

2016 NIST (^{133}Xe) and Transfer ($^{131\text{m}}\text{Xe}$, $^{133\text{m}}\text{Xe}$, ^{135}Xe) Calibration Report

Troy Robinson

March 2017



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Troy Robinson

March 2017

**Idaho National Laboratory
Global Security and International Safeguards
Idaho Falls, Idaho 83415**

<http://www.inl.gov>

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ABSTRACT

A significantly improved calibration of the High Purity Germanium detectors used by the Idaho National Laboratory Noble Gas Laboratory was performed during the annual NIST calibration. New sample spacers provide reproducible and secure support of samples at distances of 4, 12, 24, 50 and 100 cm. Bean, 15mL and 50mL Schlenk tube geometries were calibrated. Also included in this year's calibration was a correlation of detector dead-time with sample activity that can be used to predict the schedule of counting the samples at each distance for each geometry. This schedule prediction will help staff members set calendar reminders so that collection of calibration data at each geometry will not be missed. This report also correlates the counting efficiencies between detectors, so that if the counting efficiency on one detector is not known, it can be estimated from the same geometry on another detector.

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ACRONYMS

HPGe- High Purity Germanium Detector

NIST- National Institute of Standards and Technology

INL- Idaho National Laboratory

NGL- Noble Gas Laboratory

Det2- Detector 2

2016 NIST (^{133}Xe) and Transfer ($^{131\text{m}}\text{Xe}$, $^{133\text{m}}\text{Xe}$, ^{135}Xe) Calibration Report

INTRODUCTION

Previous years NIST calibrations focused on checking the Detector 2 (Det2) NIST calibration because it is often the favored High Purity Germanium (HPGe) detector due to its high efficiency. This year there were three main goals: 1) measure the dead-time vs. sample activity for all three detectors at various counting geometries, 2) determine the NIST efficiencies for a larger set of counting geometries, and 3) to determine the well detector NIST efficiency to update the running average of the detector efficiencies. All goals were achieved and the results are described in this report. In addition, transfer calibration for $^{133\text{m}}\text{Xe}$, $^{131\text{m}}\text{Xe}$, and ^{135}Xe were also conducted to round out the recalibration of the new detector sample spacers.

PREVIOUS CALIBRATION GEOMETRIES AND COUNTING EFFICIENCIES

In previous gas lab operations, the well detectors (Det2 and Det3) were solely used for final spike quantification and the coaxial detector (Det1) was only used for initial activity determinations at distance. The anticipated increase in sample production for the INL Noble Gas Lab (NGL) will require the detectors to perform counting tasks they previously were not calibrated to carry out. For this purpose, Det2 and Det3 were calibrated for counting at various distances, giving them the counting capability of Det1. The INL NGL has previously received requests for extremely high activity samples that were impossible to count at the closer geometries (24cm or less) due to excessive dead time at the time of count. As a result, Det1 was calibrated to be able to carry out sample counts at 50 and 100 cm distances using stacked plastic Marinelli beakers (see Figure 1 for examples of 4, 12 and 24 cm distance using stacked Marinelli beakers). Stacking enough plastic Marinelli beakers to achieve the 50 and 100 cm distance (approximately 8 beakers to reach 100 cm) was very precarious due to the lack of stability in the stack of Marinelli beakers. To overcome this, new spacers were commissioned and fabricated (See Figure 2 for a comparison of the Marinelli vs plastic tube spacer) and allow stable and reproducible counting distances of 4, 12, 24, 50, 100 cm (See Figure 3).

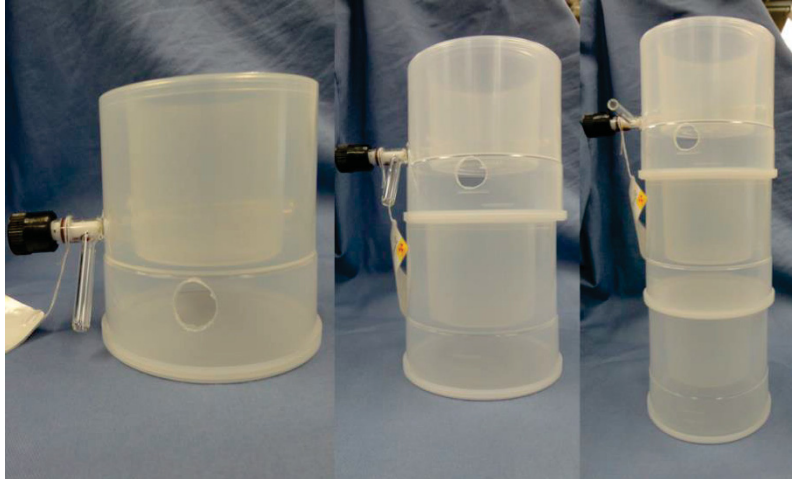


Figure 1. Marinelli spacer examples left to right: 0 or 4 cm, 12 or 16 cm, 24 or 28 cm. Distances for Marinellis change based on if the bottom lid is in place, or removed allowing the Marinelli well to rest directly on the detector head.



Figure 2. Comparison of Marinelli spacer vs Plastic tube spacers for the 4 cm distance. Notice the plastic tube spacer on the right wraps around the detector head offering more stability, especially at the 50 and 100 cm distances.



Figure 3. New plastic tube spacers. Distances show from left to right: 100, 50, 24, 12, 4 cm.

COUNTING EFFICIENCIES USING STACKING MARINELLI SPACERS

The ANSI standard [1] for HPGe counting techniques recommends that a minimum of 40,000 counts be obtained for calibration assays. Previous to learning of this recommendation, our laboratory had thought that 10,000 counts was sufficient for calibration as well as quantification, as that generally yields approximately 1% counting uncertainty. Previously determined counting efficiencies using Marinelli spacers, and the well detector are shown in

Table 1. Counting Efficiencies determined using stacking Marinelli spacers. Red text indicates efficiencies that were calculated using data with fewer than 40,000 counts (the minimum recommended for calibration in the ANSI Standard [1] for HPGe counting). Results were obtained using 2014 Calibration samples.

| ¹³³ Xe Efficiency with Marinelli Spacers | | | | | | |
|---|--------------------------|----------------|--------------------------|----------------|--------------------------|----------------|
| Bean | | | | | | |
| Distance (cm) | Det1 Marinelli Spacer | | Det2 Marinelli Spacer | | Det3 Marinelli Spacer | |
| | %Eff | 1 σ unc | %Eff | 1 σ unc | %Eff | 1 σ unc |
| Well | | | 85.71% | ±1.19% | 78.18% | ±1.25% |
| 0 | 11.94% | ±0.17% | | | | |
| 4 | 3.17% | ±0.05% | | | | |
| 12 | 0.744% | ±0.012% | | | | |
| 24 | 0.219% | ±0.003% | | | | |

| ¹³³ Xe Efficiency with Marinelli Spacers | | | | | | |
|---|--------------------------|----------------|--------------------------|----------------|--------------------------|----------------|
| 15mL Schlenk Tube | | | | | | |
| Distance (cm) | Det1 Marinelli Spacer | | Det2 Marinelli Spacer | | Det3 Marinelli Spacer | |
| | %Eff | 1 σ unc | %Eff | 1 σ unc | %Eff | 1 σ unc |
| 0 | 6.29% | ±0.09% | | | | |
| 4 | 2.13% | ±0.03% | | | | |
| 16 | 0.37% | ±0.01% | | | | |
| 20 | 0.28% | ±0.00% | | | | |
| 24 | | | | | | |

| ¹³³ Xe Efficiency with Marinelli Spacers | | | | | | |
|---|--------------------------|----------------|--------------------------|----------------|--------------------------|----------------|
| 50mL Schlenk Tube | | | | | | |
| Distance (cm) | Det1 Marinelli Spacer | | Det2 Marinelli Spacer | | Det3 Marinelli Spacer | |
| | %Eff | 1 σ unc | %Eff | 1 σ unc | %Eff | 1 σ unc |
| 0 | 4.55% | ±0.06% | | | | |
| 8 | 0.97% | ±0.02% | | | | |
| 20 | 0.255% | ±0.004% | | | | |

PLASTIC TUBE SPACER GEOMETRY CALIBRATION EFFICIENCIES

Calibration assays were also recorded with the plastic tube spacers to calculate counting efficiencies. These efficiencies compare reasonably with the stacking Marinelli spacers, but are not identical due to small differences in spacer material shielding and slight differences in the actual vs. nominal distance. These results were obtained using 2016 NIST calibration samples. Sample counting dead-time was limited to lower than 25% for Det1 and Det3 and 60% for Det2. Past results for Det2 had shown that 60% was sufficiently low to obtain reproducible counting and calibration results (see Figure 4). The 25% dead time threshold was selected by studying the calibration data and finding the limit where trend artifacts from dead-time were no longer present. Calibration count data was corrected for self-attenuation using the pressure measured during the material transfer to the bean sample container.

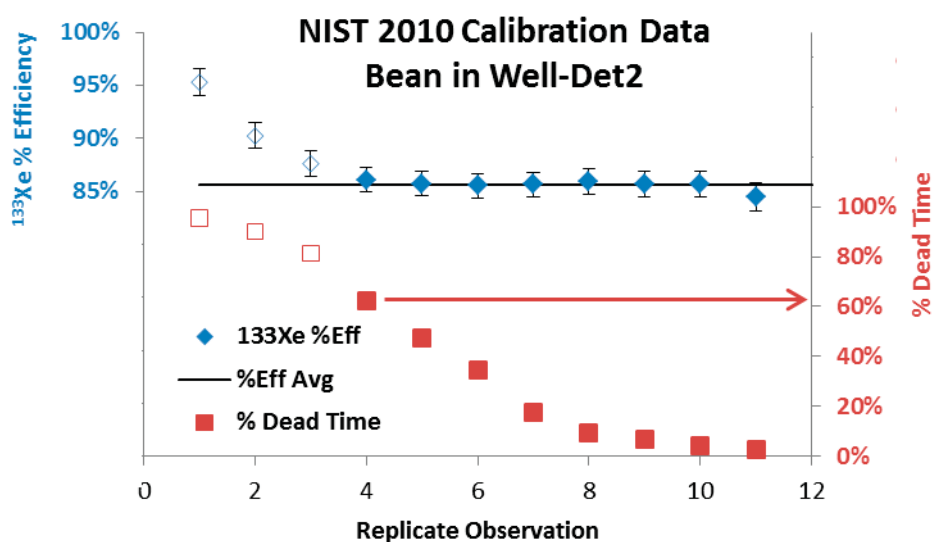


Figure 4. NIST 2010 Calibration data for bean in Well on Det2. At approximately 60% dead time, the detector appears to provide count data that is reliable, judging from the trend in the early counts and random error of subsequent measurements.

Table 2. Calibration Efficiencies using the plastic tube spacers. Results were obtained using 2016 NIST Calibration samples.

| ¹³³ Xe Efficiency with Tube Spacers | | | | | | |
|--|---------------------|----------|---------------------|-----------|---------------------|----------|
| Bean | | | | | | |
| Distance (cm) | Det1 Tube Spacer | | Det2 Tube Spacer | | Det3 Tube Spacer | |
| | %Eff | 1σ unc | %Eff | 1σ unc | %Eff | 1σ unc |
| Well | N/A | | 85.94% | ±0.95% | 76.92% | ±0.85% |
| 0 | 12.52% | ±0.14% | 7.22% | ±0.09% | 13.94% | ±0.18% |
| 4 | 3.50% | ±0.04% | 1.14% | ±0.01% | 3.00% | ±0.03% |
| 12 | 0.740% | ±0.01% | 0.204% | ±0.002% | 0.592% | ±0.01% |
| 24 | 0.213% | ±0.002% | 0.058% | ±0.001% | 0.168% | ±0.002% |
| 50 | 0.055% | ±0.001% | 0.0150% | ±0.0002% | 0.0446% | ±0.0005% |
| 100 | 0.0143% | ±0.0002% | 0.00392% | ±0.00004% | 0.0117% | ±0.0001% |

| ¹³³ Xe Efficiency with Tube Spacers | | | | | | |
|--|---------------------|----------|---------------------|-----------|---------------------|----------|
| 15mL Schlenk Tube | | | | | | |
| Distance (cm) | Det1 Tube Spacer | | Det2 Tube Spacer | | Det3 Tube Spacer | |
| | %Eff | 1σ unc | %Eff | 1σ unc | %Eff | 1σ unc |
| 0 | 5.73% | ±0.07% | 1.767% | ±0.02% | 5.279% | ±0.07% |
| 4 | 2.45% | ±0.03% | 0.711% | ±0.008% | 2.084% | ±0.02% |
| 12 | 0.631% | ±0.007% | 0.175% | ±0.002% | 0.506% | ±0.006% |
| 24 | 0.195% | ±0.002% | 0.0533% | ±0.0006% | 0.156% | ±0.002% |
| 50 | 0.0508% | ±0.0006% | 0.0137% | ±0.0002% | 0.0417% | ±0.0005% |
| 100 | 0.0133% | ±0.0001% | 0.00357% | ±0.00004% | 0.0109% | ±0.0001% |

| ¹³³ Xe Efficiency with Tube Spacers | | | | | | |
|--|---------------------|----------|---------------------|-----------|---------------------|----------|
| 50mL Schlenk Tube | | | | | | |
| Distance (cm) | Det1 Tube Spacer | | Det2 Tube Spacer | | Det3 Tube Spacer | |
| | %Eff | 1σ unc | %Eff | 1σ unc | %Eff | 1σ unc |
| 0 | 4.43% | ±0.05% | 1.275% | ±0.02% | 4.171% | ±0.05% |
| 4 | 1.99% | ±0.02% | 0.563% | ±0.006% | 1.677% | ±0.02% |
| 12 | 0.548% | ±0.01% | 0.152% | ±0.002% | 0.445% | ±0.005% |
| 24 | 0.174% | ±0.002% | 0.0473% | ±0.0005% | 0.138% | ±0.002% |
| 50 | 0.044% | ±0.000% | 0.0121% | ±0.0001% | 0.0364% | ±0.0004% |
| 100 | 0.0113% | ±0.0001% | 0.00308% | ±0.00003% | 0.009% | ±0.0001% |

HISTORICAL ROLLING AVERAGE OF WELL DETECTOR EFFICIENCIES

The well detector efficiencies matched well between the 2014 and 2016 NIST calibrations. Det2 well efficiencies dating back to 2010 were available, whereas Det3 calibrations using the NIST calibration samples date back to 2014. Plots of the efficiency results for Det2 and Det3 are shown in Figure 5 and Figure 6, respectively. The effect of the 2016 data on the overall rolling averages are shown in Table 3. It is apparent from Table 3 that Det2's data did not see a significant change in the weighted average efficiency, whereas Det3's weighted average increased by nearly 1σ . This is likely due to the larger number of data points available to determine Det2's weighted average. It is anticipated that the rolling average for Det3 will settle as more data points are collected throughout the years.

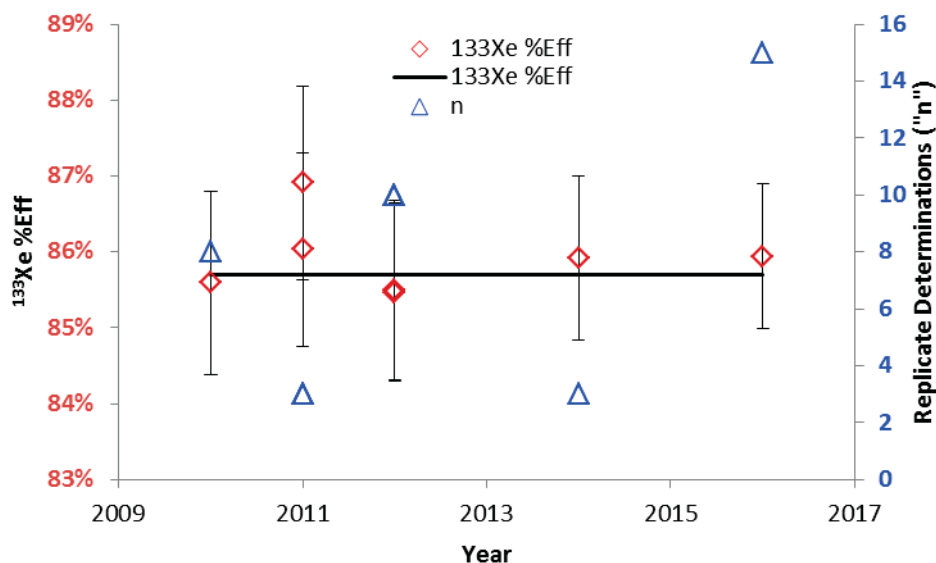


Figure 5. Historical Det2 well detector efficiencies for the Bean geometry. Red diamonds show the efficiency (left axis), and blue triangles indicate the number of replicates averaged to obtain the average efficiency (right axis). In 2011 and 2012 two data points shown because two NIST samples were counted and efficiencies were obtained separately for each sample counted. The average shown as a solid horizontal line was calculated from the weighted average of determinations, weighted by number of replicate counts for each sample.

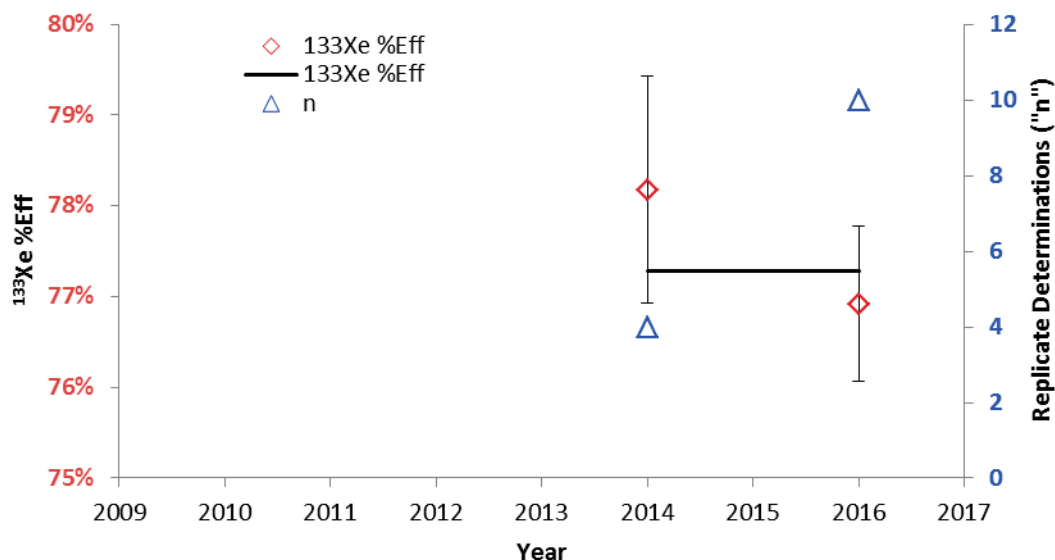


Figure 6. Historical Det3 well detector efficiencies for the Bean geometry. Red diamonds show the efficiency (left axis), and blue triangles indicate the number of replicates averaged to obtain the average efficiency (right axis). The average shown as a solid horizontal line was calculated from the weighted average of determinations, weighted by number of replicate counts for each sample.

Table 3. ^{133}Xe Efficiency comparison of the weighted average from 2014, and the new weighted average that includes the 2016 calibration data.

| | Old Weighted Avg Eff | New Weighted Avg Eff |
|----------------|----------------------|----------------------|
| Det2-Bean-Well | $85.71 \pm 1.19\%$ | $85.77 \pm 1.13\%$ |
| Det3-Bean-Well | $78.18 \pm 1.25\%$ | $77.28\% \pm 0.98\%$ |

DEAD-TIME VS. SAMPLE ACTIVITY MEASUREMENTS

NIST calibration samples are provided with an activity that has ranged from 5.0×10^5 to 2.2×10^6 Bq ^{133}Xe . This activity is much too high to quantify at our historical counting distances. As a result, these samples were often allowed to decay for weeks to months prior to beginning the calibration counts. Because of timing conflicts with the gas standard production work carried out in the noble gas lab, the NIST samples decaying were sometimes allowed to decay too long to collect more than 2 replicates at the desired counting geometries. In an effort to prevent this problem, a sample set of dead-time vs. sample activity was needed to predict when the samples would be useful at different counting distances. During the 2016 NIST calibration, this data was collected for 100, 50, 24, 12, 4 cm, and well counting geometries for Det1, Det2, and Det3. The data collected for the bean counting geometry are shown in Figure 7Figure 12. These figures show that some of the data is linear (see Figure 11Figure 12) and some is not (see Figure 7Figure 10). When the non-linear data is plotted as log-log plots, it becomes nearly linear. When using a linear correlation of the log-log data the dead-time can be estimated from the sample activity within 10%.

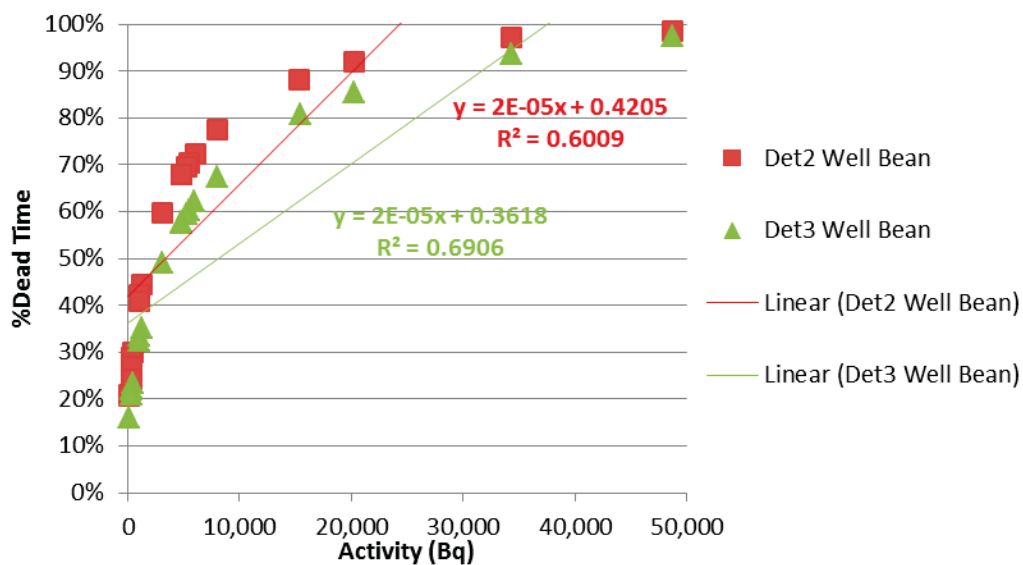


Figure 7. Dead-time vs. Sample Activity for Bean in Well for Det2 and Det3.

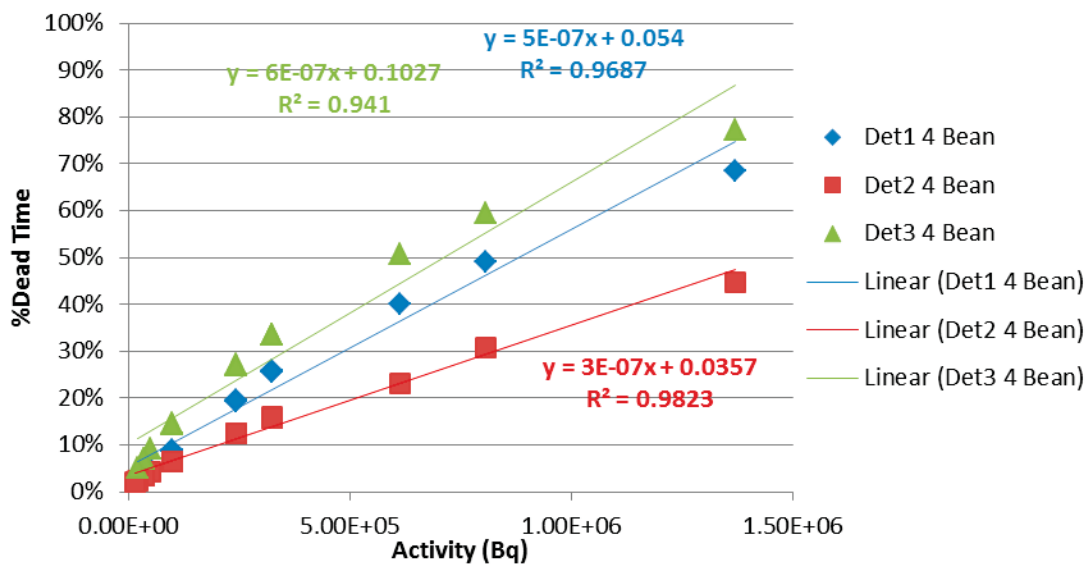


Figure 8. Dead-time vs. Sample Activity for Bean at 4cm for Det1, Det2, and Det3.

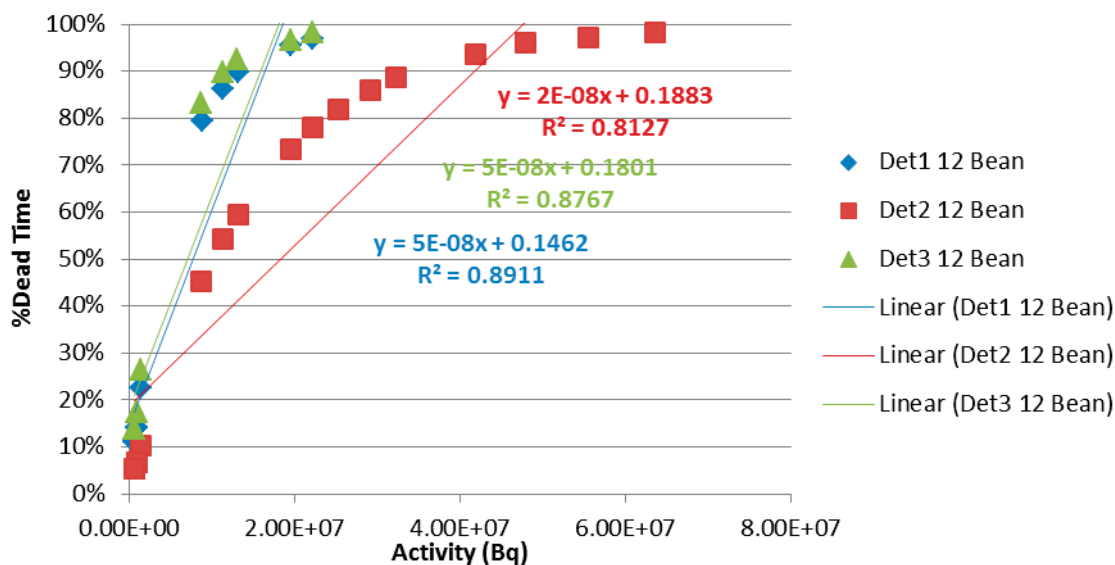


Figure 9. Dead-time vs. Sample Activity for Bean at 12cm Det1, Det2, and Det3.

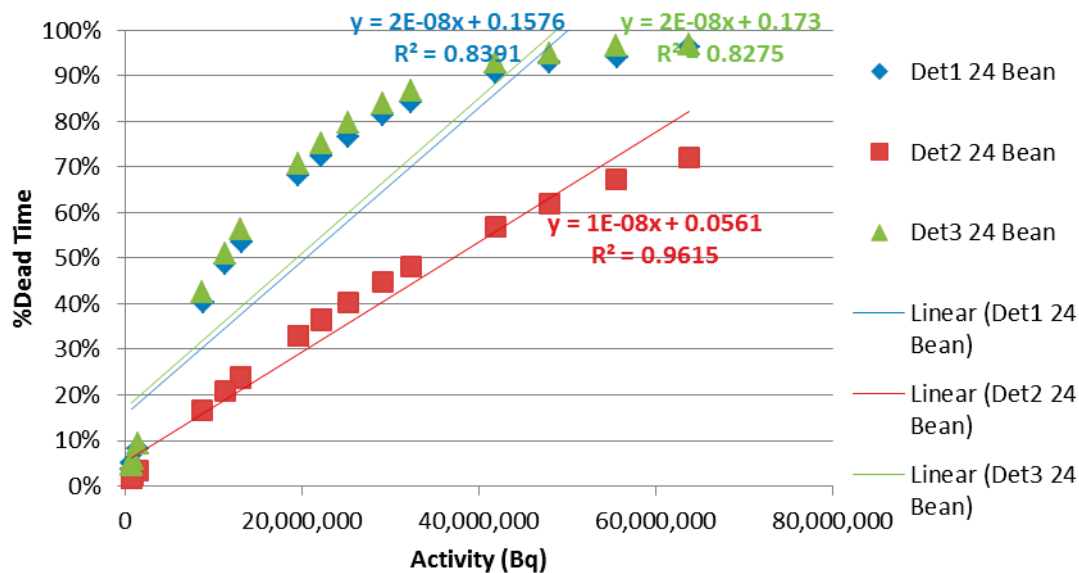


Figure 10. Dead-time vs. Sample Activity for Bean at 24cm for Det1, Det2, and Det3.

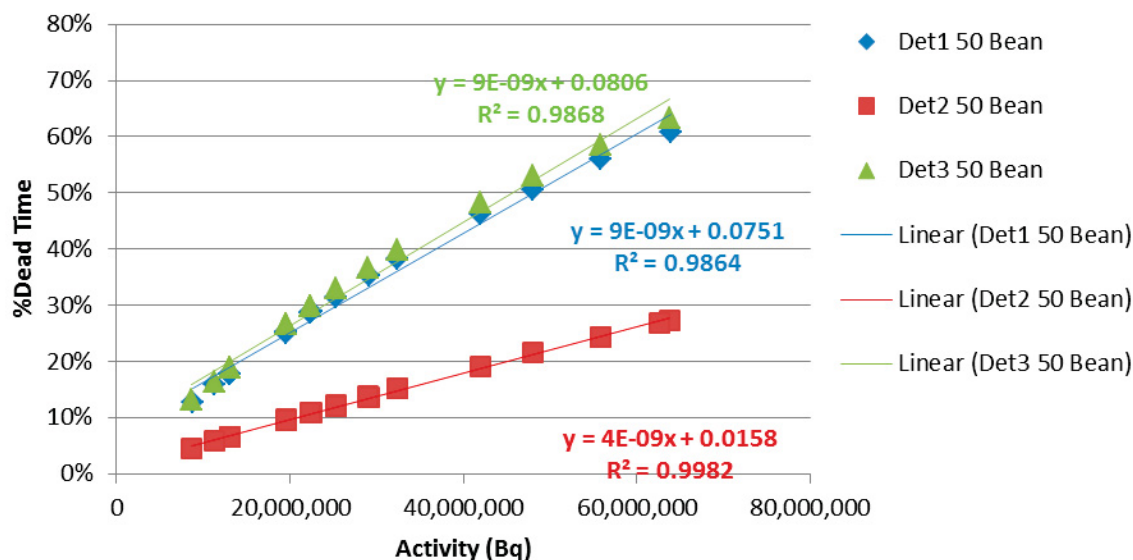


Figure 11. Dead-time vs. Sample Activity for Bean at 50cm Det1, Det2, and Det3.

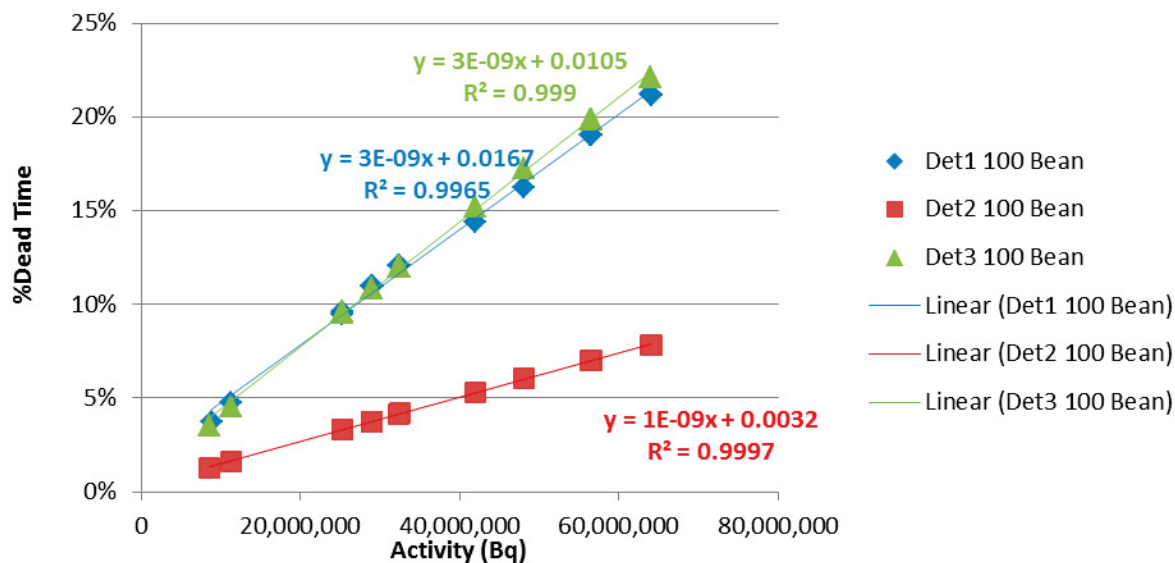


Figure 12. Dead-time vs. Sample Activity for Bean at 100 cm Det1, Det2, and Det3.

These data enable the prediction of the schedule of when to count the samples at each distance and geometry. In the future when NIST samples are received, dead-time estimates can be used to set counting time-lines and set calendar reminders so the decaying samples are not allowed to decay too much. Three examples of the type of time-line prediction for sample use are shown in Figure 13 Figure 15.

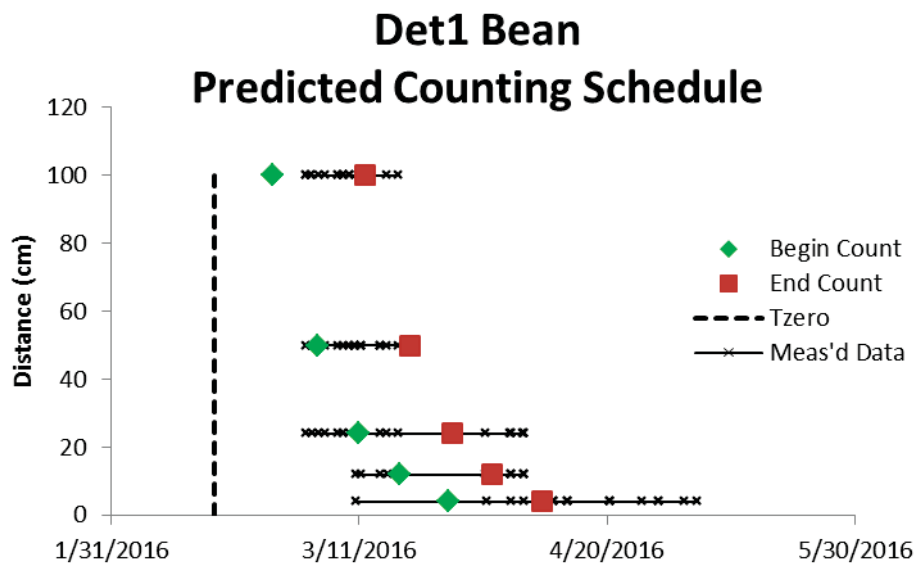


Figure 13. Det1 Bean counting schedule estimated from dead-time measurements. Small x data points are measurements performed during this calibration. Some measurements were taken before the “begin” data point by design to complete the dead-time data set.

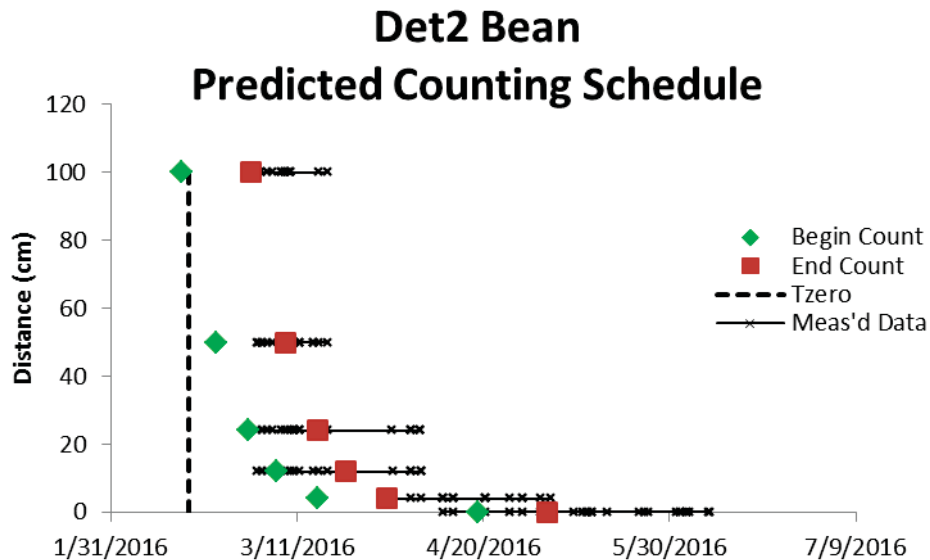


Figure 14. Det2 Bean counting schedule estimated from dead-time measurements. Small x data points are measurements performed during this calibration. Some measurements were taken before the “begin” data point by design to complete the dead-time data set.

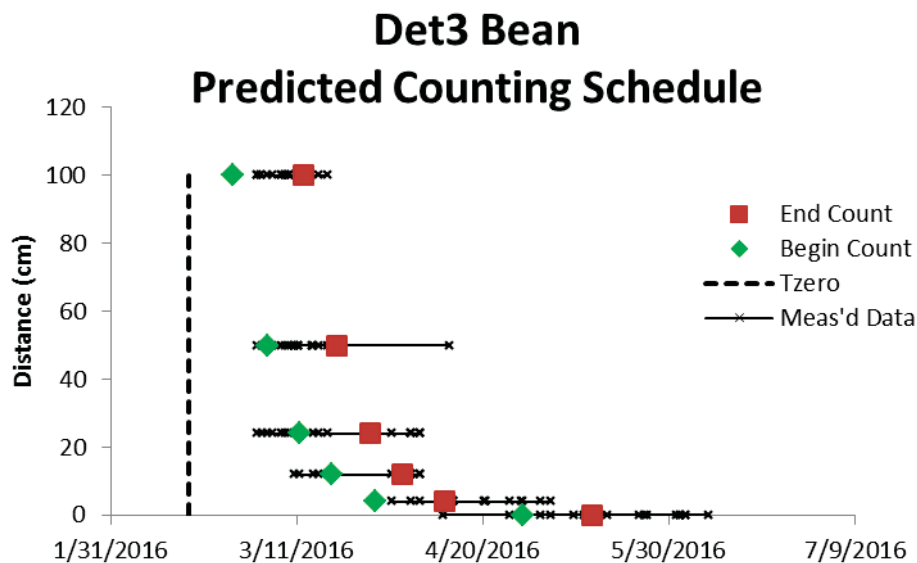


Figure 15. Det3 Bean counting schedule estimated from dead-time measurements. Small x data points are measurements performed during this calibration. Some measurements were taken before the “begin” data point by design to complete the dead-time data set.

TRANSFER CALIBRATION FOR ^{131m}Xe , ^{133m}Xe , AND ^{135}Xe

To maximize the usefulness of the new spacers a series of transfer calibrations were performed to obtain calibration efficiencies for the characteristic gamma energies for ^{131m}Xe , ^{133m}Xe and ^{135}Xe (163, 233, and 249 keV, respectively). The commonly used counting geometries in the INL NGL of bean, 15mL and 50mL Schlenk tube were calibrated. These results were obtained by isolation of a high activity aliquot in the assay container, assay of the activity in the container using a previously calibrated geometry (stacking Marinelli geometry), and then assay of the quantified aliquot using the uncalibrated geometry.

The previously calibrated geometries were created using multi-energy gamma standards that were supplied by Eckert and Ziegler (E&Z) and contained the following isotopes (in order of increasing gamma energy): ^{241}Am , ^{109}Cd , ^{57}Co , ^{139}Ce , ^{203}Hg , ^{113}Sn , ^{85}Sr , ^{137}Cs , ^{88}Y , and ^{60}Co . These isotopes were selected primarily as isotopes that do not exhibit true coincidence gamma emission which complicates spectrum analysis and calculation of the calibration efficiency. The E&Z multi-energy standards are provided as isotopes dissolved in epoxy and solidified in the bean, 15mL and 50 mL Schlenk tube geometries, or as isotopes evaporated on cellophane or Styrofoam beads. The cellophane or Styrofoam bead version of the multi-energy standard is preferable for radioactive xenon quantification due to its lower density giving lower sample induced attenuation than the epoxy variants.

All counts of the ^{131m}Xe , ^{133m}Xe and ^{135}Xe samples contained at least 40,000 counts in the region of interest and no more than 25% dead time for Det1 and Det3 and 60% dead time for Det2. To eliminate the possibility of bias from a single calibrated geometry being used to quantify the known activity for these calibrations, the average activity from assays at three different known geometries were used to quantify each aliquot. The feed activities for this calibration exercise are shown in Table 4.

Table 4. Transfer Calibration activity aliquot quantification information. Notation of (n = “#” x “#”) signifies the (“number of geometries” x “replicates at each geometry”)

| | ^{131m} Xe Activity (Bq) | Quantification Geometry | ^{133m} Xe Activity (Bq) | Quantification Geometry | ¹³⁵ Xe Activity (Bq) | Quantification Geometry |
|-------------------------|--|----------------------------|--|-------------------------|---------------------------------------|----------------------------|
| Bean(1) | 2,160,000 ± 73,000 (n = 3 x 4) | Det1 (4, 12, 24 cm) | 43,000 ± 1,000 (n = 4 x 1) | Det1 (4, 12, 24 cm) | 101,000 ± 2,000 (n = 3 x 2) | Det1 (4, 12, 24 cm) |
| Bean(2) | 80,600 ± 2,800 (n = 3 x 2) | Det1 (4, 12, 24 cm) | | | 62,000 ± 1,000 (n = 3 x 2) | Det1 (4, 12, 24 cm) |
| 15mL Schlenk Tube | 113,000 ± 4,000 (n = 3 x 2) | Det1 (0, 4, 16 cm) | 30,700 ± 700 (n = 3 x 1) | Det1 (0, 4, 16 cm) | 170,000 ± 3,000 (n = 3 x 2) | Det1 (0, 4, 16 cm) |
| 50mL Schlenk Tube | 121,000 ± 4,000 (n = 3 x 2) | Det1 (0, 8, 20 cm) | 34,300 ± 800 (n = 3 x 1) | Det1 (0, 8, 20 cm) | 3,780,000 ± 41,000 (n = 3 x 2) | Det1 (0, 8, 20 cm) |

Table 5. Transfer Calibration Efficiencies determined for $^{131\text{m}}\text{Xe}$ on Det1, Det2, and Det3 in the Bean, 15mL and 50mL Schlenk tube geometries. Due to a lack of activity, and detector availability for longer counts, efficiencies shown in **goldenrod text** are estimated from the reproducible inter-detector ratios (see Figure 16Figure 19). The efficiency shown in **red text** are calculated from counts that contained less than 40,000 counts. Efficiencies for the well, and 0 cm were not measured due to the long half-life of $^{131\text{m}}\text{Xe}$ and not having time to allow the sample to decay.

| $^{131\text{m}}\text{Xe}$ Efficiency with Tube Spacers | | | | | | |
|--|---------------------|----------------|---------------------|----------------|---------------------|----------------|
| Bean | | | | | | |
| Distance (cm) | Det1 Tube Spacer | | Det2 Tube Spacer | | Det3 Tube Spacer | |
| | %Eff | 1 σ unc | %Eff | 1 σ unc | %Eff | 1 σ unc |
| Well | N/A | | 67.9% | $\pm 2.4\%$ | 60.0% | 2.1% |
| 0 | 17.07% | $\pm 0.59\%$ | 8.41% | $\pm 0.29\%$ | 12.11% | $\pm 0.42\%$ |
| 4 | 4.30% | $\pm 0.15\%$ | 1.748% | $\pm 0.06\%$ | 2.68% | $\pm 0.09\%$ |
| 12 | 0.940% | $\pm 0.03\%$ | 0.379% | $\pm 0.01\%$ | 0.589% | $\pm 0.02\%$ |
| 24 | 0.285% | $\pm 0.010\%$ | 0.114% | $\pm 0.004\%$ | 0.181% | $\pm 0.006\%$ |
| 50 | 0.073% | $\pm 0.002\%$ | 0.029% | $\pm 0.001\%$ | 0.045% | $\pm 0.002\%$ |
| 100 | 0.0192% | $\pm 0.0007\%$ | 0.0077% | $\pm 0.0003\%$ | 0.0119% | $\pm 0.0004\%$ |

| $^{131\text{m}}\text{Xe}$ Efficiency with Tube Spacers | | | | | | |
|--|---------------------|----------------|---------------------|----------------|---------------------|----------------|
| 15mL Schlenk Tube | | | | | | |
| Distance (cm) | Det1 Tube Spacer | | Det2 Tube Spacer | | Det3 Tube Spacer | |
| | %Eff | 1 σ unc | %Eff | 1 σ unc | %Eff | 1 σ unc |
| 0 | 8.37% | $\pm 0.29\%$ | 3.113% | $\pm 0.11\%$ | 5.377% | 0.186% |
| 4 | 3.22% | $\pm 0.11\%$ | 1.245% | $\pm 0.04\%$ | 2.050% | 0.071% |
| 12 | 0.839% | $\pm 0.029\%$ | 0.333% | $\pm 0.012\%$ | 0.525% | 0.018% |
| 24 | 0.268% | $\pm 0.009\%$ | 0.107% | $\pm 0.004\%$ | 0.168% | 0.006% |
| 50 | 0.0679% | $\pm 0.002\%$ | 0.0272% | $\pm 0.0009\%$ | 0.042% | 0.001% |
| 100 | 0.0176% | $\pm 0.0006\%$ | 0.00702% | | 0.01104% | |

| $^{131\text{m}}\text{Xe}$ Efficiency with Tube Spacers | | | | | | |
|--|---------------------|----------------|---------------------|----------------|---------------------|----------------|
| 50mL Schlenk Tube | | | | | | |
| Distance (cm) | Det1 Tube Spacer | | Det2 Tube Spacer | | Det3 Tube Spacer | |
| | %Eff | 1 σ unc | %Eff | 1 σ unc | %Eff | 1 σ unc |
| 0 | 6.71% | $\pm 0.23\%$ | 2.371% | $\pm 0.08\%$ | 4.218% | 0.148% |
| 4 | 2.67% | $\pm 0.09\%$ | 1.026% | $\pm 0.04\%$ | 1.701% | 0.060% |
| 12 | 0.743% | $\pm 0.093\%$ | 0.295% | $\pm 0.010\%$ | 0.472% | 0.017% |
| 24 | 0.242% | $\pm 0.009\%$ | 0.097% | $\pm 0.003\%$ | 0.151% | 0.005% |
| 50 | 0.062% | $\pm 0.002\%$ | 0.0249% | $\pm 0.0009\%$ | 0.038% | 0.001% |
| 100 | 0.0159% | $\pm 0.0006\%$ | 0.00629% | | 0.0098% | 0.0003% |

Table 6. Transfer Calibration Efficiencies determined for $^{133\text{m}}\text{Xe}$ on Det1, Det2, and Det3 in the Bean, 15mL and 50mL Schlenk tube geometries. Due to a lack of activity, and detector availability for longer counts, efficiencies shown in **goldenrod text** are estimated from the reproducible inter-detector ratios (see Figure 16Figure 19). Efficiencies shown in **red text** are calculated from counts that contained less than 40,000 counts.

| $^{133\text{m}}\text{Xe}$ Efficiency with Tube Spacers | | | | | | |
|--|---------------------|----------------|---------------------|----------------|---------------------|----------------|
| Bean | | | | | | |
| Distance (cm) | Det1 Tube Spacer | | Det2 Tube Spacer | | Det3 Tube Spacer | |
| | %Eff | 1 σ unc | %Eff | 1 σ unc | %Eff | 1 σ unc |
| Well | N/A | | | | | |
| 0 | 14.609% | $\pm 0.402\%$ | 5.982% | $\pm 0.138\%$ | 8.444% | 0.235% |
| 4 | 3.486% | $\pm 0.081\%$ | 1.422% | $\pm 0.033\%$ | 2.012% | 0.056% |
| 12 | 0.780% | $\pm 0.019\%$ | 0.313% | $\pm 0.008\%$ | 0.451% | 0.013% |
| 24 | 0.242% | $\pm 0.006\%$ | 0.097% | $\pm 0.003\%$ | 0.138% | |
| 50 | 0.0611% | | 0.0248% | $\pm 0.001\%$ | 0.0352% | |
| 100 | 0.0164% | $\pm 0.0004\%$ | 0.00670% | | 0.00944% | |

| $^{133\text{m}}\text{Xe}$ Efficiency with Tube Spacers | | | | | | |
|--|---------------------|----------------|---------------------|----------------|---------------------|----------------|
| 15mL Schlenk Tube | | | | | | |
| Distance (cm) | Det1 Tube Spacer | | Det2 Tube Spacer | | Det3 Tube Spacer | |
| | %Eff | 1 σ unc | %Eff | 1 σ unc | %Eff | 1 σ unc |
| 0 | 6.92% | $\pm 0.154\%$ | 2.492% | $\pm 0.06\%$ | 4.043% | 0.093% |
| 4 | 2.58% | $\pm 0.058\%$ | 1.019% | $\pm 0.02\%$ | 1.528% | 0.035% |
| 12 | 0.69% | $\pm 0.016\%$ | 0.277% | $\pm 0.006\%$ | 0.400% | 0.009% |
| 24 | 0.227% | $\pm 0.006\%$ | 0.0907% | | 0.130% | 0.004% |
| 50 | 0.0577% | | 0.0230% | | 0.034% | 0.001% |
| 100 | 0.0156% | $\pm 0.0004\%$ | 0.00624% | | 0.0092% | |

| $^{133\text{m}}\text{Xe}$ Efficiency with Tube Spacers | | | | | | |
|--|---------------------|----------------|---------------------|----------------|---------------------|----------------|
| 50mL Schlenk Tube | | | | | | |
| Distance (cm) | Det1 Tube Spacer | | Det2 Tube Spacer | | Det3 Tube Spacer | |
| | %Eff | 1 σ unc | %Eff | 1 σ unc | %Eff | 1 σ unc |
| 0 | 5.69% | $\pm 0.13\%$ | 2.025% | $\pm 0.05\%$ | 3.226% | 0.074% |
| 4 | 2.27% | $\pm 0.05\%$ | 0.867% | $\pm 0.02\%$ | 1.317% | 0.030% |
| 12 | 0.642% | $\pm 0.015\%$ | 0.254% | $\pm 0.006\%$ | 0.371% | 0.009% |
| 24 | 0.215% | $\pm 0.009\%$ | 0.0811% | | 0.124% | 0.003% |
| 50 | | | | | | |
| 100 | 0.01398% | $\pm 0.0004\%$ | 0.00538% | | 0.00808% | |

Table 7. Transfer Calibration Efficiencies determined for ^{135}Xe on Det1, Det2, and Det3 in the Bean, 15mL and 50mL Schlenk tube geometries. The efficiency shown in red text are calculated from counts that contained less than 40,000 counts. The 0 cm data was not measured.

| ^{135}Xe Efficiency with Tube Spacers | | | | | | |
|--|---------------------|----------------|---------------------|----------------|---------------------|----------------|
| Bean | | | | | | |
| Distance (cm) | Det1 Tube Spacer | | Det2 Tube Spacer | | Det3 Tube Spacer | |
| | %Eff | 1 σ unc | %Eff | 1 σ unc | %Eff | 1 σ unc |
| Well | N/A | | 41.401% | $\pm 1.53\%$ | 37.75% | 1.53% |
| 0 | | | | | | |
| 4 | 3.40% | $\pm 0.13\%$ | 1.334% | $\pm 0.05\%$ | 1.92% | $\pm 0.07\%$ |
| 12 | 0.769% | $\pm 0.03\%$ | 0.302% | $\pm 0.01\%$ | 0.433% | $\pm 0.02\%$ |
| 24 | 0.238% | $\pm 0.009\%$ | 0.095% | $\pm 0.004\%$ | 0.134% | $\pm 0.005\%$ |
| 50 | 0.062% | $\pm 0.002\%$ | 0.0245% | $\pm 0.0009\%$ | 0.036% | $\pm 0.001\%$ |
| 100 | 0.0162% | $\pm 0.0006\%$ | 0.0067% | $\pm 0.0003\%$ | 0.0090% | $\pm 0.0003\%$ |

| ^{135}Xe Efficiency with Tube Spacers | | | | | | |
|--|---------------------|----------------|---------------------|----------------|---------------------|----------------|
| 15mL Schlenk Tube | | | | | | |
| Distance (cm) | Det1 Tube Spacer | | Det2 Tube Spacer | | Det3 Tube Spacer | |
| | %Eff | 1 σ unc | %Eff | 1 σ unc | %Eff | 1 σ unc |
| 0 | 6.69% | $\pm 0.24\%$ | 2.393% | $\pm 0.09\%$ | 3.785% | $\pm 0.14\%$ |
| 4 | 2.56% | $\pm 0.09\%$ | 0.983% | $\pm 0.04\%$ | 1.465% | $\pm 0.05\%$ |
| 12 | 0.687% | $\pm 0.03\%$ | 0.275% | $\pm 0.01\%$ | 0.390% | $\pm 0.01\%$ |
| 24 | 0.225% | $\pm 0.008\%$ | 0.090% | $\pm 0.003\%$ | 0.128% | $\pm 0.005\%$ |
| 50 | 0.059% | $\pm 0.002\%$ | 0.024% | $\pm 0.001\%$ | 0.033% | $\pm 0.001\%$ |
| 100 | 0.0154% | $\pm 0.0006\%$ | 0.0061% | $\pm 0.0002\%$ | 0.0088% | $\pm 0.0003\%$ |

| ^{135}Xe Efficiency with Tube Spacers | | | | | | |
|--|---------------------|----------------|---------------------|----------------|---------------------|----------------|
| 50mL Schlenk Tube | | | | | | |
| Distance (cm) | Det1 Tube Spacer | | Det2 Tube Spacer | | Det3 Tube Spacer | |
| | %Eff | 1 σ unc | %Eff | 1 σ unc | %Eff | 1 σ unc |
| 0 | 5.47% | $\pm 0.21\%$ | 1.916% | $\pm 0.07\%$ | 3.034% | $\pm 0.11\%$ |
| 4 | 2.19% | $\pm 0.08\%$ | 0.827% | $\pm 0.03\%$ | 1.244% | $\pm 0.05\%$ |
| 12 | 0.613% | $\pm 0.02\%$ | 0.244% | $\pm 0.01\%$ | 0.348% | $\pm 0.01\%$ |
| 24 | 0.205% | $\pm 0.008\%$ | 0.082% | $\pm 0.003\%$ | 0.116% | $\pm 0.004\%$ |
| 50 | 0.054% | $\pm 0.002\%$ | 0.0214% | $\pm 0.0008\%$ | 0.030% | $\pm 0.001\%$ |
| 100 | 0.0139% | $\pm 0.0005\%$ | 0.0055% | $\pm 0.0002\%$ | 0.0079% | $\pm 0.0003\%$ |

Table 8. Simulated gas multi-energy gamma calibrations using the new sample spacers. The ‘x’ indicates that the distance and geometry was calibrated using the multi-energy standard. A calibration using a multi-energy standard will give you an interpolated efficiency at any energy across the gamma spectrum.

| Multi-Energy Calibration | | | |
|--------------------------|---------------------|---------------------|---------------------|
| Bean (97510) | | | |
| | Det1 Tube Spacer | Det2 Tube Spacer | Det3 Tube Spacer |
| Well | | x | x |
| 0 | x | x | x |
| 4 | x | x | x |
| 12 | x | x | x |
| 24 | x | x | x |
| 50 | x | x | x |
| 100 | | | |

| 15mL Stube (97511B) | | | |
|---------------------|---------------------|---------------------|---------------------|
| | Det1 Tube Spacer | Det2 Tube Spacer | Det3 Tube Spacer |
| 0 | x | x | x |
| 4 | x | x | x |
| 12 | x | x | x |
| 24 | x | x | x |
| 50 | x | x | x |
| 100 | | | |

| 50mL Stube (97512A) | | | |
|---------------------|---------------------|---------------------|---------------------|
| | Det1 Tube Spacer | Det2 Tube Spacer | Det3 Tube Spacer |
| 0 | x | x | x |
| 4 | x | x | x |
| 12 | x | x | x |
| 24 | x | x | x |
| 50 | x | x | x |
| 100 | | | |

DETECTOR EFFICIENCY RATIO

It was observed during the transfer calibration that the efficiencies measured made very reproducible ratios between Det1, Det2 and Det3. These ratios did not hold up at close distance (0 cm and 4 cm) probably due to the relative performance differences caused by the detector shapes between Det1 (planar) and Det2 and Det3 (well detectors). It appears that the ratio observed at 24 cm is a good estimate for how the detector ratios will appear at greater distances from the detector (see Figure 16/19).

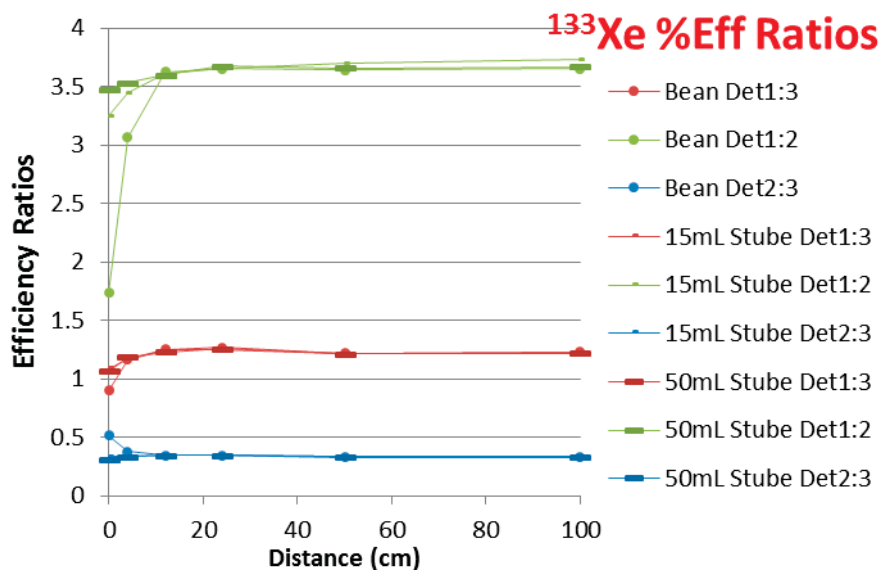


Figure 16. Calibration Efficiency ratios observed between Det1, Det2 and Det3 for ^{133}Xe .

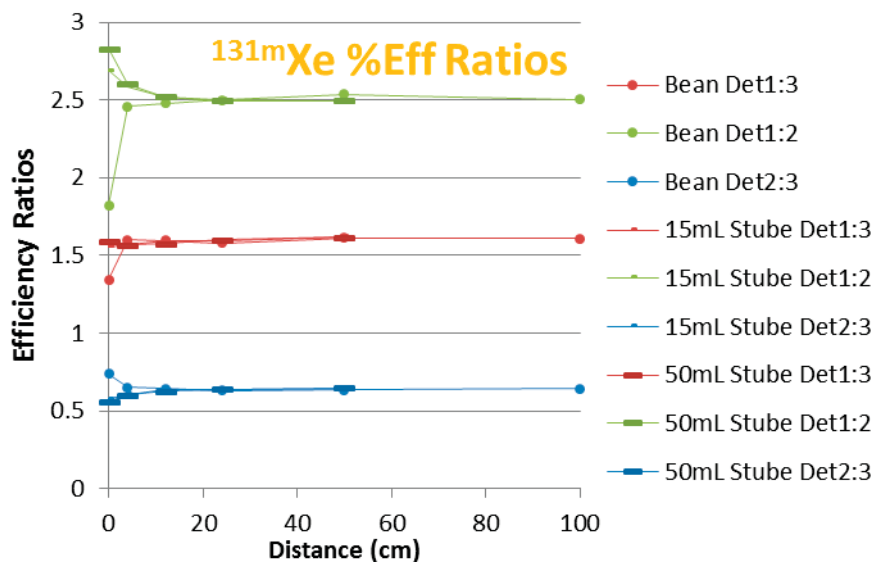


Figure 17. Calibration Efficiency ratios observed between Det1, Det2 and Det3 for ^{131m}Xe .

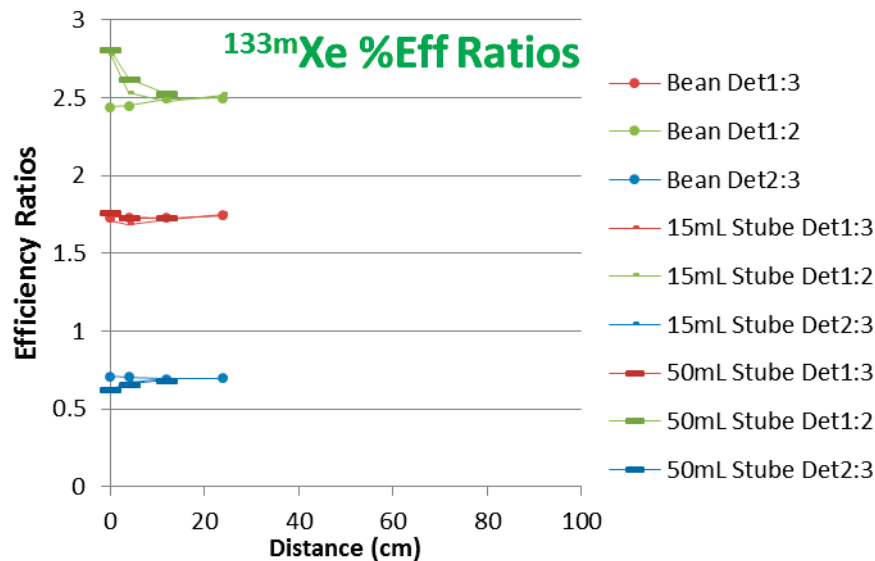


Figure 18. Calibration Efficiency ratios observed between Det1, Det2 and Det3 for ^{133m}Xe .

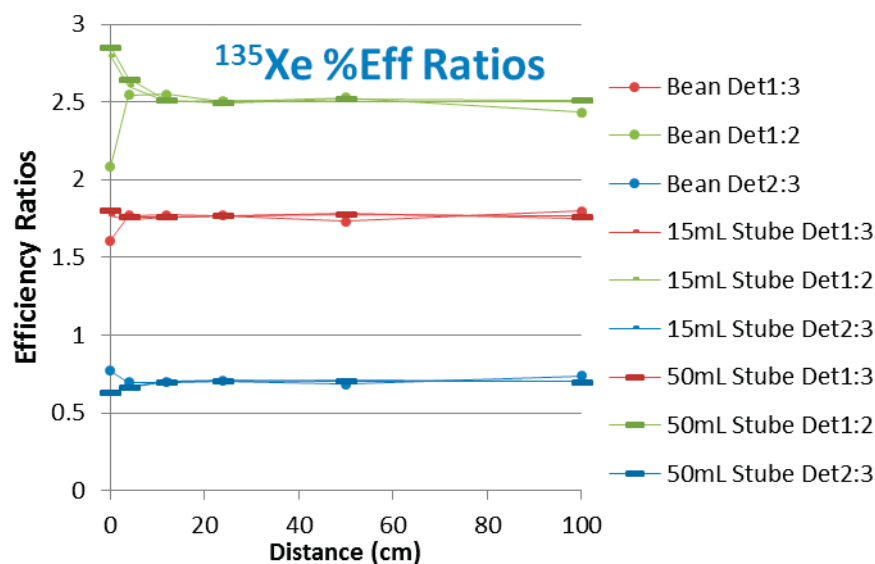


Figure 19. Calibration Efficiency ratios observed between Det1, Det2 and Det3 for ^{135}Xe .

There were instances where obtaining a count at extreme distances was difficult given the constraints of detector availability, available activity quantities, and decay branching ratios. Because of these shortcomings we used the detector efficiency ratios to estimate the efficiencies that there was not time and activity to collect. These efficiencies are shown in **goldenrod text** and have no uncertainty related to them (see Table 5Table 7). Calibration of ^{133m}Xe was particularly difficult because of the relatively short half-life (2.19 days), branching ratio (10%), and difficulty procuring large quantities of this isotope

(typically only 15% of total activity from a ^{132}Xe irradiated target results in $^{133\text{m}}\text{Xe}$). Many of these efficiencies had to be estimated at great distances from the detector, but it is not anticipated that these geometries will be needed, due to the difficulty of procuring large quantities of this isotope. The 24 cm distance was sufficiently calibrated, and we anticipate that this will be more than sufficient for the program's needs.

CONCLUSION

The Noble Gas Laboratory at INL has collected the data that enables the prediction of when high activity NIST ^{133}Xe calibration samples can be most effectively used based on the activity and reference date of the standard. All three detectors have been calibrated at distances of well, 0, 4, 12, 24, 50 and 100 cm in the bean, 15mL and 50 mL Schlenk tube geometries. This is a larger set of calibrated geometries than was previously available and will reduce the chance of counting bottlenecks that may occur during standard production involving short-lived isotopes. For isotopes that are difficult to procure in large quantities, or decay too quickly, a method of estimating the counting efficiency of one detector based on an observed relative performance ratio between detectors enables estimating the counting efficiency when the ability to measure the efficiency is not available. This is mainly useful at counting distances greater than or equal to 12 cm. The running average counting efficiency for Det2 and Det3 was established and updated. Future calibrations will be rolled into an updated running counting efficiency for the well detector counting efficiency.

REFERENCES

1. "American National Standard for Calibration and Use of Germanium Spectrometers for the Measurement of Gamma-Ray Emission Rates of Radionuclides" **ANSI N42.14-1999 (R2004)**