Measuring Low Radon Levels in Drinking Water Supplies

Jerry D. Lowry

Because a relatively low maximum contaminant level (approximately 300 pCi/L) is expected to be set for radon in the near future, research was conducted with liquid scintillation counting to determine whether this method of analysis could be used for low levels of radon. Counting with an optimized window and an optimized water-to-fluor ratio resulted in achievement of a lowest quantifiable level of 150 pCi radon/L. Lower levels can be quantified using a longer counting period.

Maximum contaminant levels (MCLs) for additional radionuclides are expected to be announced in the fall of 1991 by the US Environmental Protection Agency (USEPA). Radon (Rn) and uranium (U) will join radium (Ra-226 and Ra-228) as regulated radionuclides. The MCL for Rn is expected to be relatively low (approximately 300 pCi/L) compared with existing unofficial criteria and will have a significant impact on public groundwater supplies.[1] Because there is no separate method in *Standard Methods* for analyzing water for Rn, the USEPA's Las Vegas laboratory is currently engaged in evaluating two analytical methods: (1) liquid scintillation counting (LSC) and (2) Lucas cell counting. Earlier work published by the USEPA recommended that these two methods be considered validated and equivalent.[2] That research concluded that the LSC method is slightly more precise and accurate than the Lucas cell method, based on a collaborative assessment involving 18 laboratories. The USEPA is continuing its evaluation of the two methods, with a greater emphasis on LSC for Rn in drinking water.[3] It is clear from that work and the large number of samples that will have to be analyzed that the LSC method is the more practical method. The LSC method is not as labor-intensive and is simple to perform.

Optimization of the LSC method for Rn has received little attention because most interest has been placed in waters having relatively high Rn (>1,000 pCi/L). In fact, until very recently a range of 10,000-40,000 pCi Rn/L in water was used by some states as an unofficial criterion for requiring treatment. For high Rn levels, the uncertainty of counting is low and optimization has not been an issue. The article most often cited by researchers employing LSC for Rn analysis is one by Prichard and Gesell.[4] Kinner et al have investigated some of the details of sampling and analysis.[5] In general, it appears that little effort has been made to optimize the LSC method for low Rn levels. The term optimization in this article means the process of establishing a standard protocol that will result in the minimum counting uncertainty. In summary, there is a need to improve the existing method of analysis for Rn in drinking water supplies. Large numbers of Rn analyses will be dictated by the new regulation, and the current LSC method has not been optimized.

Recent research sponsored by the AWWA Research Foundation has significantly improved the existing LSC method for Rn and has introduced new methods for the analysis of U and Ra. This article summarizes the research involving LSC analysis of Rn.

Laboratory

All of the Rn analyses and experiments were conducted in a combined commercial and research laboratory* that routinely performs Rn and other radionuclide analyses for the water supply industry. More than 5,000 Rn samples have been analyzed by the laboratory, and analysis has progressively approached the optimized Rn protocol described in this article during the past two years (the final protocol was adopted for routine analysis in November 1990).

Experimental methods

Before explaining the methods used in this research, it is useful to provide some definitions of general terms used in the field of LSC. LSC cocktails are the solutions that contain the fluors that provide the link between atoms decaying and the measurement of light energy by a photomultiplier tube. It is common to refer to the LSC cocktail as the fluor. The cocktail solvent and its solutes facilitate a transfer of kinetic energy into emitted photons. In the case of Rn, the fluor is immiscible and is also responsible for the extraction of Rn from the water. Progeny of Rn are not extracted but are produced by the ingrowth process from the extracted Rn. This process takes approximately 4 h, and at that point the LSC fluor contains Rn and its first four progeny, in secular equilibrium with Rn. Secular equilibrium means that Rn and all four progeny are at the same level of activity. All isotopes are counted by alpha or beta decay, and associated gamma radiation is also present.

In general, a typical commercial LSC used for Rn analysis does not represent the alpha energy of decay accurately on the spectral abscissa scale. For example, an alpha decay is detected at an energy approximately one tenth of the absolute energy value. These instruments represent the beta decay energy more closely, are approximately calibrated for 0-2,000 keV, and are commonly referred to as beta-counting instruments. Although the energy scale is not representative of the exact energy unit, meV or keV, the alpha-decaying isotopes in the fluor are counted with near 100 percent efficiency. The resulting spectrum contains counts of both alpha and beta decay, and channel numbers, rather than energy units, are used on the abscissa scale.

A window is a term used to define the region of interest by channel numbers

*RADLAB, Lowry Engineering Inc., Unity, Maine

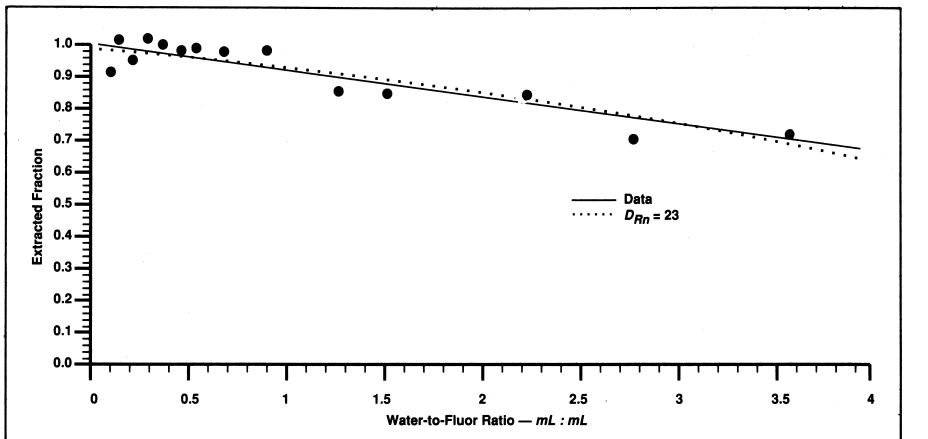

Figure 1. Distribution coefficient data for radon in water and mineral oil fluor

and can range from a one-channel width to a full window, which is 2,000 channels for the specific LSC used in this work. For any given window, there is an associated background count rate that results from a blank vial containing Rn-free water and the fluor. An important characteristic of any counting instrument detecting radioactive decay is the distribution of background counts along the energy scale (abscissa). For instruments that detect alpha, beta, and gamma activity, the distribution is generally weighted toward the low end of the scale, and is particularly high in the first 25 to 50 channels, because of cosmic activity, fallout, the scintillation vial, and terrestrial radiation. In addition to using heavy lead shielding to minimize background radiation, it is common practice to use a discriminator control to exclude the high background channels from the spectral data during the sample and the blank counts. Through discrimination of the counting window, the counting uncertainty can be minimized. Because the distribution of counts from Rn and its progeny along the energy scale is not the same as the background distribution, it is not a simple procedure to determine the optimum counting window. The optimum counting window is not constant as Rn increases from low to high levels; however, it is only at low levels of Rn that the absolute optimum window is important.

The LSC fluor used throughout the research is a high efficiency, mineral-oil-based scintillation cocktail.* Standards for Rn counting were made from dilutions of a USEPA (National Bureau of Standards-traceable) Ra-226 standard solution. Natural groundwater containing high levels of Rn was used in certain experiments for which there was a need for a short counting time and a low counting uncertainty. The LSC vials were standard 23-mL borosilicate glass with either polycone- or aluminum-faced caps. The LSC instrument† was interfaced to a personal computer and line printer. The LSC instrument gave 4,000 half-channel spectral information from its multichannel analyzer.

Although a specific LSC instrument and fluor were used throughout this research, the results are applicable generally. Some minor variations in the optimum window of discrimination and fine details of the protocol will probably occur with different LSC instruments and fluors, but the general concepts and process of optimization will remain unchanged. With any new setup of equipment and fluor, an optimization process, as described in this article, should be conducted.

Uncertainty

A Rn analysis has uncertainty associated with the sampling step, with sample setup in the laboratory, and with counting. An analysis of extensive field and laboratory data indicates that the sampling and laboratory handling steps have an associated combined uncertainty <3.0 percent (2-sigma). Earlier studies[6] reported higher uncertainty for these sources, but hundreds of duplicate public water supply samples analyzed over the past two years have shown that the uncertainty from these sources is low.

For example, the most recent 108 independent duplicate samples (Rn > 500 pCi/L) taken by public water utility personnel across the United States have an average deviation of only 3.17 percent between duplicates compared with their average. These data reflect the total uncertainty associated with the entire process of sampling and analysis, as well as any real change in the Rn level during duplicate sampling. By taking a large number of samples with an Rn level of ≥500 pCi/L, the contribution of random counting uncertainty from the data set is virtually eliminated. Thus, the routine combined field sampling and laboratory sample setup uncertainty is <3.0 percent. It is difficult to discern from these data the actual true deviation of Rn between vials that are sampled successively at a sample tap. It is clear, however, that for certain personnel at specific water utilities, the combined sampling and sample setup uncertainty is routinely <2.0 percent. In summary, deviations between replicate samples of significantly >3.0 percent, which cannot be explained by counting uncertainty, are almost certainly the result of poor sampling, poor setup, or actual temporal variation of the Rn level in the water at the time of sampling. If the samples are true splits, then the error would be from poor sampling or setup.

Ineffectively sealed sample vials have been suspected as a source of Rn loss while the sample is in transit to the laboratory. Because the vials are glass, the cap seal is the only possible route of escape. Silicon rubber septa faced with PTFE are commonly used to seal sample vials. Solid aluminum-lined caps are an alternative. Another alternative is to use direct injection into the LSC fluor vial in the field, without use of an intermediate sample vial for transit to the laboratory.

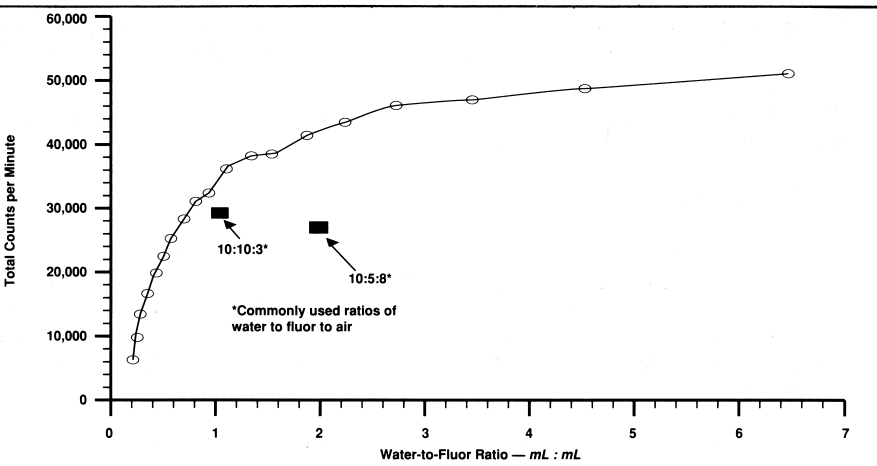

Figure 2. Extraction data for radon with variable water-to-fluor ratios

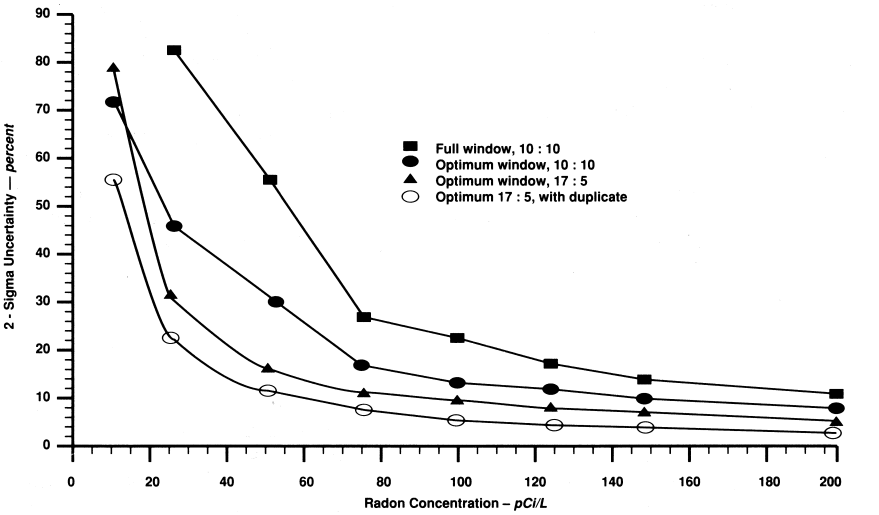

Figure 4. Two-sigma counting uncertainties for four LSC protocols for radon *60 –minute counting period*

*NEF 957A, New England Nuclear/Dupont, Boston, Mass.
†Model 1500, Packard/Canberra, Downers Grove, Ill.

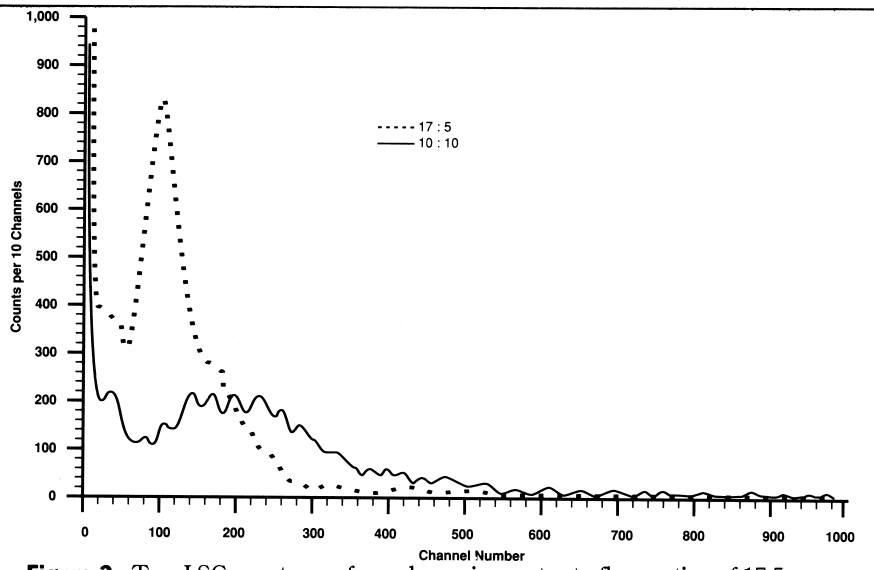

Figure 3. Two LSC spectrums for radon using water-to-fluor ratios of 17:5 and 10:10

TABLE 1

*Results of leakage and setup technique experiment (Rn values in pCi/L)**

Replicate	Direct Syringe	Aluminum-Lined Caps	PTFE-Faced Caps	Incorrectly Placed PTFE Seals
1	400,919	382,830	399,778	149,268
2	390,202	396,652	403,613	190,847
3	404,878	395,870	403,205	202,318
4	406,845	395,585	398,320	194,804
5	405,880	398,214	399,801	193,740
6	406,949	401,246	405,149	184,875
7	403,216	406,149	396,944	179,253
8	404,688	398,077	400,966	191,085
9	399,991	402,074	401,932	183,310
10	403,018	406,513	401,154	166,929
Average	402,658	398,321	401,086	183,643
Standard deviation	4,956.5	6,727.6	2,500.8	15,507.9

*Sampling temperature of water was 10°C; counting temperature was 20°C.

To investigate these alternatives, an experiment was run involving direct injection, aluminum-lined caps, and PTFE-faced septum caps. The Rn source was a natural groundwater containing about 400,000 pCi Rn/L. The high-level Rn source and a 30-min counting time resulted in a counting uncertainty (2-sigma) of about 0.25 percent and minimized that source of uncertainty. Ten replicates for four conditions were used to evaluate the differences between methods of sealing and setup. The fourth group used PTFE-faced silicon rubber septa, with the PTFE incorrectly facing away from the water. This group was included because it is not rare to have this mistake occur in the field sampling step, and past observations of duplicates with and without the incorrectly placed septum indicated significant absorption or leakage with silicon rubber. After collection, all vials were stored at 20°C for four days prior to counting to simulate the average period between sample collection and analysis.

The results of the simple leakage experiment are summarized in Table 1. Although a slight variation was seen in the average values between the direct syringe technique, the PTFE-faced cap, and the aluminum-lined cap, the differences are not statistically significant (95 percent level). However, the incorrectly placed PTFE seal showed a leakage of approximately 54 percent, which was found to be highly significant from a statistical basis. The insignificance of the deviations between the first three groups is even more important when it is considered that all the water came from a single large glass container, open to the atmosphere, and that the entire sampling process took approximately 30 min. The direct syringe vials were sampled first; then the remaining three groups of 10 were sampled randomly. In summary, the results of this experiment clearly demonstrate that the sampling and sample setup steps have a combined uncertainty of <2.0 percent. Finally, it is clear that aluminum is equivalent to PTFE for sealing, and its use would eliminate the possibility of leakage because of an incorrectly placed PTFE-faced septum.

A final consideration should be discussed with regard to possible leakage from all vials; that leakage would go undetected because of the lack of an internal standard or tracer. Through the repeated counting of LSC vials over relatively long periods of time, the author has determined that leakage is not occurring to a significant degree. Using water containing high levels of Rn for the initial setup, a vial can be counted at daily or weekly intervals. When this is done, it is clear that virtually all the reduction in Rn over time is explained by simple decay mathematics.

The loss of Rn during the sampling and sample setup steps is a direct function of the level of Rn present in the sample, because the level of Rn in typical air is very low in comparison. This means that the percentage loss is a constant, all other factors being equal, for any Rn level greater than several picocuries per litre. This is not true for the uncertainty of counting radioactive decay, which rises dramatically as the count rate approaches the background count rate. Therefore, the counting uncertainty becomes the major source of variation at low Rn levels.

Counting uncertainty. Before presenting the results of the research, it is necessary to review the basics of counting statistics. The commonly cited equation for calculating the uncertainty that is associated with radioactive decay (single isotope) is

$$u = k(c/t_s + b/t_b)^{1/2} \quad (1)$$

in which u is the uncertainty in the sample count rate, k is the proportionality constant related to the degree of statistical confidence (2.00 for two standard deviations or 95.5 percent), c is the total count rate, b is the background count rate, and t_s and t_b are the counting times for the sample and background, respectively.

Equation 1 is valid for a simplistic view of the uncertainty of Rn counting; however, it is not completely accurate and yields a lower-than-actual uncertainty if a wide window is counted. Lucas described the mathematics of counting for a series of decays and reported a *J*-factor to correct the counting uncertainty given by considering the decaying of one isotope only.[6] Radon and its progeny are measured by LSC; the *J*-factor corrections would be appropriate if a full window was counted. The actual *J*-factor correction is significantly less if a relatively narrow window (optimum protocol) is counted, because the narrow window rejects a large portion of the gamma-beta decay response and does not include all progeny equally. Keeping in mind the complex calculations that would be required for even an empirical *J*-factor correction and the fact that the correction would be small, Eq 1 is considered to adequately describe the uncertainty associated with Rn analysis using LSC.

LSC analysis for Rn

The typical LSC analysis that is used by most laboratories contains the following elements:

- sampling in the field with a 40-mL vial with a PTFE-faced septum cap (alternately, a direct syringe method is used to set up the LSC vial directly in the field),
- transfer of 10 or 5 mL of water beneath 10 mL of fluor contained in a 23-mL glass LSC vial,
- counting on a commercial beta-type LSC instrument after a 4-h progeny ingrowth period, and
- the counting (energy) window is typically wide open or omits the first 25 to 50 channels to exclude the region of highest background level.

This LSC method results in a low counting error for Rn levels >500 pCi/L and has been adequate in the past for relatively high Rn measurements. However, with a new MCL at a level of 300 pCi/L or lower, the method must be improved to yield adequate results. Because Rn decays in transit to the laboratory, it is necessary to be able to measure Rn at a level of approximately 50 percent of that needed for compliance. The author's data show that the average time period between sampling and analysis is actually closer to three days, so four days is conservative. In addition, there is a need to measure Rn at even lower levels in order to monitor treatment performance. Therefore, future demands will dictate that the LSC method be capable of measuring Rn at low levels with a reasonable counting error and time.

The research into the optimization of the LSC method progressed in the following steps:

- determination of the Rn extraction coefficient, D_{Rn}, for the mineral-oil-based fluor routinely used,
- determination of the optimum water-to-fluor ratio for the LSC analysis (related to the following step),
- determination of the optimum counting window (discriminated window) to minimize counting uncertainty, and
- development of a protocol for routine high-volume Rn analysis by LSC.

Extraction of Rn

Rn is chemically inert and cannot be extracted into typical nonimmiscible solvent-based fluors with a particularly high efficiency. An experiment to determine the extraction coefficient for Rn into a mineral-oil-based fluor was conducted by filling the LSC vial (22 mL total liquid volume, 1 mL air space) with varying ratios of water to fluor. The results are shown in Figure 1, in which a value of D_{Rn} of 23 is indicated. The equation for recovery of Rn in water by a solvent is

$$\text{Recovery (percent)} = 100 * D_{Rn} * V_{org} / (D_{Rn} * V_{org} + V_{aq}) \quad (2)$$

in which D_{Rn} is the distribution coefficient for Rn, V_{org} is the volume of fluor, and V_{aq} is the volume of water. The relatively low extraction coefficient for Rn means only partial recovery will be obtained unless a relatively low water-to-fluor ratio is used. A low water-to-fluor ratio may approach a quantitative recovery; however, this reduces the Rn available for extraction and subsequent counting. The net result is that the total amount of Rn in the fluor, rather than the fractional recovery, should be maximized. Thus, the water-to-fluor ratio should be increased over what is commonly used by most laboratories.

Data that demonstrate the concept of maximizing the gross Rn extracted (high water-to-fluor ratio) rather than the recovery fraction (low water-to-fluor ratio) are shown in Figure 2. As these data demonstrate, there is a potential for significantly increased counts—and an associated lower counting uncertainty—if a higher water-to-fluor ratio is used. Also shown in Figure 2 are the currently accepted water-to-fluor ratios of 1 (10 mL fluor) and 2 (5 mL fluor). It is important to note that both protocols, i.e., the ratios of 1 and 2 with only 10 mL of water, have a significant portion of the volume in the LSC vial left to air space. This does not optimize the total amount of Rn within the vial. It should also be pointed out that the 5-mL fluor protocol involving a water-to-fluor-to-air ratio of 10:5:8 can produce lower counting uncertainty than the one employing a ratio of 10:10:3. This is due to a shift and resolution difference in the resulting LSC spectral response, which requires further investigation to determine the optimum water-to-fluor ratio. The shift and resolution differences are understood to be a function of the nonuniformity of the photomultiplier tube face and the difference in volume and position of the fluor phase in the vial and, in turn, in the instrument.

The question regarding optimization can be reduced to the following. Can the LSC analysis protocol for Rn be improved, as measured by a lower counting uncertainty, by increasing the water-to-fluor ratio to effect a greater number of counts per unit time? Because the counting uncertainty is inversely related to the square root of the number of counts, it would appear that the answer would be affirmative. However, because of the effect that a variable water-to-fluor ratio has on the position and resolution of the spectral response, the answer is more complex.

A shifting spectrum and resolution change the background counts associated with any given water-to-fluor ratio. In general, an increase in the fluor volume causes a higher background and a lower resolution. Both result in a negative effect on the counting uncertainty. The position of the spectrum, however, moves toward a region of lower background, and this creates a positive effect. The spectrums shown in Figure 3 illustrate these effects, if it is recognized that the background spectrum has a higher count rate in the lower channel region. Although the 17:5 spectrum is desirable from a resolution and total count standpoint, the region of interest is located in a region of higher background. Thus, the only way to determine the optimum water-to-fluor ratio for an optimal protocol is to perform a series of experiments in which the ratio and the Rn level are varied.

To determine the optimum protocol for Rn LSC analysis, a series of 120 Ra-226 LSC standards was made to cover Rn levels of 0 (background), 10, 25, 50, 75, 100, 125, 150, 200, 500, and 1,000 pCi/L. These standards were made in duplicate for water-to-fluor ratios of 10:5, 10:10, 17:5, 18:4, 19:3, and 20:2. After a minimum of 40 days for Rn ingrowth, the standards were counted for 60 min to obtain a full-spectrum file that could be analyzed. The background samples were counted for 240 min. The individual spectral data were analyzed for the optimum counting window by a computer program that determined the window that gave the lowest counting uncertainty, as given by Eq 1.

The results of the analysis showed that a water-to-fluor ratio of 17:5 was optimum for the determination of Rn at low levels by LSC. Ratios of 18:4 and 19:3 gave near-optimum results.

A comparison of selected data that demonstrate the value of an optimized LSC protocol for Rn is shown in Figure 4. The "full window 10:10" data are typical of current Rn analysis protocols employed by most laboratories, except that even higher uncertainties are sometimes given because the background is not counted for an extended period. Applying an optimum discrimination to the spectrum lowers the uncertainty (optimum window 10:10); the optimum window in this case was approximately channel 140–500. Additional improvement in the counting uncertainty is achieved by using the optimum water-to-fluor ratio of 17:5. The optimum window in this case was approximately channels 70–300. Finally, even lower uncertainty can be achieved by performing a duplicate analysis. This is mathematically equivalent to using twice the counting time, if the samples are truly identical.

Minimum detection limit

It has become common practice for analytical results to be expressed with-

out statistical limits, using the concept of detection limit, as promoted by the USEPA.[7] This is unfortunate because the resulting data base is left censored and of reduced value for later analysis. Porter et al[8] discussed this fact from a scientific standpoint and recommended that sample results should be correctly represented with their associated levels of uncertainty.

Fortunately, radiochemical measurements have usually continued to be reported in the correct manner, without a detection limit. Therefore, for a true zero level, an equal number of replicates with negative and positive values would be expected. The concept of reporting negative values with their associated statistical uncertainty is not well accepted by those who are accustomed to receiving only positive or less-than-detection results. In fact, in past studies, the author and others have incorrectly used a detection limit of, for example, 100 pCi/L.[9] In retrospect, this was in error and the concept of a detection limit for Rn should be dismissed. Alternately, the concept of a reasonable or tolerable overall uncertainty should be used to judge whether results are acceptable. This is routinely referred to as the lowest quantifiable level, but values that are lower than this level should be reported.

As shown in Figure 4, it is not difficult to keep the counting uncertainty <10 percent (2-sigma) at an Rn level of 65 pCi/L at the time of analysis. In fact, it is possible to have a total sampling and analytical error of <10 percent at an Rn level of 80–100 pCi/L. If a 15 percent total uncertainty is accepted, then an Rn level of 45 pCi/L can be used. Alternatively, counting time can be increased and lower levels can be achieved.

With regard to the use of LSC for determining Rn compliance, taking into account a conservative one half-life decay before the sample is counted, the anticipated MCL of 300 pCi/L could be counted with an overall uncertainty of <7–8 percent. In fact, an MCL level of 150 pCi/L could be determined with an overall total uncertainty of 11 percent, using a 60-min count time and duplicate analysis. A 15 percent overall uncertainty would allow an MCL of 90 pCi/L to be determined.

It must be understood that counting time is a variable that can easily and automatically be controlled by the LSC instrument, through a limit on the uncertainty of the total count rate as the sample is being counted. For samples with higher Rn levels, the maximum (adjustable) counting time is automatically terminated via an acceptable statistical level (adjustable) to maximize the total sample throughput of the instrument while maintaining the desired upper bound of statistical uncertainty. Considering the average Rn level in groundwater and the ability of an optimized LSC to lower the counting uncertainty, it is practical for the LSC method to process the large number of samples that will result from the new MCL, even at an Rn level significantly below the anticipated MCL of 300 pCi/L. For example, the Rn level of water utility samples (wells) analyzed by the author's laboratory during the past 12 months has averaged approximately 900 pCi/L. Assuming a decay-reduced level at the time of counting of 450 pCi/L, a counting time of 10 min, and duplicate analyses would yield an overall uncertainty (3 percent for sampling and setup) of 10 percent. Certainly, the capability of the LSC method should not limit the value of the MCL in the range 200–300 pCi/L, as the USEPA has suggested.[10] Additional LSC instruments and longer counting times can be used to address the problem of analysis for Rn <100 pCi/L, if necessary.

Conclusions

Based on the research presented in this article, some conclusions were reached and some recommendations were made:

- Significant improvement in the LSC analysis of Rn at low levels can be achieved through optimization of the protocol and counting parameters.
- The limited extraction of Rn into the counting fluor makes it important to maximize the water-to-fluor ratio and minimize the air void in the counting vial. This must be done in conjunction with an optimization of the counting parameters of the LSC instrument.
- A water-to-fluor volume ratio of 17:5 was found to be optimum for the conditions and LSC instrument used in this study. The often-used water-to-fluor volumes of 10:10 and 10:5 are not optimum for LSC analysis of Rn.
- Counting with an optimized window and an optimized water-to-fluor ratio gives the LSC method a lowest quantifiable level of 150 pCi/L (one half-life decay and 10 percent total sampling and analytical uncertainty). For a 2-sigma total sampling and analytical uncertainty of 15 percent, and a one half-life decay, a lowest quantifiable level of 90 pCi/L (45 actual) can be achieved. All of these values are for a 60-min counting period and duplicate analysis. Lower values can be achieved if the counting period is increased; however, the values cited are for cost-effective commercial analysis of large numbers of samples.
- No significant difference was found between the results of the direct syringe setup and the field sample vial and lab setup technique. There was no significant difference found between a PTFE or aluminum seal on the glass sample container.
- Although no experiments were conducted in this research to find better fluors for Rn extraction, it appears from a review of the physical properties of some alternative organic compounds that the potential may exist to improve efficiency.[11] Such an investigation should be carried out. If extraction efficiency can be increased, even lower Rn levels could be measured with reasonable uncertainty.

Acknowledgment

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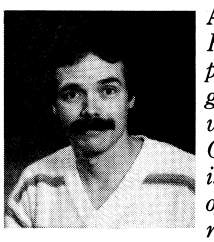

About the author: *Formerly an associate professor of civil engineering at the University of Maine, Orono, Jerry D. Lowry is now chief executive officer and director of research and development for Lowry Engineering Inc., Box 189, Back Troy Rd., Unity, ME 04988. A graduate of the University of Kansas in Lawrence (BS, MS, and PhD degrees), Lowry has 13 years' experience in radon removal research. He is a member of AWWA and ASCE.*