

**Moisture Gain in Activated Charcoal When Measuring Radon
Concentrations in Air by Liquid Scintillation Methods**

An Honors Thesis (HONRS 499)

by

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May 1991

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I. Introduction:

Radon is an inert gas which is produced from the radioactive decay of Uranium-238 deep beneath the ground. The decay chain of alpha and beta emissions between Uranium-238 and Radon-222 is shown in Fig.1. Radon itself is radioactive, and it too begins a series of alpha, beta, and gamma emissions which eventually ends at an isotope of lead. The decay products of radon are referred to as its "daughter products", and they are shown in Fig.2.

If radon enters buildings where people spend a great deal of time, the possibility exists for the people to inhale the gas and/or its daughters. Once this radioactive material has entered the lungs, further decay will damage the delicate lung tissues. Over a period of years, this damage can be sufficient to induce lung cancer¹. In buildings where radon concentrations are elevated, the probability for lung cancer is further increased. For this reason, the screening of homes, schools, and work places for elevated radon levels is an important issue in the healthcare field.

In 1987 the EPA released its first national radon study, revealing that 25% of the homes tested had concentrations above 4.00 picoCuries per liter (pCi/l), which is the EPA's chosen "action level". The study recommended that everyone should have his/her home tested for radon. Since the release of this study, measuring radon concentration levels in air has become an area of intense interest.

One very popular, reliable, and cost-effective method of

screening for radon is by exposing activated charcoal canisters, and then counting the gamma-ray radiation from radon daughters using sodium iodide detectors. However, very few gamma-scintillation systems are automated. As a result, the analysis of the canisters requires a considerable amount of time and labor. In addition, the canisters typically contain 75-grams of charcoal, which increases the shipping and handling costs, thereby increasing the entire cost of the process.

In contrast, lightweight vials containing one or two grams of activated charcoal can be analyzed by an automated, highly efficient liquid scintillation system². The liquid scintillation alternative provides an attractive method for analyzing large volumes of radon tests at low cost. In order to reduce moisture uptake by the charcoal, which decreases the efficiency of the detector, current testing by this method employs the use of a desiccant packet contained inside the vials. Although the cost of two grams of charcoal is almost negligible, kits utilizing a desiccant can cost as much as \$2.50 per vial. Therefore, the question was asked whether or not a procedure for determining radon concentrations by liquid scintillation without the use of a desiccant could be developed. This procedure would require correcting the data for moisture gain in a similar manner to that of the 75-gram canister and NaI detector method.

The analysis of the 75-gram canisters according to EPA protocol³ contains an empirically determined calibration factor which takes humidity levels into account, but no such factor

currently exists for the vials. Consequently, it was decided to investigate whether a relationship exists between canister and vial moisture gains, and use this relationship to find appropriate calibration factors for the vials. An earlier study conducted at Ball State by Marty D. Reese⁴ concluded that moisture corrected 2-gm vials used in liquid scintillation tests could accurately predict radon concentrations for 48-hour testing periods.

This study is a continuation of the previous one, focusing on testing times other than 48 hours. EPA protocol for canisters provides for exposure periods from one to seven days, however, exposure periods from two to four days are recommended and seem to yield the most reliable results. Therefore, the 2-gm vial measurements will be investigated for one- to seven-day exposure periods.

II. Radon Concentration Determinations by Activated Charcoal Canister Methods:

The specifications for the 75-gram activated charcoal canisters used in this study are shown in Table I. At each testing site, seven of the canisters were opened simultaneously. At the end of each subsequent 24-hour period, one of the canisters was resealed, allowing the radon and its daughters to reach equilibrium inside the canister. The result was a series of seven canisters, varying in testing time from 24 hours to 168 hours.

The canisters were then analyzed in a spectrometer system consisting of four Sodium Iodide (NaI) detectors (Fig.3). Each detector counted one canister at a time, giving a gross count of the gamma rays given off by the radon daughters in that canister. A typical "radon" spectrum obtained by a NaI detector is shown in Fig.4 with the labeled photopeaks corresponding to the 295-, 352-, and 609-keV gamma-ray emissions of Ra B and Ra C.

For each of the canisters, an integral sum of the darkened region was obtained for a ten-minute count period. This sum of detected gammas, the gross count rate, was then used to determine the radon concentration in pCi/l using the formula shown below:

$$RN = \frac{\text{Net CPM}}{(T_s) (E) (CF) (DF)}$$

where Net CPM is the gross count rate minus the background, T_s is the exposure time, E is the detector efficiency, CF is the humidity correction factor, and DF is the decay factor.

III. Liquid Scintillation/Vial Method - No Moisture Correction:

The specifications for the 2-gram activated charcoal vials used in the liquid scintillation method are shown in Table II. The activated charcoal used in this investigation was the same type as that used in the 75-gm canisters; however, only two grams were used. For this study, seven of the vials were placed directly beside the seven canisters mentioned in the previous section. As before, one vial was closed at the end of each 24-hour period. After the moisture gain was measured, 10 ml of the specified mineral oil scintillation fluid was added to each vial, and the activity was allowed to reach an equilibrium in the fluid. A study by Schreoder, Vanags, and Hess⁵ determined that ten hours was the minimum time required for the activity to reach a stable equilibrium inside the vials.

Each vial was then counted 50 minutes for radon and radon daughter activity (alphas and betas) with a Beckman 3801-Liquid Scintillation system. Figure 5 shows spectra for Tritium, Carbon-14, and Radon obtained with the Beckman 3801 system. The wide distribution of energies in the radon spectrum is due to the various beta decays associated with the radon daughters. The two distinguishable peaks at higher energies are associated with the alpha emissions of Radon-222 (5.49 MeV), Radium A (6.00 MeV), and Radium C' (7.69 MeV).

As was the case with the canisters, the count rate for the vials had to be corrected for decay, exposure time, and background, but the moisture gain was not taken into consideration initially.

The equation used to determine the vial adjusted count per minute values is shown below:

$$\text{Adj. CPM} = \frac{\text{Net CPM}}{1 - e^{-\frac{(0.693)t_{\text{ex}}}{T_{1/2}}}} \times \frac{e^{-\frac{(0.693)t_d}{T_{1/2}}}}{\text{CF}(0)}$$

where Net CPM is the gross count minus the background count, t_d is the delay time, t_{ex} is the exposure time, and $T_{1/2}$ is the half-life of Rn-222. The value of $\text{CF}(0) = 0.1025$ was used for the zero moisture gain calibration factor, and was obtained directly from the EPA's empirically derived mass calibration curve, which will be discussed in the next section.

IV. Determination of Moisture Correction Factors:

As the tests were being conducted, careful mass measurements to later determine moisture content were performed both before and after each vial-canister pair was exposed. The mass gains were analyzed, and the resulting relationship for testing times up to and including 5 days is shown in Fig.6, with the vial mass gains on the horizontal axis and the canister mass gains on the vertical axis. A least squares fit was performed on the mass data, yielding the equation $\text{delM}(c) = 13.4 * \text{delM}(v) + 1.2 \text{ g}$. The regression analysis, including the uncertainty in slope and intercept values is shown in Table III.

Once this equation was found, the vial mass gains were normalized to equivalent canister mass gains. Exposure times greater than 5 days provided inaccurate results, since the vial moisture gains at these levels did not exhibit any direct correlation with the canister moisture gains. The data from these six- and seven-day exposure times was not included in the subsequent analyses.

For each vial the predicted canister mass gain and the exposure time, T, were utilized to determine a CF(T) factor for the moisture gain. The method for determining this CF(T) was the same as that of the canister method. The equation used requires three separate correction factors, and is shown below:

$$\text{CF}(T) = \text{CF}(48) \times \frac{\text{AF}(T)}{\text{AF}(48)}$$

First, the predicted mass was used to find an initial CF(48)

from the curve shown in Fig.7. This value is labeled CF(48), because the curves used in this analysis were empirically derived by the EPA from 48-hour mass gain data. To locate CF(48), the predicted mass gain was located on the horizontal axis. The value on the vertical axis where the mass gain crossed the curve was the CF(48) value.

This initial CF was multiplied by the ratio of AF(T)/AF(48), which were both determined from the curves shown in Fig.8. According to EPA protocol, the 20% humidity curve was used for canister mass gains less than one gram, the 50% curve for canister mass gains between one and four grams, and the 80% curve for canister gains greater than four grams.

To locate AF(T), the actual exposure time (in hours) was located on the horizontal axis. The location on the vertical axis where the exposure time crossed the correct humidity curve was the AF(T). It was stated earlier that these curves were empirically determined from 48-hour data, therefore, the AF(48) term is used to correct for exposure times other than 48 hours. The value for AF(48) was determined from Fig.8 by locating the 48-hour point on the proper humidity curve.

Once the final CF for each vial was determined, this value was inserted into the adjusted CPM equation:

$$\text{Adj. CPM} = \frac{\text{Net CPM}}{1 - e^{-\frac{(0.693)t_{\text{ex}}}{T_{1/2}}}} \times \frac{e^{\frac{(0.693)t_d}{T_{1/2}}}}{\text{CF}(T)}$$

to determine the mass corrected vial CPM values. This equation is

identical to the one used in the previous section. Plots of the experimental radon concentrations versus both adjusted vial CPM values are shown in Figs.9a and 9b, with the vial adjusted counts per minute on the horizontal axes and the radon concentrations in pCi/l on the vertical axes. Both of these graphs suggest that a linear relationship exists between the adjusted CPM values and the radon concentrations determined by the canister method. A linear least squares fit of both of these sets of data revealed the relationships shown on the two graphs. It should be noted that the quality of the fit obtained by the mass correction method provided better agreement between the experimental data and the simple straight-line curve. The least squares analysis supported this conclusion when one considered the statistical uncertainties in the slope and intercept coefficients. The linear regressions for the data in each of these graphs appear in Table IV, and include the uncertainties in each value.

V. Analysis of Canister/Vial Radon Concentration Comparisons:

For the relationship shown in Fig.9a between the canister radon levels and the uncorrected vial adjusted CPM, values the slope of the calculated line ($Y = 0.00092 X + 0.04$ (pCi/l)) was determined by least squares analysis to exhibit a statistical uncertainty of 6.2%. For the moisture corrected relationship

($Y = 0.00074 X - 0.23$ (pCi/l)), shown in Fig. 9b, the corresponding uncertainty in the slope was determined to be 3.2%, and the uncertainty in the intercept value was 50% lower than that of the uncorrected fit.

For further analytical purposes, each adjusted CPM was used to determine its corresponding predicted radon concentration in pCi/l by plugging them back into the formulas given in Figs. 9a and 9b. The relative error between each calculated concentration and its experimental concentration was then determined. The average of these values for four concentration intervals was computed, and these results are presented in Table V. Although the fractional (%) errors in the 0 - 1 pCi/l interval appear large, EPA protocol assumes absolute uncertainty limits by the canister method to be ± 0.5 pCi/l. It should also be noted that the EPA only requires 25% accuracy in their proficiency testing programs; therefore, the mass corrected levels above 1 pCi/l also fall within an acceptable range.

VI. Prediction of Radon Concentration Values Using 2-gm Vial Method:

Once it was determined that a linear relationship existed between the moisture corrected CPM's obtained by liquid scintillation methods and the radon concentrations determined by the traditional canister method, it was decided to run some test samples to observe the accuracy of the new method. Eleven pairs of vials and canisters were exposed at various test sites for exposure times ranging from one day through five days.

The vials were counted in the liquid scintillation system just as before, and their mass-adjusted CPM's were determined by the same equation. This time, however, the resulting adjusted CPM values were then converted to predicted radon concentration levels by the equation $RN \text{ (pCi/l)} = 0.00074 * \text{adj CPM} - 0.23$. The experimental radon concentration was also determined by the activated charcoal canister method, so that both sets of results could be compared. Once again, the absolute errors and percent errors were determined. The test data results are presented in Table VI. The average percent error for all concentrations above 1 pCi/l was computed at 20.5%, and the average absolute error for levels below this range is 0.16 pCi/l. Overall, this data fits the guidelines used by the EPA, however, when the data was broken down into slightly different ranges these values changed. When the concentrations were observed between 1 and 4 pCi/l the average percent error was only 14.4%, but the error at levels greater than 4 pCi/l was twice as high, at 28.1%.

VII. Conclusion:

It has been shown that the moisture correction AF and CF curves developed by the EPA for the 75-gm activated charcoal canisters can effectively be used in the liquid scintillation 2-gm charcoal vial method for exposure times between one and five days, thus eliminating the need for a desiccant. No longer requiring a desiccant will reduce the cost of commercial kits from about \$2.50 per vial to just a few cents per vial.

However, the newly proposed method requires more testing before it can become an accepted method for determining radon concentrations. The error in the test data at radon concentration levels greater than 4 pCi/l was too large to be acceptable. The results of this investigation seem to indicate reliable results can be obtained for one- to five-day exposures, but the question of whether or not radon screening results for exposure times greater than five days can be reproduced by the method still exists. Following are several suggestions which may help address these problems in the future.

The first suggestion for further research is to examine the relationship between canister and vial mass gains to determine what functional relationship (if any) may exist. Although the linear fit used in this study closely approximates the data for one- to five-day exposures, perhaps another analysis with different variables will reveal a better representation of the data. As more and more data concerning this relationship is acquired, it may be possible to reduce uncertainties currently caused by poor mass gain

correlations, particularly at higher moisture gains. Also, the data for longer exposure times needs to be re-evaluated. It is possible that closer scrutiny of this data will reveal that it too can be handled by liquid scintillation methods.

Another suggestion is to place two or three times more of the vials, 14 or 21 for example, at each test site. Then, instead of closing one at the end of each 24-hour period, two or three could be closed. This would provide better counting statistics for the liquid scintillation method, and ensure that the results being acquired are reproducible.

The final suggestion is to investigate the effect of using different scintillation fluids. Mid way through this study, a scintillation fluid called Toluene/Popop was examined.

Preliminary results obtained using Toluene/Popop as the scintillation fluid appear in the Appendix C at the end of this paper.

In conclusion, it has been shown that the 2-gram vial method is a reliable method of radon testing for one- to five-day exposures, however, using these techniques will require more testing before they can effectively measure all levels of radon for all exposure times. The suggestions for further investigation may help to improve the method in the near future. The automation and efficiency of the liquid scintillation system, in combination with the cost-effectiveness of using only two grams of activated charcoal and no desiccant make the use of liquid scintillation techniques a promising method for radon testing in the future.

VIII. References:

1. United States Environmental Protection Agency- "A Citizens Guide to Radon - What it is and What to Do About It," OPA-86-004, (August 1986).
2. Perlman - "Detecting and Measuring Radon by Liquid Scintillation Counting," Beckman Technical Information, (1987).
3. Gray and Windam - "EERF Standard Operating Procedures for Rn-222 Measurement Using Charcoal Canisters," EPA 520/5-87-005, (March 1987).
4. Reese - "The Effects of Moisture Gain on Activated Charcoal When Measuring Radon Concentrations in Air by Liquid Scintillation Methods," Ball State University, (March 1991).
5. Schroeder, Vanags, and Hess - "An Activated Charcoal-Based, Liquid Scintillation Analyzed Airborne Rn Detector," Health Physics 57, 1 (July 1989).

Figure 1

Decay Scheme of Uranium-238

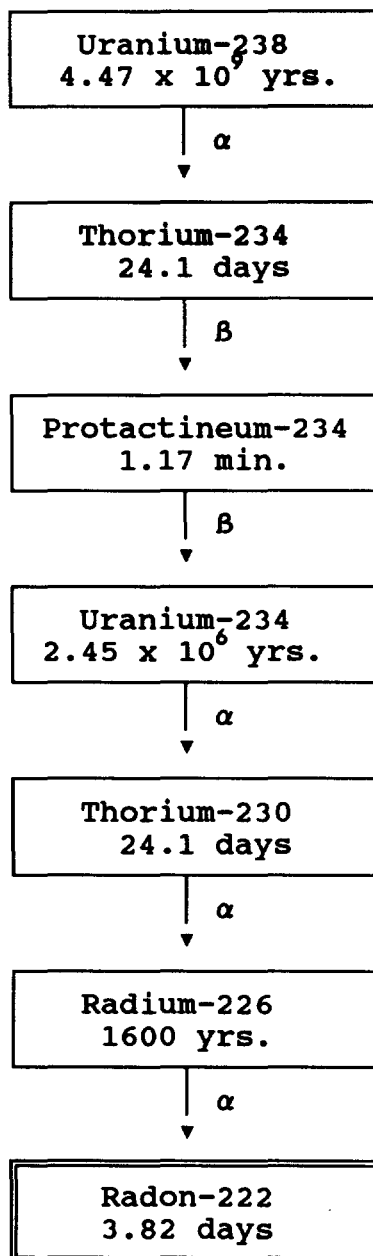
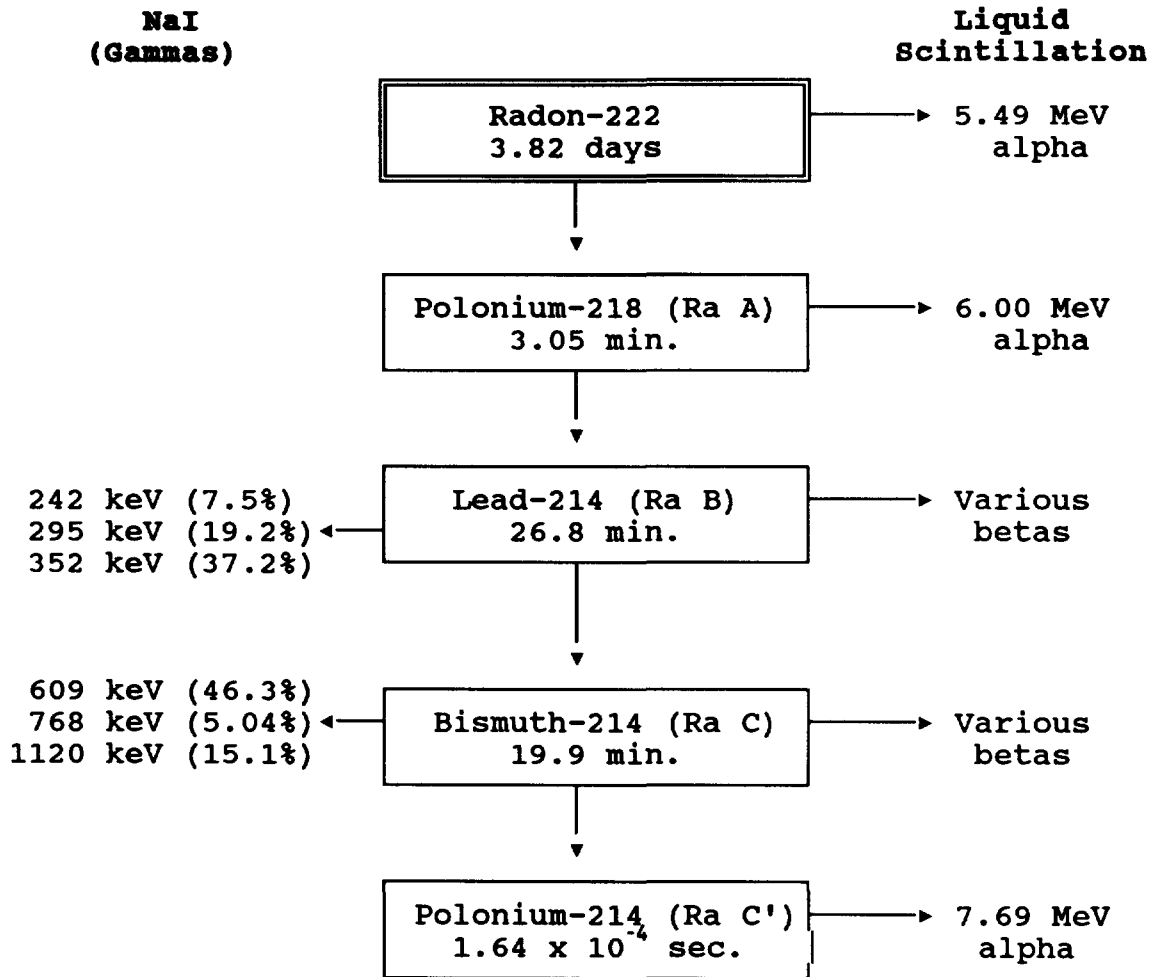


Figure 2

Decay Scheme of Radon-222



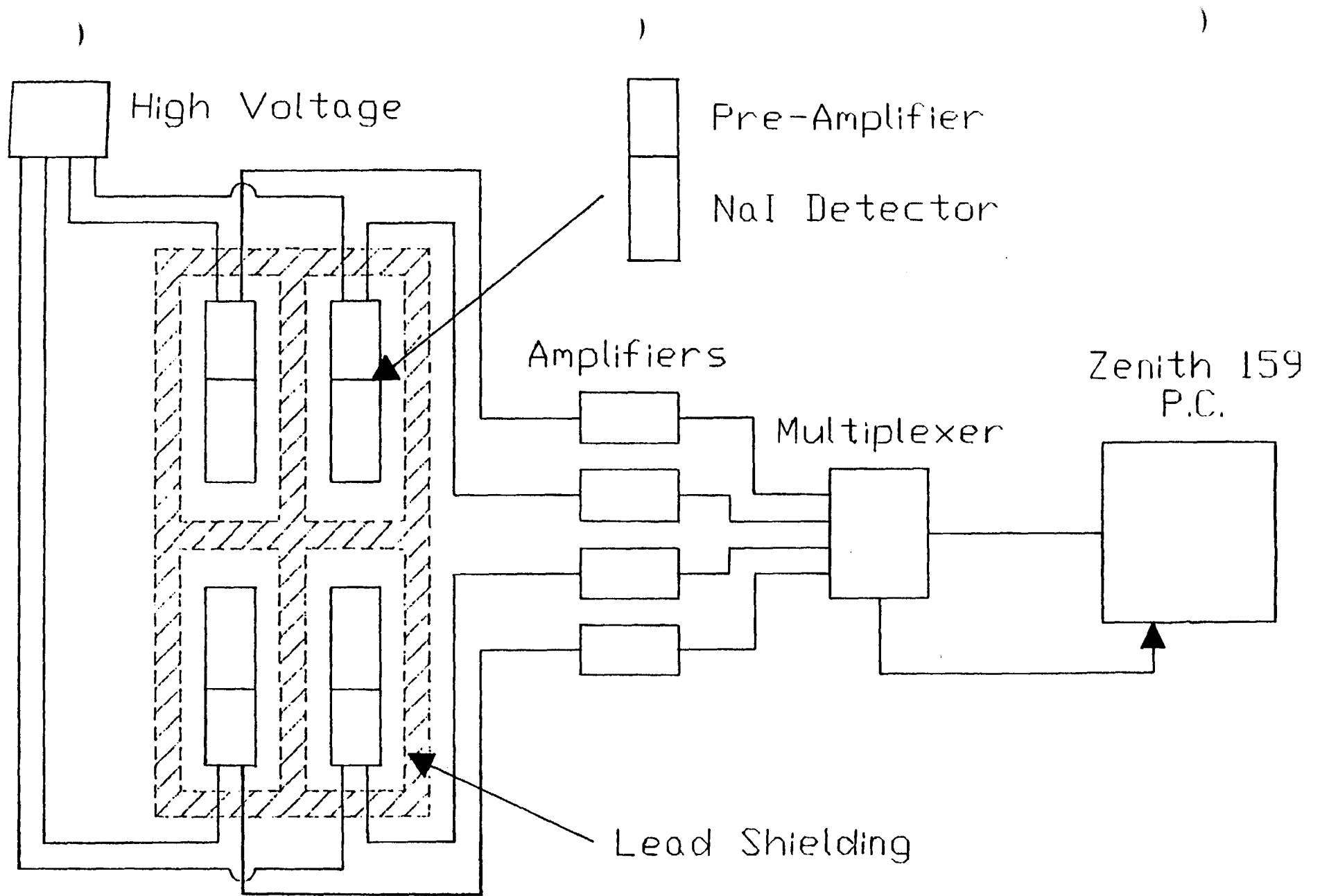


Figure 3 - Diagram of Four-Detector Spectrometer System

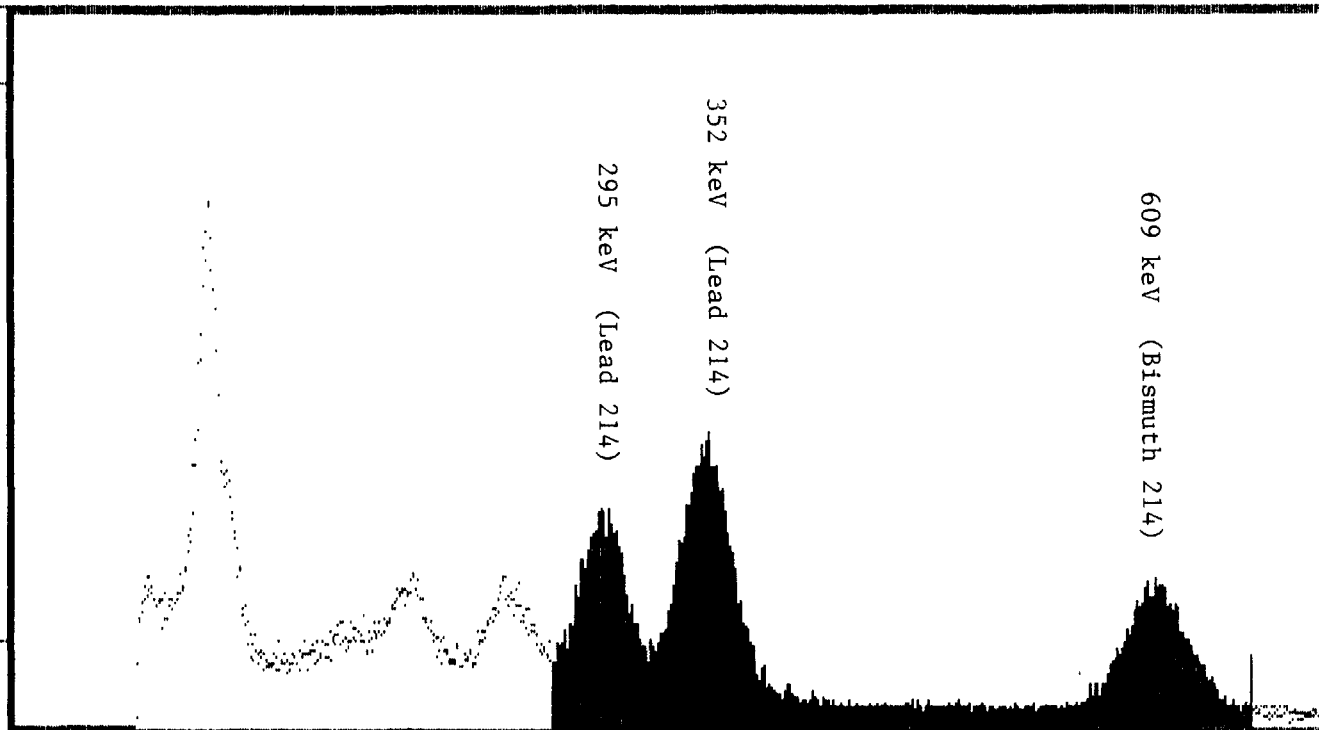
Nucleus DMR-II

01:11:50 am Apr 18, 1991

Apr 18, 1991
01:15:25 am

Acquire: Off
Mode: PHA
Timer: Live
Scale: 1K
Input: 1
Roi No: 1
Roi: On
Gain: 1024
Offset: 0
Adc: Add
Display: 1024
Armed: On

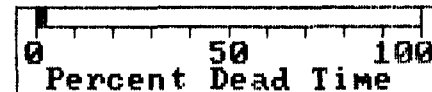
Chn: 962
Cts: 31



Preset: 60 Elapsed: 60 Remaining: 0

ROI Int: 56603 ± 238 ROI Net: 22541 ± 301

Centroid: 537.65 Chn FWHM: 43.92 Chn FWTM: 67.47 Chn



| | |
|--|--|
| | |
| | |
| | |

F1-Acquire F2-Erase F3-Preset F4-Expand F5-Ident F6-Load F7-Save Esc-ROI

Figure 4 - Typical Radon Spectrum Obtained by NaI Detector

Fig. 5

Doped Source Spectra
Liquid Scintillation System

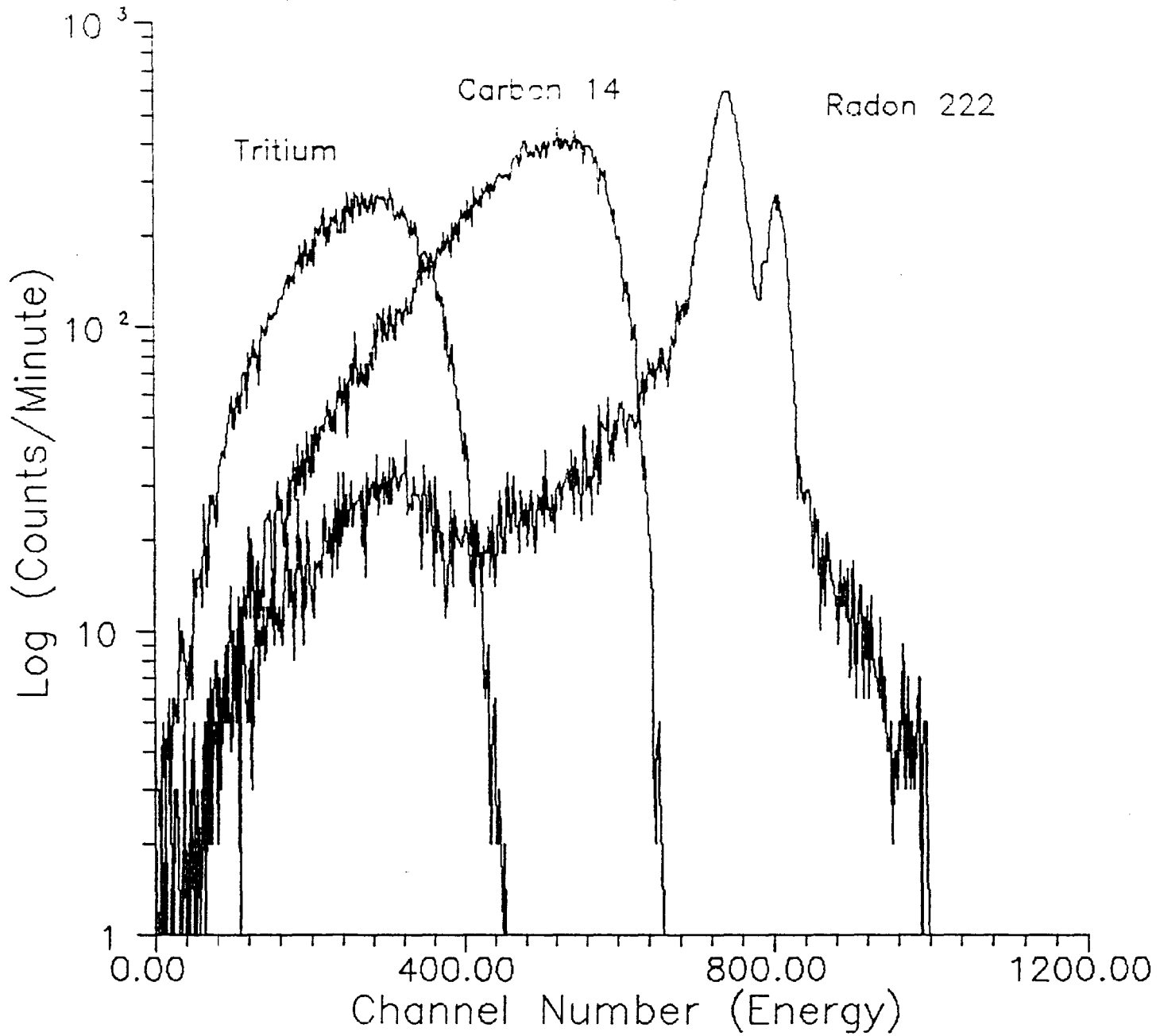
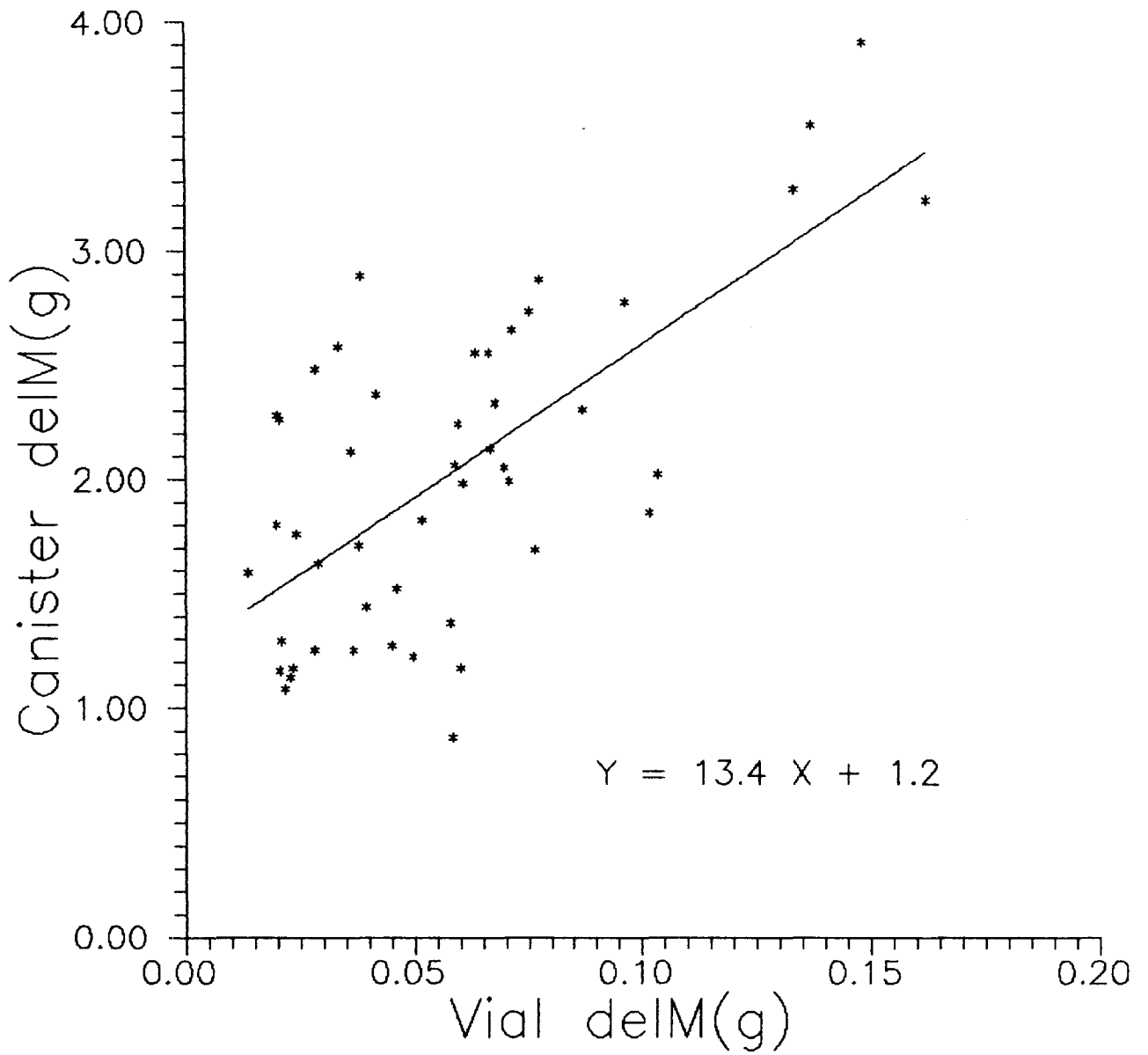
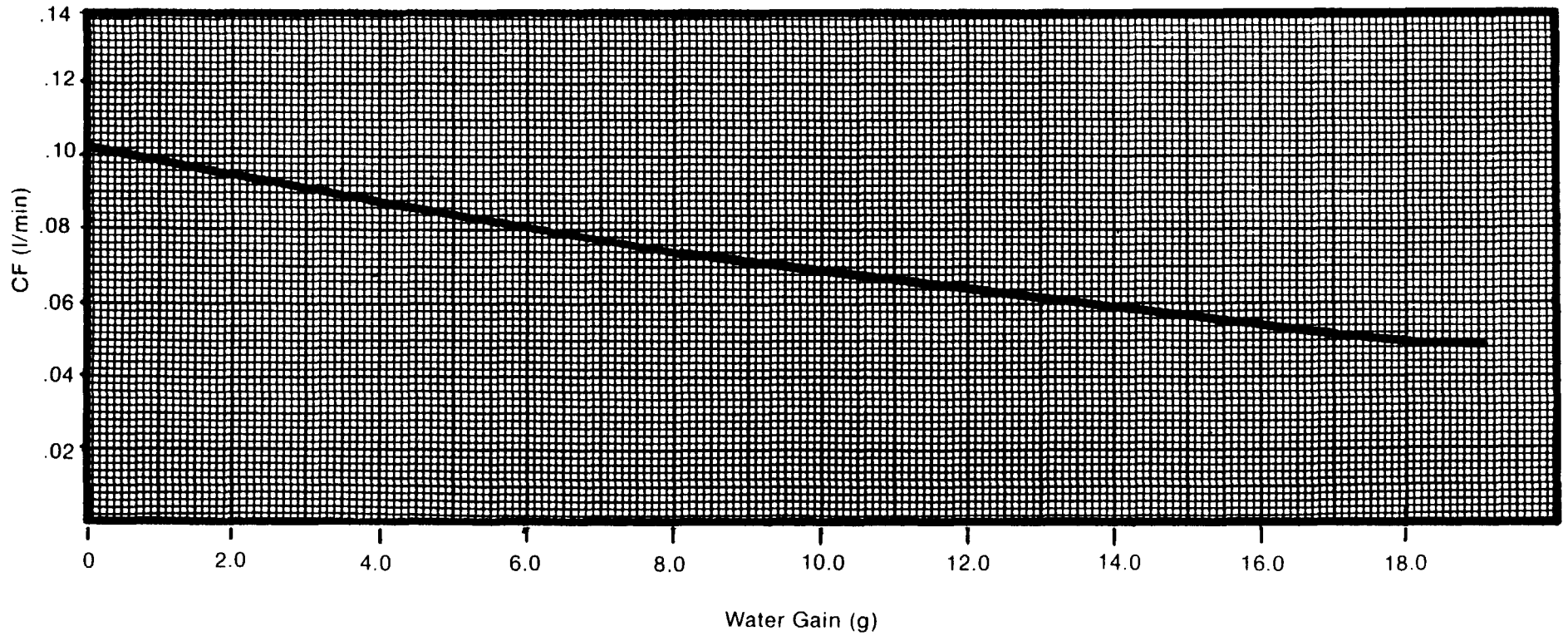


Figure 6
Graph of Vial Mass Gains vs.
Canister Mass Gains



**FIGURE 7. WATER GAIN VERSUS CALIBRATION FACTORS (CF)
FOR A TWO DAY EXPOSURE**



**Figure 8. EXPOSURE TIME VERSUS ADJUSTMENT FACTORS (AF)
FOR LOW, MEDIUM, AND HIGH HUMIDITY**

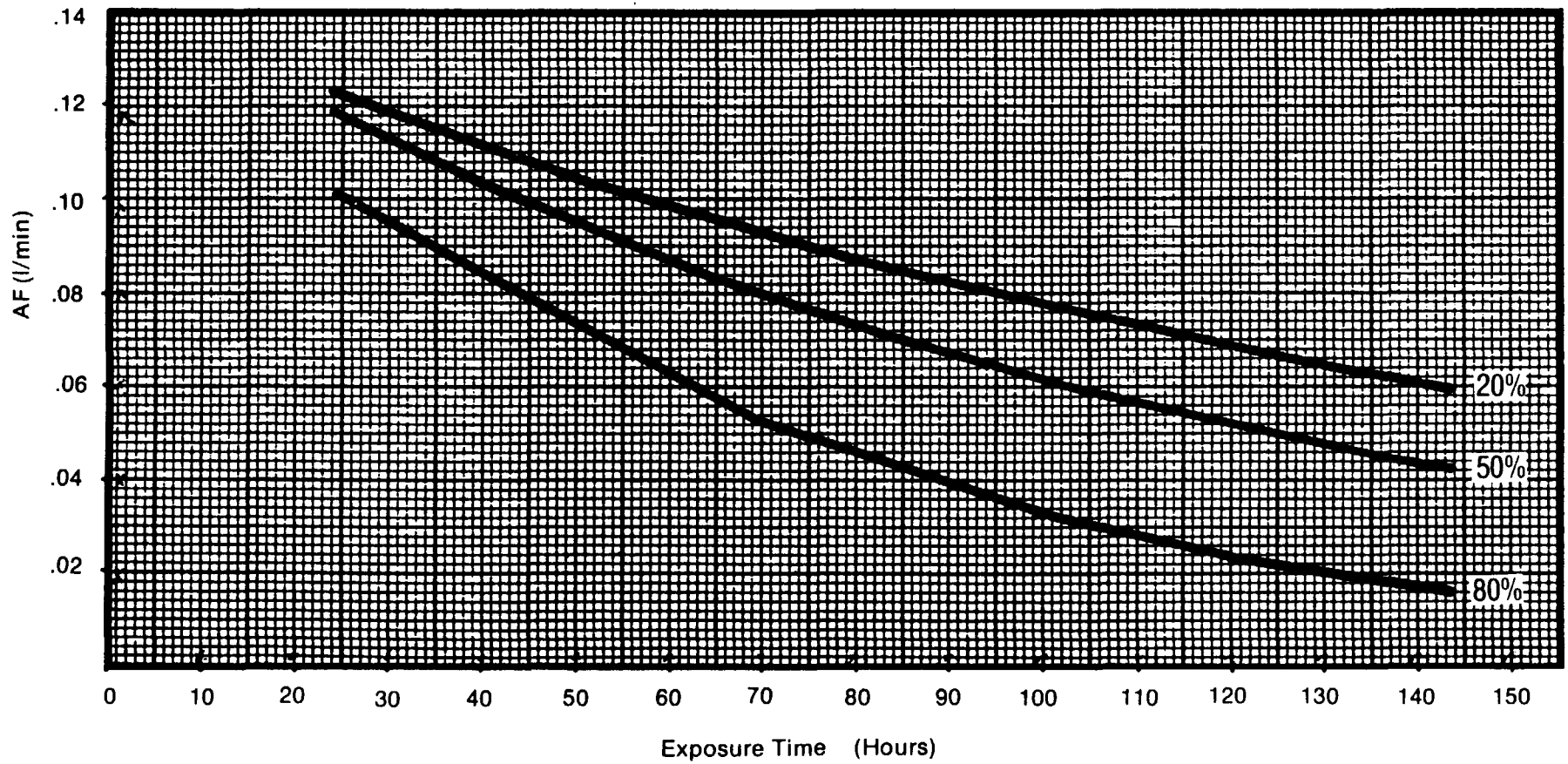


Figure 9a

Graph of Vial Adj. CPM vs. RN (pCi/l) Concentration
No Moisture Gain Correction Factor

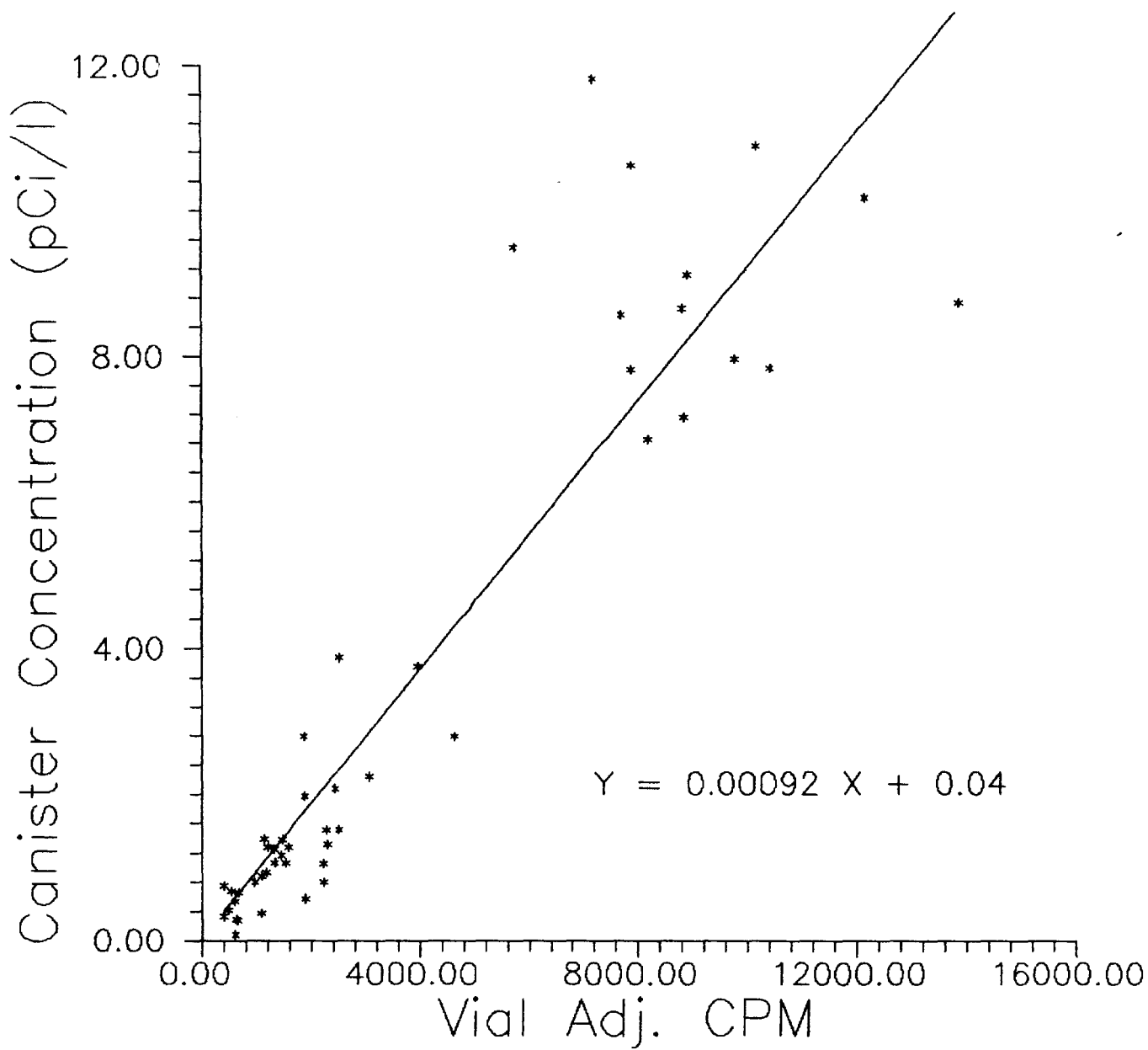


Figure 9b

Graph of Vial Adj. CPM vs. RN (pCi/l) Concentration
Moisture Gain Correction Method

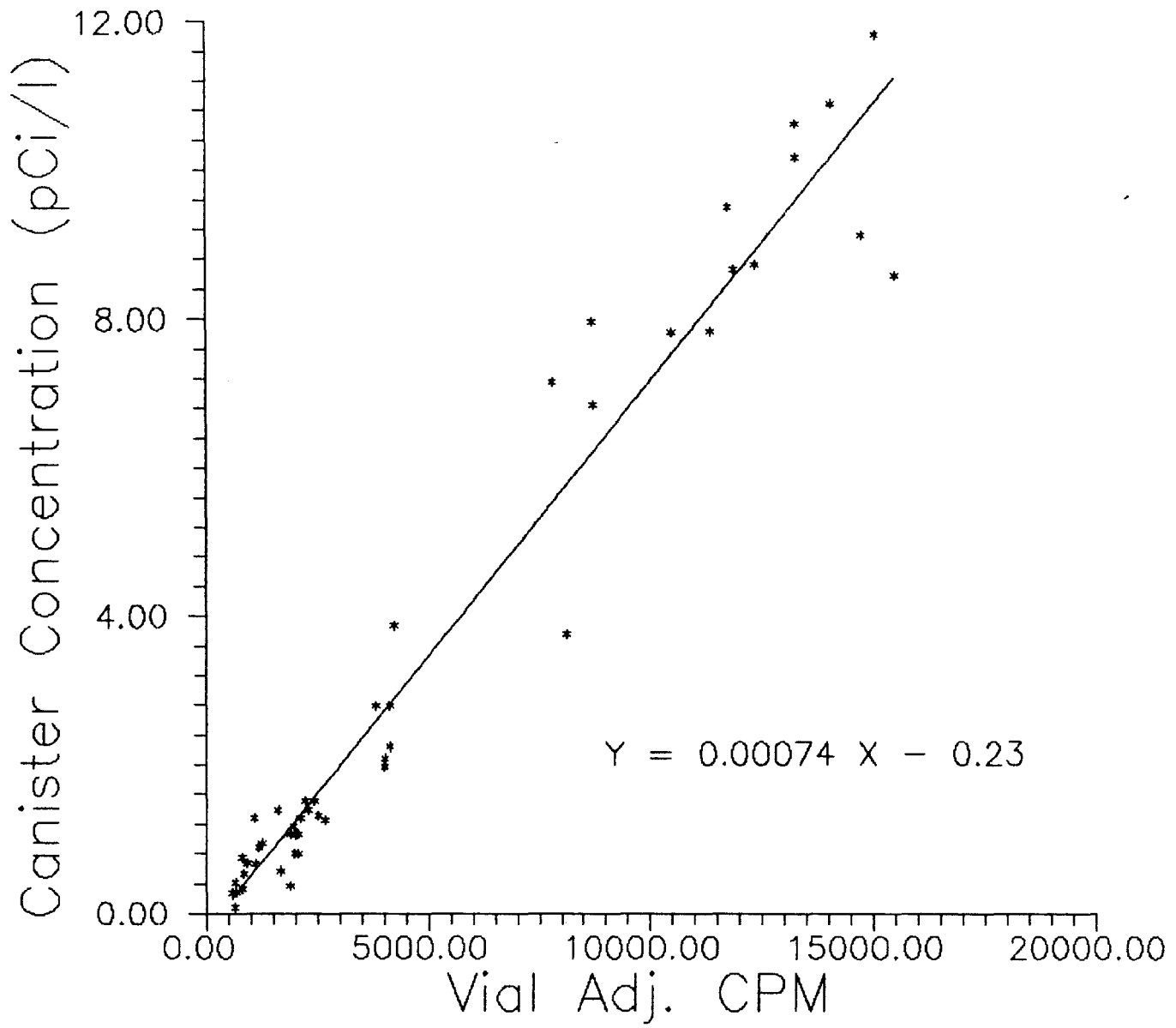


Table I

Canister Specifications

Canister : 4-inch metal can
Manufacturer : F & J Specialty Products, Miami Springs, FL
Charcoal : 75 grams of 8 X 16 mesh
Calgon Corp. Type PCB

Table II

Liquid Scintillation Vial Specifications

Scintillation Vial : 3/4" diameter 20 ml glass vial
Charcoal : 2 grams (same type as for canisters)
Scintillation Fluid : 10 ml of High Efficiency Mineral Oil
Scintillator PSS-007H
Manufacturer : Biotechnology Systems (DuPont)

Table III

Linear Regression Analysis of Vial vs. Canister Mass Gains

| | |
|-----------------------------|----------|
| Constant [Y-intercept (gm)] | 1.248491 |
| Std Err of Y Est | 0.523726 |
| R Squared | 0.455333 |
| No. of Observations | 48 |
| Degrees of Freedom | 46 |
| X Coefficient(s) [Slope] | 13.37925 |

Table IV

Linear Regression Analysis of Moisture Corrected
Vial Adjusted CPM vs RN Concentration (pCi/l)

| | |
|--|----------|
| Constant [Y-intercept (pCi/l)] | -0.23391 |
| Std Err of Y Est | 0.790617 |
| R Squared | 0.955253 |
| No. of Observations | 48 |
| Degrees of Freedom | 46 |
| X Coefficient(s) [Slope ((pCi/l)/CPM)] | 0.000739 |
| Std Err of Coef. | 2.36E-05 |

Table V

Average Errors Between Predicted Radon Concentration from the 2-gm Vials and Experimental Radon Concentration from the 75-gm Canisters Over Selected Concentration Ranges

$$\text{Where relative error} = \frac{| RN_{\text{exp}} - RN_{\text{pred}} |}{RN_{\text{exp}}} \times 100\%$$

| RN Level | Average Error | Number in Range | Errant Results |
|-------------|---------------|-----------------|----------------|
| 0 - 1 pCi/l | ± 0.26 pCi/l | 15 | 1 |
| 1 - 4 pCi/l | 22 % | 19 | 6 |
| 4 - 8 pCi/l | 12 % | 5 | 0 |
| ≥ 8 pCi/l | 10 % | 9 | 1 |

Table VI

Predicted Radon Concentrations and Error Analysis Using the
Moisture Corrected 2-gm Liquid Scintillation Vial Method

| ID # | Exposure Time (d) | Canister pCi/l | Predicted pCi/l | Absolute Error | Percent Error |
|------|-------------------|----------------|-----------------|----------------|---------------|
| 1 | 1.3 | 0.3 | 0.18 | 0.12 | 40 |
| 2 | 2.29 | 0.46 | 0.65 | 0.19 | 41.3 |
| 3 | 1.11 | 1.17 | 1.14 | 0.03 | 2.56 |
| 4 | 2.03 | 1.22 | 1.42 | 0.2 | 16.39 |
| 5 | 1.72 | 1.44 | 1.61 | 0.17 | 11.81 |
| 6 | 3.1 | 1.51 | 1.77 | 0.26 | 17.22 |
| 7 | 2.64 | 1.54 | 1.91 | 0.37 | 24.03 |
| 8 | 2.56 | 4.86 | 3.38 | 1.48 | 30.45 |
| 9 | 2.99 | 9.44 | 6.68 | 2.76 | 29.24 |
| 10 | 4.1 | 8.7 | 7.72 | 0.98 | 11.26 |
| 11 | 4.99 | 14.58 | 8.52 | 6.06 | 41.56 |

Table VI

Predicted Radon Concentrations and Error Analysis Using the
Moisture Corrected 2-gm Liquid Scintillation Vial Method

| ID # | Exposure Time (d) | Canister pCi/l | Predicted pCi/l | Absolute Error | Percent Error |
|------|-------------------|----------------|-----------------|----------------|---------------|
| 1 | 1.3 | 0.3 | 0.18 | 0.12 | 40 |
| 2 | 2.29 | 0.46 | 0.65 | 0.19 | 41.3 |
| 3 | 1.11 | 1.17 | 1.14 | 0.03 | 2.56 |
| 4 | 2.03 | 1.22 | 1.42 | 0.2 | 16.39 |
| 5 | 1.72 | 1.44 | 1.61 | 0.17 | 11.81 |
| 6 | 3.1 | 1.51 | 1.77 | 0.26 | 17.22 |
| 7 | 2.64 | 1.54 | 1.91 | 0.37 | 24.03 |
| 8 | 2.56 | 4.86 | 3.38 | 1.48 | 30.45 |
| 9 | 2.99 | 9.44 | 6.68 | 2.76 | 29.24 |
| 10 | 4.1 | 8.7 | 7.72 | 0.98 | 11.26 |
| 11 | 4.99 | 14.58 | 8.52 | 6.06 | 41.56 |

Appendix C

Evaluation of Toluene/Popop Data

As mentioned in the paper, this study also investigated the use of other types of scintillation fluid. This appendix will list some of the preliminary results obtained for the 2-gm vials with Toluene/Popop replacing mineral oil as the scintillation fluid.

After the 2-gm vials were closed, 10-ml of Toluene/Popop was added to each one, and the radon inside was allowed to reach an equilibrium state with its daughters. One interesting discovery was the fact that radon in Toluene/Popop may only require four to five hours to reach equilibrium, in contrast with the ten-hour minimum time required for the mineral oil fluid.

The other major insight into the Toluene/Popop scintillation fluid is its extremely high efficiency. In fact, counts taken using Toluene/Popop as the scintillator reveal count rates nearly double the corresponding count rates obtained using mineral oil. Any increase in efficiency will result in a corresponding decrease in statistical uncertainty, therefore, it is usually desirable to use the highest-efficiency fluid available.

The data obtained from the liquid scintillation system was then analyzed by the proposed moisture correction method described in the paper. The vial adjusted count rates obtained with Toluene/Popop seem to obey a very good linear relationship to the canister RN concentrations (see graph on next page). This data indicates that the use of Toluene/Popop scintillation fluid should be investigated in greater depth in the future.

Appendix C – Toluene/Popop

Vial Adj. CPM vs. Concentration (pCi/l)

