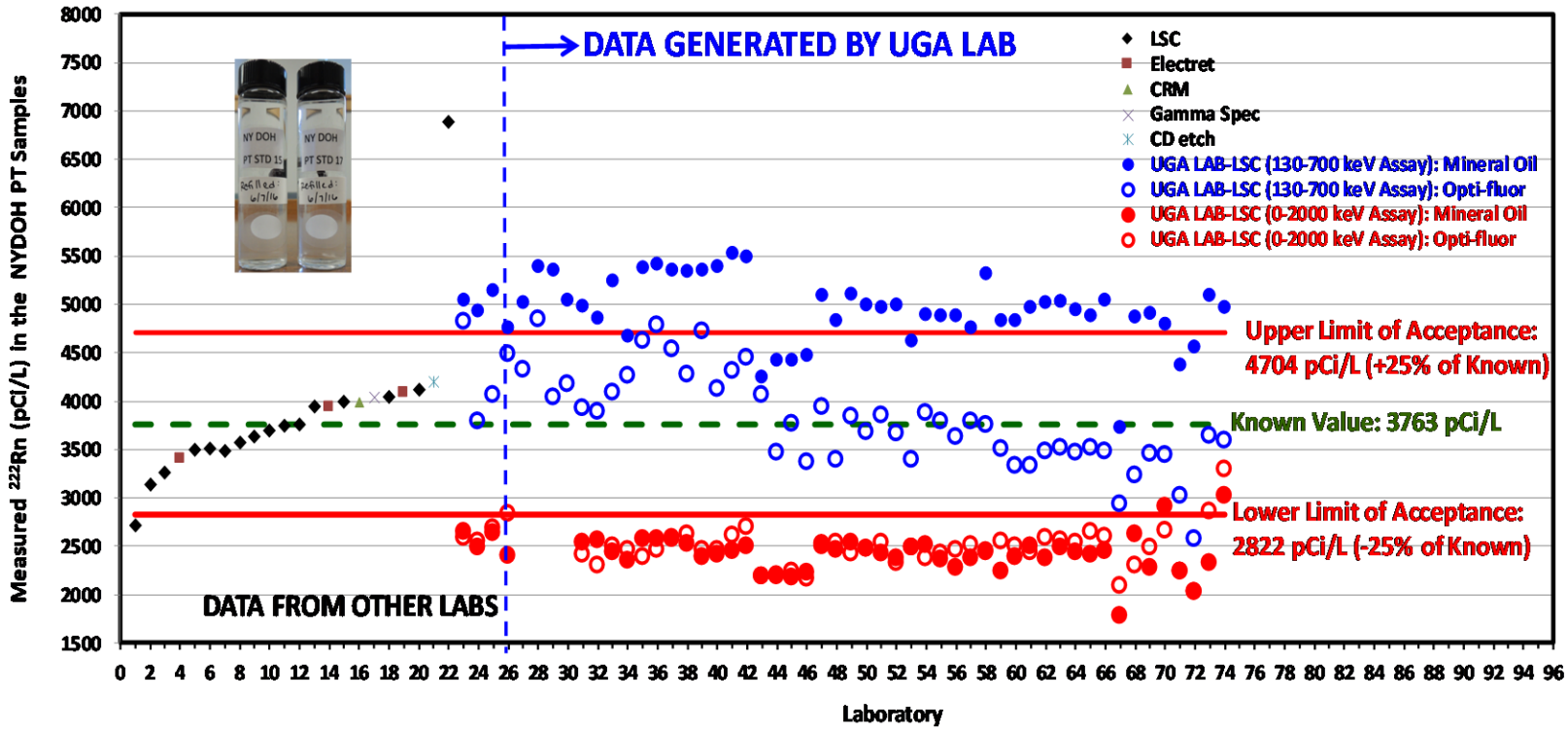

Figure (6). Radon levels in the NYDOH proficiency testing standard samples in 12 consecutive measurements at 40-60-day interval at the University of Georgia (UGA) laboratory to compare:

- Two different LSC assays: 0 - 2000 keV versus 130 - 700 keV.
- Two different scintillation fluids: mineral oil versus opti-fluor.

Note : The UGA laboratory results generated by LSC have been plotted together with the results (obtained from the coauthor M.E. Kitto , New York State Department of Health) from different laboratories across the United States generated by various methods.

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