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Diverse Active Well Neutron Coincidence Counter Utility at the Savannah River National Laboratory

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ABSTRACT

In this paper we describe use of the Aquila active well neutron coincidence counter for nuclear material assays of ^{235}U in multiple analytical techniques at Savannah River Site (SRS), at the Savannah River National Laboratory (SRNL), and at Argonne West National Laboratory (AWNL). The uses include as a portable passive neutron counter for field measurements searching for evidence of ^{252}Cf deposits and storage; as a portable active neutron counter using an external activation source for field measurements searching for trace ^{235}U deposits and holdup; for verification measurements of U-Al reactor fuel elements; for verification measurements of uranium metal; and for verification measurements of process waste of impure uranium in a challenging cement matrix. The wide variety of uses described demonstrate utility of the technique for neutron coincidence verification measurements over the dynamic ranges of 100 g – 5000 g for U metal, 200 g – 1300 g for U-Al, and 8 g – 35 g for process waste. In addition to demonstrating use of the instrument in both the passive and active modes, we also demonstrate its use in both the fast and thermal neutron modes.

INTRODUCTION

In this paper we describe testing, efficiency calibration, and use of the Aquila[1] active well neutron coincidence counter (AWCC) for nuclear material assays and surveys. The instrument was brought to the Savannah River Site (SRS) in 1998 by a Brookhaven National Laboratory group for testing of neutron multiplicity algorithms.[2] A separate group of Savannah River National Laboratory (SRNL) researchers recognized the applicability of the instrument for assay of enriched U-Al fuel ingots at SRS K-Area nuclear material storage facility (KAMS). We set up the instrument using Los Alamos National Laboratory INCC software[3] to perform verification measurements of U-Al in the 1999 annual Department of Energy (DOE) inventory of reactor fuel ingots stored at SRS. Startup of the instrument using the INCC software, efficiency calibration, and the 1999 verification measurements are described thoroughly in reference 4. The instrument has been used subsequently for these annual DOE inventory verification measurements in 2000 – 2006 and will continue to be used for the foreseeable future.

In addition to the KAMS U-Al verification measurements, the authors have discovered and devised multiple other applications for the active neutron coincidence counting technique. These involve uses in the active and passive acquisition modes and in the thermal and fast fission acquisition modes, uses as a portable neutron survey instrument, and utility for verification measurements of three distinct types of highly enrich uranium over three diverse dynamic ranges. Specifically we have performed experimental programs that include

- Verification measurements of U-Al fuel ingots in the fast fission neutron coincidence counting mode with a dynamic range of 100 – 1300 g.

- Verification measurements of U metal at Argonne West National Laboratory in the fast fission neutron coincidence counting mode with a dynamic range of 100 – 5000g.
- At-line verification measurements of U process waste in the HB-Line facility at SRS in the thermal fission neutron coincidence counting mode with a dynamic range of 5 – 35 g.
- Evaluation of neutron background and personnel exposure concerns in the deactivated Californium Production facility at SRNL using the AWCC as a passive survey instrument.
- Evaluation of criticality concerns in the decommissioned Naval Fuels facility using an external activation source to adapt the instrument as an active survey instrument.
- Passive neutron multiplicity verification measurements of Pu and mixed U/Pu items.

We describe the experimental programs for each of these measurements in detail in the **EXPERIMENTAL** section of this paper. We further describe plans to use this instrument in DOE Office of Security development work for future non-proliferation programs in the DOE, International Atomic Energy (IAEA), and Department of Homeland Security.[5-10]

EXPERIMENTAL

The Aquila AWCC uses two Am-Li(α,n) neutron sources to activate thermal fission in the samples. The AWCC instrument is shown on its side in the photograph of Figure 1. A photograph of the Am-Li activation sources and of one of the polyethylene moderators is shown in Figure 2. The two sources have ^{241}Am α-activities of 4.4×10^{10} dps and yield (α,n) neutron rates of approximately 50000 n/sec. With no sample in place, these sources yield an active background of approximately 6537 neutron singles events/sec (eps) in the fast fission mode and an active coincidence background of (< 0.1 eps). Thus the instrumental singles efficiency is approximately 7% for the activation sources, with a corrected random coincidence rate of $<2 \times 10^{-5}$. With the sources placed in the center of the sample cavity the detection efficiency was measured to be 25.3(3)%[11]. The latter efficiency of 25% is our best measure of sample neutron singles efficiency. We discuss the random coincidence correction, which is a very important part of neutron coincidence measurements, below.

Stability in the active background is a very important feature for successful operation of this instrument. In our initial set up back in 1999 we observed over a 112-day period a one sigma standard deviation of 35 cps in the active singles background, and a one sigma standard deviation of 0.8 eps in the active doubles background. These variances were obtained from five background counts taken fairly evenly spaced over the 112-day period. The passive neutron background (obtained with no Am-Li activation sources) was always < 1 cps. These background rates compare favorably with those taken by Cowgill and Lu.[1]

Active well coincidence neutron counting is a well known technique for assay of ^{235}U content in solid samples and has been described previously.[3,12,13] Details for this instrument are described in references 1 and 4. The instrument used in the work

described here is the extended cavity Aquila Technologies serial number KCC-51-DEV well counter. It contains an assembly of 42 ^{3}He detector tubes mounted in two concentric

Figure 1. Photograph of SRS K-Area active well coincidence counter lying on its side showing the sample chamber and neutron moderators.

Figure 2. Photograph of one of the Am-Li activation sources and neutron moderator.

circles in polyethylene moderator blocks. The neutron signals are summed using a PSR-B shift register coincidence counting circuit with the detector parameters shown in Table 1. For all of the work described in this paper the instrument was operated with the Los Alamos National Laboratory INCC neutron coincidence software program.[3] This program is used for set up, operation, data acquisition, and data analysis to relate detected coincidence events to ^{235}U mass. The program also triggers printing of hardcopy output. Examples are included below.

Table 1. Detector parameters and timing characteristics used for this work.

Parameter	Setting
Gate width	64.0 μsec
Predelay time	4.5 μsec
High voltage	1680 V
Die-away time	52.36 μsec
Efficiency	0.25
Deadtime coefficient A	0.893×10^{-6}
Deadtime coefficient B	0.201×10^{-12}

U-Al Fuel Ingot Verification Measurements at SRS

Our initial use of the Aquila instrument was for the annual DOE inventory of U-Al fuel elements that are stored at the SRS K-Area Material Storage facility (KAMS). These fuel elements were all manufactured at the SRS M-Area fuel fabrication facility. Though the existing fuel is far from virgin material, the M-Area specifications[14] required that it be

very pure of fission product and plutonium components. Because the SRS fuel is pure of plutonium, it has a very low passive neutron emission rate and is very well suited for assay of the ^{235}U content by active well induced fission neutron counting. Even though the fission product β–γ activity is very low, assay of ^{235}U by γ-PHA is not suitable because the fuel elements are dense enough to approach infinite thickness for the low energy 186-keV γ-ray from ^{235}U.

We performed an initial set of measurements and activities in the Savannah River Site (SRS) K reactor assembly area. We conducted two separate calibrations of active doubles response versus known mass of ^{235}U using U-Al fuel ingots. The ^{235}U masses varied over the range 247 g to 1190 g. Background counts were taken with the AWCC cavity empty as opposed to using a blank ingot. The two response calibration curves were taken fifty-four days apart. We show data from both in this paper to provide an example of the instrument stability. The data we show below demonstrate excellent instrumental stability.

Data for the first calibration at SRS were taken using six 100-second counts for every standard. All of the doubles response versus known ^{235}U are shown in Table 2. The data are plotted in Figure 3. The solid curve is the best quadratic fit to the calibration symbols with the curve forced through the origin. The equation for the calibration curve is

$$\text{Doubles(cps)} = 0.198m - 0.0000575m^2, \quad (1)$$

where m is known mass in grams of ^{235}U. The calibration data of Table 2 include $^{235}U/^{236}U$ enrichment ratios, which vary over the range 1.39 to 3.95. These data demonstrate the independence of the method from this parameter.

Table 2. ^{235}U calibration measurements taken with the K-Area KCC-51-DEV active well coincidence counter using 100 second counting times and automatic background subtraction and random coincidence subtraction as described in text.

Standard	known mass (grams)	singles rate (cps)	doubles rate (events/sec)	triples rate (events/sec)	$\frac{^{235}U}{^{236}U}$
031	617	558.7	97.19±5.43	Not observed	3.93
023	1190	921.5	164.0±10.0	19.6±2.0	1.94
011	754	725.5	86.01±6.47	13.8±3.0	1.94
051	517	488.2	115.0±4.5	Not observed	1.39
032	640	543.0	105.8±10.2	Not observed	3.95
026	247	277.4	35.17±11.52	5.5±1.5	1.94
054	493	548.6	84.47±4.38	7.1±2.9	1.39
043	666	553.1	105.4±11.8	13.2±2.9	1.95

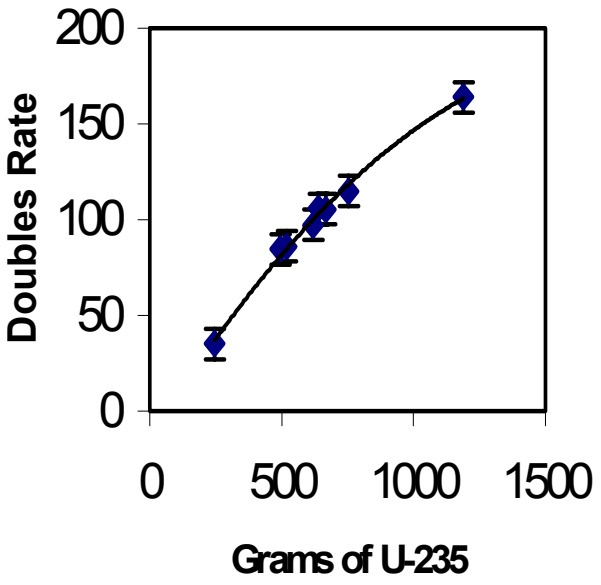

Figure 3. Fit of SRS calibration data of equation (1).

Note in Table 2 that the uncertainty in the doubles rate for the 247 gram standard is almost 33% for this short 100-second counting time curve. For the second acquisition of calibration data we were interested in reducing that uncertainty as well as demonstrating the stability of the calibration curve after the instrument had been powered off for a long time period. To demonstrate both of these objectives we acquired a new calibration curve using five of the same calibration standards spanning the same range of mass. This time we acquired data on the 247-g and 1190-g standards for six 300-second counts, and we acquired the data for the other three standards for the six 100-second counts. These data were acquired 54 days after the calibration data of Figure 3, where the instrument was powered off for the intervening duration.

The second set of calibration data are plotted in Figure 4 along with the best fit calibration curve. Note we obtain much better precision for the 247-g standard. The equation for the curve is

$$\text{Doubles(cps)} = 0.188m - 0.0000434m^2. \quad (2)$$

Equations (1) and (2) are very similar in shape and agree within experimental uncertainty over the entire calibration range. Equation (2) has a positive first derivative all the way out to masses greater than 4 kg. The excellent agreement between the two curves demonstrates the good stability of the instrument.

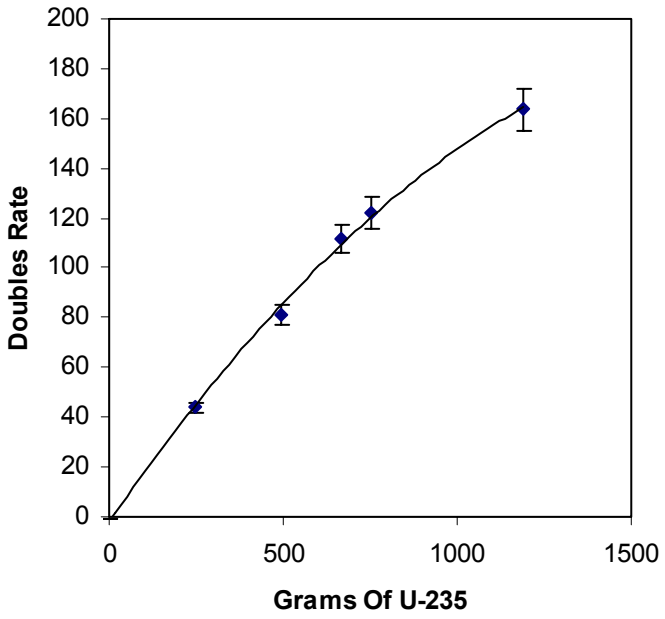

Figure 4. Fit of SRS calibration data of equation (2).

We have used this instrument for the verification measurements required for the 1999 – 2006 DOE inventory of U-Al fuel ingots stored in the K-reactor Assembly area at SRS. The instrument has been very stable over this period of six years. We were able to use the efficiency calibration curve of equation (2) for the 1999 – 2001 verifications. It was necessary to recalibrate in 2002 and again in 2004 as the background singles and doubles rates had changed significantly in K-Assembly. In Table 3 we show the measured active backgrounds and active doubles rates for the 1190-g and 247-g standards for the 1999, 2002, and 2004 calibrations. The shape of the calibration curves never changed significantly from those of equations (1) and (2).

Table 3. Comparison of 1999, 2002, and 2004 calibration data.

	1999	2002	2004
Background singles	5555	6567±4	6797±4
1190 doubles	164±10	169±5	175±4
247 doubles	35±12	43±2	46±2

The 1999 – 2006 verification measurements are plotted in Figure 5. The best linear fit of the data in Figure 5 has a slope of 1.0037, which demonstrates excellent correlation of measured doubles rate with U-235 mass. These data demonstrate very good analytical performance characteristics for the active well counter for use in assay of U-Al fuel ingots.

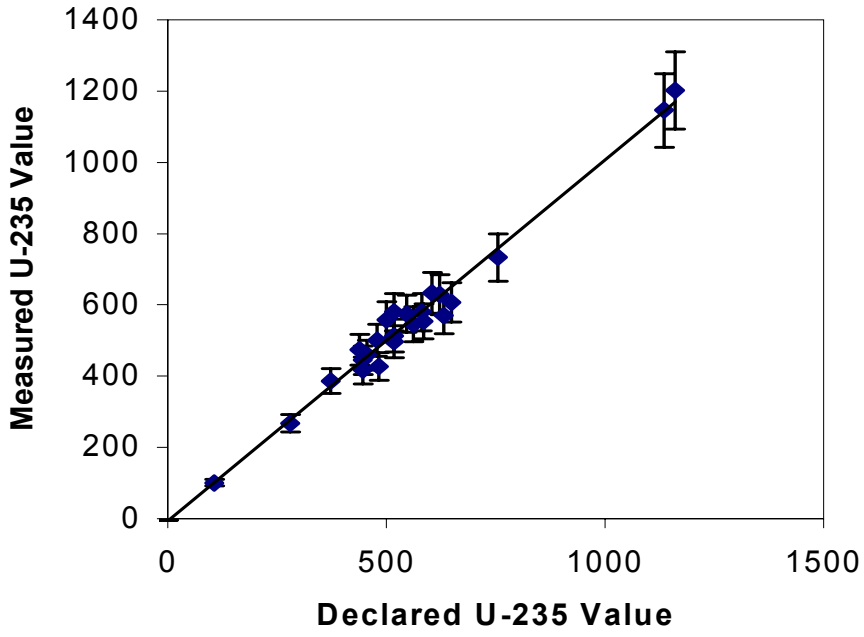

Figure 5. Measured twenty-six point verification data acquired for instrument qualification.

We have already noted the independence of this measurement technique from the $^{235}U/^{236}U$ ratio. In our verification acquisitions of 1999 – 2005 we have observed the declared enrichment for each of the items. The total uranium masses varied over almost a factor of 1.5. The ratio of declared ^{235}U mass to total mass varied over the range 0.082 to 0.14. The capability of the instrument to perform these U-Al verifications appears to be independent of ^{235}U enrichment.

Argonne West Metalic U Measurements

In May of 1999 we conducted an extensive set of data acquisitions at Argonne West National Laboratory in Idaho Falls. These experiments were conducted mainly to test a Brookhaven National Laboratory multiplicity algorithm, but were also very valuable to test the instrument's ^{235}U coincidence assay capabilities at higher masses and coincidence rates. In these experiments we conducted important acquisitions to test the passive coincidence rates contributed by samples up to 5 kg in ^{235}U mass and up to 35g in ^{236}U mass and 1460 g in ^{238}U mass. We also observed the effect of (α, n) reactions and neutron multiplication on the singles rate and coincidence rate. In the Argonne West experiments we used pure, metallic U samples with 78% enrichment.

The acquisitions at Argonne West (ANL) were performed by starting with a sample that contained 5.098 kg of ^{235}U and then removing an aliquot of the sample as we progressed from one acquisition to the next. Therefore each sample counted represented identically a portion of the previous sample. This is opposed to the SRS measurements that were made on completely distinct samples each time. For each ANL acquisition we obtained multiple counts (generally ten) of duration varying from 60 seconds up to 3600 seconds. Data were acquired using the fast fission counting parameters of Table 1.

The data acquired are summarized in Table 4. The $^{235,236,238}U$ masses were determined gravimetrically and calculated from the known stoichiometry. The instrument singles, doubles, and triples responses are shown in the table, including two active background counts. In Table 5 we have listed the singles and doubles response for three passive acquisitions. The data of Table 5 were acquired with the Am-Li activation sources removed from the instrument. Note that the passive singles background is very nearly independent of sample mass and that the doubles and triples rates are only very slowly varying.

The doubles data of Table 4 are plotted in Figure 6. The coincidence data are fit with the quadratic curve

$$\text{Doubles(cps)} = 1.8 + 0.109m + 0.0000004m^2. \quad (3)$$

The extremely good linearity of equation (3) demonstrates that the instrument response is a reliable measure of ^{235}U mass over the entire range 0 to 5 kg. That is, second order non-linear effects like detector deadtime, neutron absorption, passive neutron production by (α,n) or spontaneous fission, and neutron multiplication are not significant factors. We note that the best fit quadratic coefficient is positive in equation (3), which might indicate neutron multiplication begins to contribute at the high masses.

Neutron Multipication

Neutron multiplication can cause a significant contribution to neutron multiplicity assays of Pu metal items. We have observed that multiplication is a very important consideration for the assay of receipted metallic Pu items at the SRS KAMS facility.[15] Modeling multiplication correctly is very important for passive multiplicity assays of Pu and for active multiplicity assays of U[16,17], so we observed our data closely for indication of such effects in the high mass region of the metallic U active coincidence assays. We are almost without doubt observing limited multiplication at ANL with the triples to doubles ratios that we discuss below. However we believe that multiplication is not a significant contributor to the overall coincidence rates observed and that multiplication is clearly not a dominant factor in our acquisitions. Multiplication, like random coincidences, would tend to make the doubles rates bend upward from linearity. Contrary to the shape of the SRS coincidence curves of Figures 3 and 4, the ANL coincidence data of Figure 6 bends very slightly upward, perhaps indicating minor contributions to multiplication at the high mass end of the curve.

Table 4. Measured neutron singles, doubles, and triples rates in events/sec for fifteen active data acquisitions obtained at Argonne West Laboratory. The samples are identified by ^{235}U mass. The ^{238}U masses are also tabulated. All enrichments are 78% ^{235}U. The singles rates do not include background subtraction. The active singles background was 6900(200) events/second.

^{235}U Mass (g)	^{238}U Mass (g)	$^{234,236}U$ Mass (g)	singles rate (events/sec)	Doubles rate (events/sec)	Triples rate (events/sec)
5098	1360	86	8302±4	575±5	221±3
4490	1198	76	8222±4	503±5	181±3
3991	1064	57	8128±4	446±4	142±3
3482	929	59	8009±4	377±4	109±3
2842	758	48	8038±4	325±4	86±2
2993	798	51	7897±4	329±4	93±2
2512	670	42	7796±4	275±4	69±2
2501	667	42	7823±4	279±4	71±2
2019	539	34	7660±3	225±3	52±2
1501	400	25	7476±3	172±3	35±1
993	265	17	7261±2	113±2	20±1
493	131	6	7245±1	54±1	8.8±0.6
260	69	4	7192.4±0.4	30.5±0.4	4.3±0.2
BG1	0	0	7093±4	-3±4	-3±2
BG2	0	0	6759±4	1±3	-3±2

Table 5. Measured singles and doubles response data acquired for four passive counts. These data were acquired with the activation sources removed from the instrument.

^{235}U Mass (g)	**Singles (cps)**	**Doubles (cps)**	**Triples (cps)**
Background	852±2	0.5±0.09	-0.05±0.09
493	849.3±0.1	0.30±0.04	0.080±0.008
2842	859±2	1.6±0.6	0.4±0.1
2842 2nd	859±1	1.6±0.4	2.2±0.4

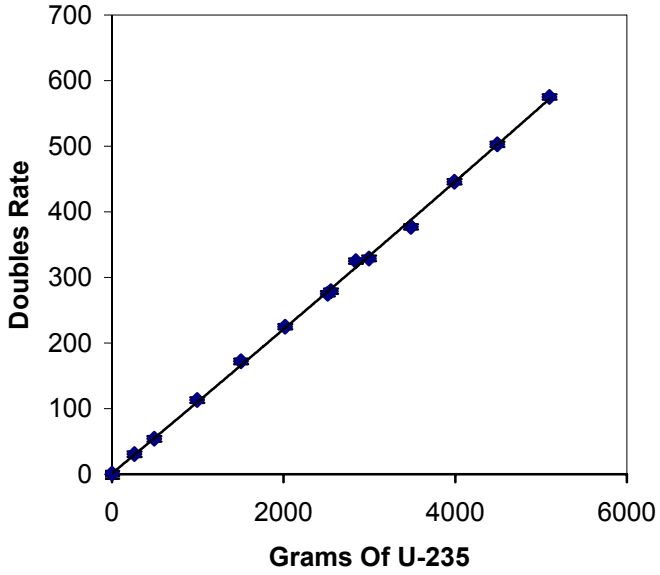

Figure 6. Neutron doubles rate data plotted versus U-235 mass for the data acquired at AWNL.

Ratio of Triples to Doubles

We finally investigate the ratio of triples events to doubles events in the SRS U-Al measurements and ARL metallic U measurements. That ratio is plotted in Figure 7 for the ANL and in Figure 8 for the SRS data. Note at SRS the ratio T(riples)/D(oubles) is nearly flat, equal to 0.11, and independent of sample mass. At ANL the data are clearly related to sample ^{235}U mass. The ratio T/D at 5098 grams is 0.38(1) and still rising. The ANL T/D data are in good agreement with data acquired by Ensslin at the Oak Ridge Y-12 plant.[18]

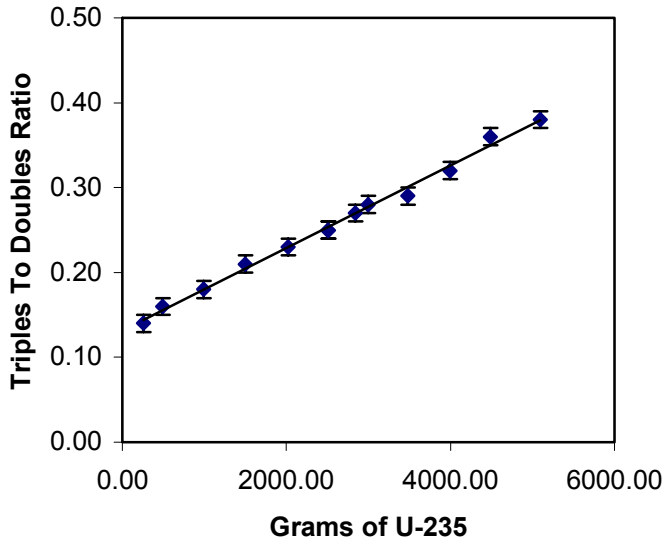

Figure 7. Ratios of observed triples to observed doubles acquired in the ANL experiments.

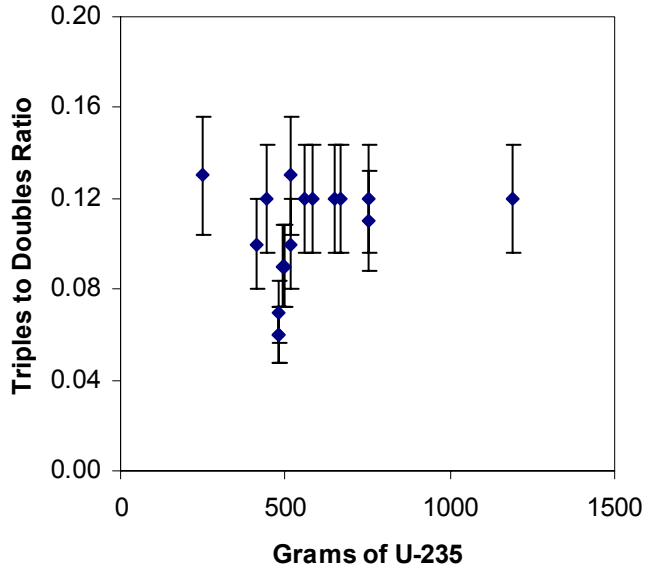

Figure 8. Ratios of observed triples to observed doubles acquired in the SRS experiments.

Since the neutron multiplicity for ^{235}U fission is 2.41, neutron multiplication can cause the T/D ratio to climb with increasing fission (increasing mass). It is not surprising that this effect is observable at ANL for masses ranging up to 5 kg with 78% enrichment and not observable at SRS for mass of only 3kg with only 14% enrichment. The increasing T/D rate at ANL is undoubtedly affected by neutron multiplication, and it is slightly influencing measured doubles rates. However, over the mass range of samples we measured, neutron multiplication has only a very slight effect. Another factor is the simple multiplicity of ^{235}U fission. With increasing fission rate, the probability of detecting a true triple coincidence goes up. The measured T/D ratios clearly indicate to us that active multiplicity analysis becomes an important consideration for highly enriched U metal samples, and we are developing that technique of analysis at our facility.

Trace Uranium Verifications in Desicooler Material at the SRS HB-Line Facility

In September of 2003 we used the Aquila active well counter for verification measurements of enriched uranium in a concrete matrix at the SRS HB-Line Facility. These measurements and the sequence of events from which the requirements evolved are described in reference 19. In this set of at-line acquisitions we used the instrument in the thermal fission mode to perform verification measurements in a challenging concrete matrix that was in the early stages of drying. In these acquisitions the dynamic range of ^{235}U content was 8 – 35 g, which is far below the range of the fast fission measurements described above. A further complication was the young age of the concrete matrix, which yielded variable moisture content and thus variable moderation.

The HB-Line facility was involved in a campaign to dispose of 4.3 kg of FB-Line Desicooler material (HEU oxides). Facility Operations planned to mix the material with Portland cement and de-ionized water to create a waste form that meets the Attractiveness Level E criterion for "SNM micro-encapsulated in refractory compounds or in solid-dilution". To meet these requirements the material was repackaged into 172 one-gallon paint cans. Each paint can would contain approximately 3 kg of cement waste and 8 – 40 g of ^{235}U. The DOE-SR approval directed that the discard campaign use the FB-Line accountability shippers values and that HB-Line Facility and the Savannah River Material Protection and Accountability group provide verification measurements of the solid waste product. At the time of planning, the HB-Line facility was not able to provide the required verification measurements with any available assay equipment. The Analytical Development Section (ADS) of the SRNL recommended use of the ADS active well coincidence counter to provide the required measurements at-line on-going with the production campaign.

We transported the Aquila AWCC to the HB-Line facility and set up the counter inside the contamination area of the facility operations. The personal computer and INCC 5.02 acquisition and analysis software were set up remotely outside of the contamination area as shown in the photograph of Figure 9. A photograph of the at-line use of the instrument

is shown in Figure 10. After each paint can preparation and a period of two hours for drying, the can was introduced directly into the AWCC cavity for a verification measurement of the reported FB-Line HEU content. A one gallon paint can fits almost exactly into the cavity of the active well.

Figure 9. Remote Set up of the HB-Line acquisition station.

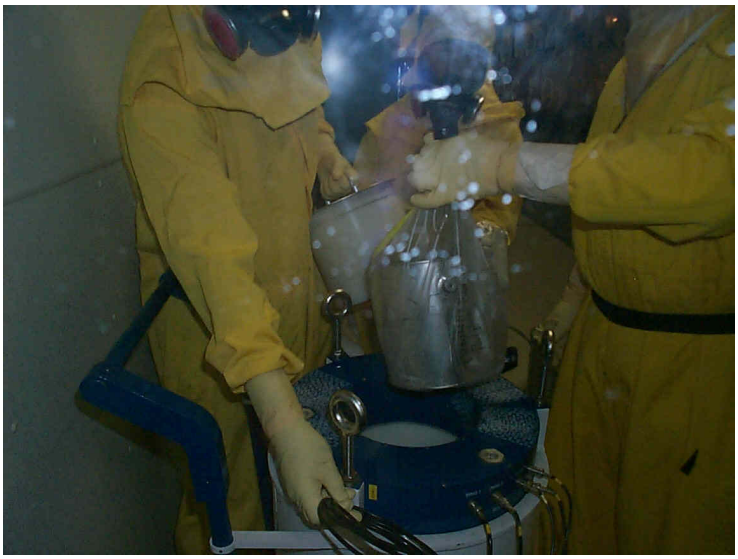

Figure 10. At-line set up of instrument in HB-Line facility.

Using one of the activation sources centered in the instrument cavity as a sample we performed passive acquisitions in both the fast and thermal mode to observe the singles rates. We observed a singles efficiency of 27.8% in the thermal mode and 25.1% in the fast mode. The latter efficiency was in extremely good agreement with the measured fast mode efficiencies above. Since sensitivity of the method would be an important issue for the low mass range, we elected to use the thermal mode of operation with the identical acquisition detector parameters and timing characteristics of Table 1.

The time demands of the project did not allow us an experimental program to re-evaluate the die-away times and deadtime correction parameters for the thermal mode of acquisition. We anticipated that the observed singles and doubles rates would be low enough that deadtime corrections would have little impact. It is likely that an experimental measure of the optimum die-away time of the detectors would be slightly longer than the fast fission value of 52.36 μsec. Optimizing the die-away time would influence the shape of our calibration curve, but we believe our calibration data below demonstrate a good instrumental response.

After selecting the thermal mode of acquisition, we fashioned a block of the cement matrix with a cavity so that we could use it to simulate the effect of the matrix. We placed one of the activation sources into the cavity of the cement block and then placed it inside the AWCC well. With that configuration we observed our singles efficiency of neutrons coming from the simulated sample matrix. Our measured efficiency was 23.1%.

HB-Line operators made three working standards of Desicooler material using the identical process to distribute the standard HEU material in actual cement matrices. These working standards had HEU contents of 8.57 g, 28.28 g, and 32.35 g. We calibrated the instrument using these three working standards using twenty 900-second acquisitions. The measured neutron rates are listed in Table 6, and the doubles efficiency calibration curve is shown in Figure 11.

Table 6. Measured rates in events per sec for the three calibration acquisitions with the working standards.

Item Number	Acquisition Number	Declared ^{235}U (g)	Singles (cps)	Doubles (cps)	Triples (cps)
WD6-04	39JK5235.CAL	32.35	295.0±0.6	110.4±0.5	10.5±0.3
WD6-05	39KJ5053.CAL	28.28	343.3±0.6	102.6±0.5	9.3±0.3
WD6-06	39LJ1712.CAL	8.57	121.1±0.6	50.7±0.4	4.5±0.2

Over the next several weeks we performed verification measurements on 67 product samples as directed by the Department of Energy-Savannah River. A scatter plot of the verification measurements is shown in Figure 12. Figure 12 excludes measurements on three items that had huge positive biases in the results. We were able to trace the bias on those items to contributions from spontaneous fission of Pu trace contamination in those items. We confirmed this by removing the activation sources from the AWCC and counted all three items in the passive acquisition mode. We observed up to 2900 cps of passive singles, up to 180 passive doubles/sec, and up to 19 passive triples/sec on these items. The DOE-SR customer accepted our evaluation of these items and waived the required verification measurements on them. Since these desicooler items came originally from FB-Line, it was not difficult to accept the likelihood of trace Pu contributions.

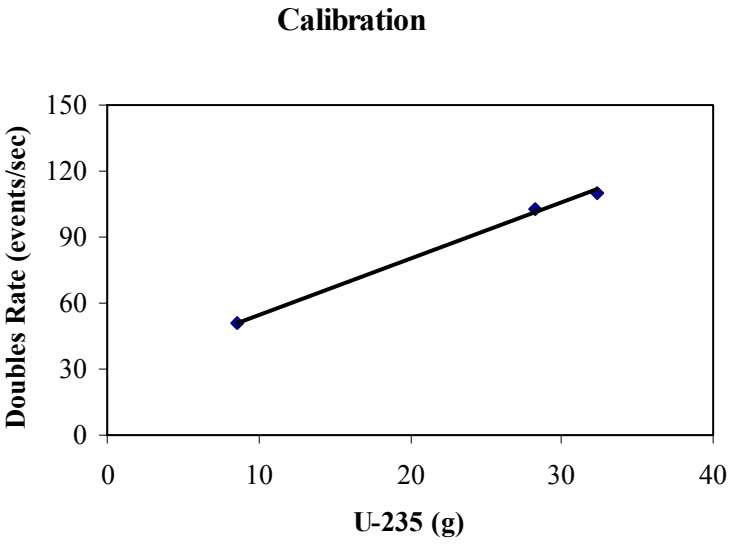

Figure 11. Desicooler Calibration.

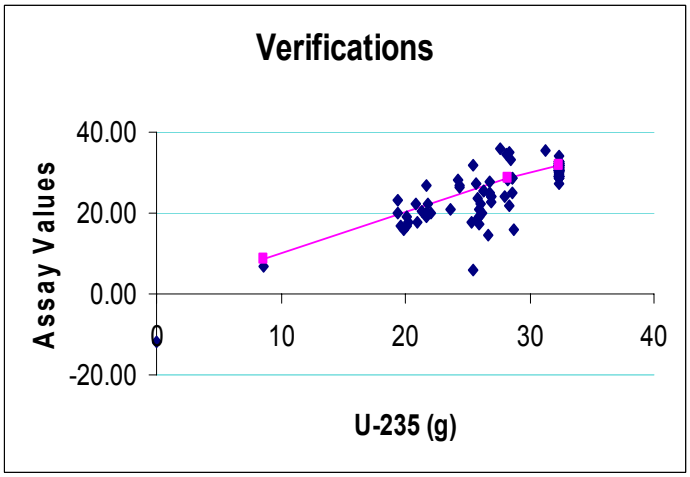

Figure 12. HB-Line Verifications

The verifications of Figure 12 and the calibrations of Figure 11 contain corrections to the observed singles and doubles rates that were derived by Cowgill in reference 2. This correction deals with leakage multiplication for the active well counter. The corrected doubles rate (D_c) is evaluated relative to the observed doubles rate (D_o) in equation (4).

$$D_c = \frac{D_o}{(S/S_o)^2} \quad , \qquad (4)$$

where S is the total singles for the item assayed (including active background) and S_0 is the active singles background. For our assays the largest doubles correction was less than

5%, and the largest verification mass correction was 7 g of HEU. While none of these corrections improved the verification measurements significantly, they did significantly improve the linearity of the calibration curve of Figure 11. Cowgill has evaluated our verification measurements obtained on metallic U at Argonne West National Laboratory above, and has demonstrated the very slight upbending of the curve of equation (3) is due to multiplication, as we suspected.[2]

This very successful campaign proved to be an adequate basis for subsequent SRNL acquisitions to book unknown U values in receipted Desicooler samples from FB-Line. When ATS received Desicooler samples whose total U content were in the region of 100g, and whose apparent content was far less than that, they requested assay assistance from AD. We performed transmission corrected γ-PHA assays on these samples as well as neutron activation acquisitions with our portable active well counter. The results of these combined acquisitions allowed SRNL MC&A to book new and more credible, lower HEU values for eight receipted samples.[20]

These book value acquisitions were performed in 2006, 29 months after the HB-Line Desicooler campaign. Before performing the thermal mode active well book value measurements, we used the active well in the passive neutron multiplicity mode to confirm there were no significant Pu contributions to the SNM contents of each sample. We discuss passive neutron multiplicity verification measurements with the active well counter in the last section of this paper.

We do not tabulate the active assays of these eight samples, but list results only. Each had measurable active doubles rates in the range –0.5 – 0.4 events/sec, but each was below the sensitivity limit for the assay method. We were able to set limits of 4 g ^{235}U in each sample. This limit of detection is in excellent agreement with the content measured by γ-PHA of 0.03 – 3.8 g HEU.

SRTC Passive Neutron Activites

In a fourth demonstration of the utility of the Aquila active well counter, we set it up as a portable neutron monitor for passive acquisitions in the Savannah River National Laboratory (Savannah River Technology Center) F-wing Californium Production Facility to evaluate the anomalous behavior of the observed neutron background of that facility.[21] Over the years of the 1990's the F-wing of SRNL was believed to have a neutron background that was due solely to the ^{252}Cf stored and held up in the facility. However by 1999 it was obvious that the neutron background was not decaying with the suitable 2.6-yr ^{252}Cf half-life. The authors were requested to investigate the issue.

Using the active well counter as a portable passive neutron detector we obtained passive singles and coincidence rates at multiple acquisition points on both the service floor and main floor of SRNL F-wing. The singles rate results are shown in Figures 13 and 14.

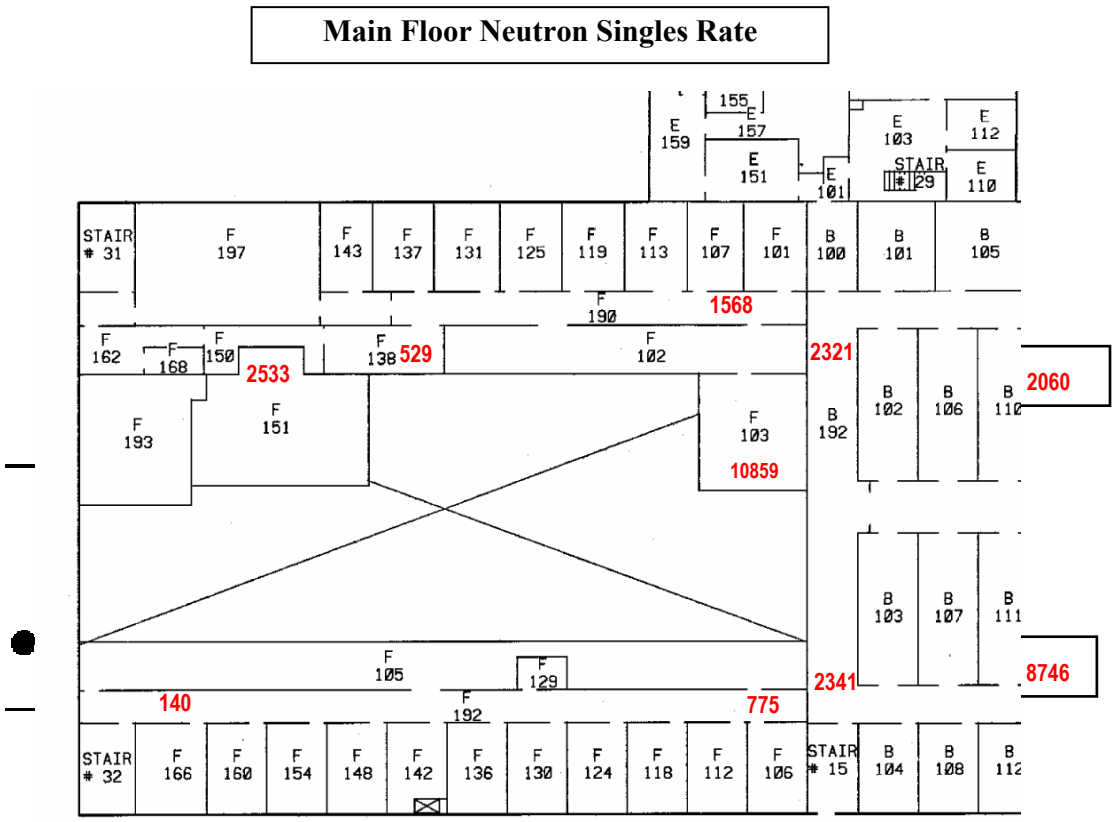

Figure 13. Neutron Singles Rates observed on the main Floor of Building 773-A F-wing.

Using this technique it was an easy process to identify the hot spots on both floors of the F-Wing facility. On the main floor the highest neutron rate was observed to be 10859 cps in room F-103, which is immediately above the service floor room F-003 where we observed a rate of 33155 cps. One hotter spot (45321 cps) and another high rate (22706 cps) were observed on the service floor. The latter two could be likely associated with the Californium Production Facility, while the former two could not. The observed passive doubles rates added very interesting data.

If the neutron background could be associated with ^{252}Cf from the Californium Production Facility as assumed, then there ought to be an observable doubles rate. That is, ^{252}Cf produces neutrons from spontaneous fission, and these neutrons can be expected to have a multiplicity of near three. We would expect the doubles rates to be strongly suppressed compared to the observed singles rates because the source was not inside our portable monitor. But we expect the doubles rates to be non-zero.

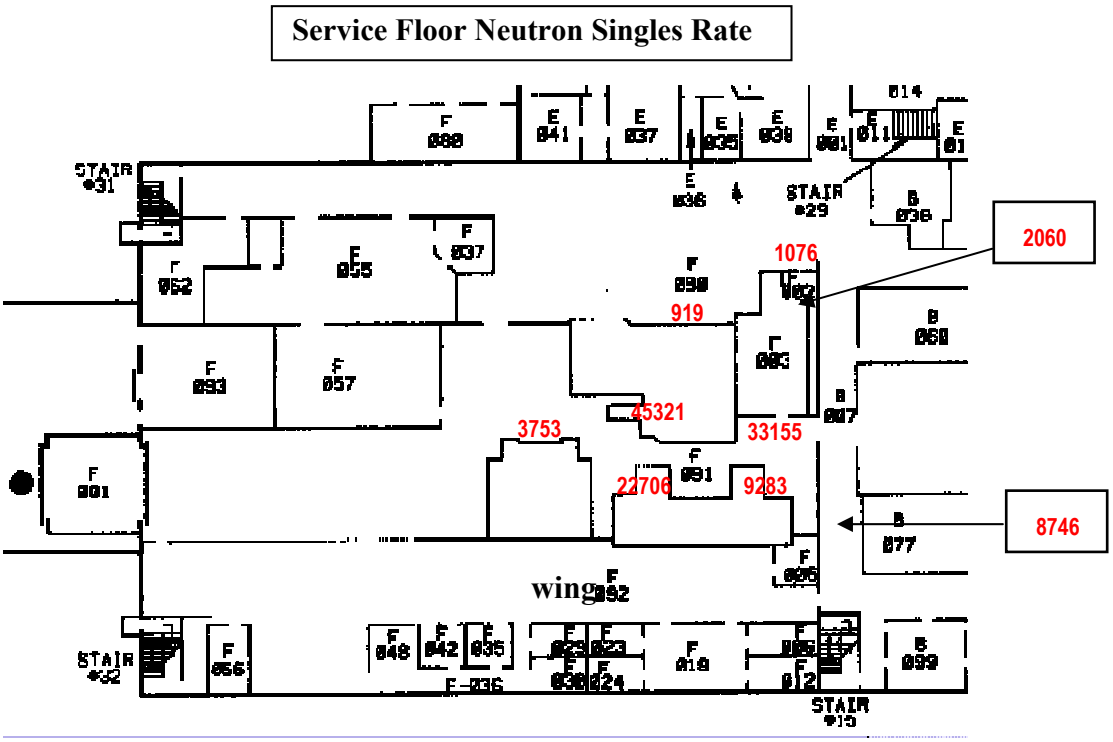

Figure 14. Neutron singles rates observed on the service floor of Building 773-A F-Wing.

Our hypothesis for the hot spot in F-003 was that the source was not ^{252}Cf but rather a PuBe or AmLi neutron activation source. Neither of these two would be expected to yield doubles events. Or experimental observations shown in Table 7 supported the hypotheses associated with the observed singles rates. Note the doubles rate associated with the singles rate of 45321 that we denote as CAM-39 is 137 events per second, and the doubles rate associated with the singles rate of 22706 that we denote as Glovebox 10 is 119 events per second. The doubles rate associated with the singles rate of 33155 that we measured outside of F-003 is (–4.6±8.6) events per second and the doubles rate associated with the singles rate of 10859 that we denote as filer 329 is only (18±12). The other two acquisitions in the area (Filter 326 and filter 327) have no measurable doubles rates.

Note also the SED facility, which is associated with Pu production, does not have a measurable doubles rate. We concluded that the singles rates near the CPF were predominantly from decay of ^{251,252}Cf while the high rates near F-003 came from a PuBe neutron activation source. To further reinforce our conclusions, we obtained passive γ-PHA analyses of each neutron acquisition point using a portable high purity germanium (HpGe) detector.

Table 7. SRTC F-Wing Neutron acquisitions.

Location	Singles	uncert	Doubles	uncert	Triples	uncert
CAM-39	45321.0	5.1	137.3	12.6	-12.8	13.7
Check point 1	2321.1	0.5	-0.4	0.3	-0.2	0.1
Check point 2	2341.3	0.9	0.3	0.5	0.1	0.1
CPF Cell	919.3	0.7	-0.6	0.2	0.0	0.0
Outside F-160	139.5	0.1	0.0	0.0	0.0	0.0
Filter 329	10859.2	10.5	18.0	12.4	0.2	7.177
Filter 327	7945.2	8.9	-13.5	9.0	-5.3	-4.3
Filter 326	11160.7	10.6	-6.2	12.7	-4.2	7.0
F-091 B-Wall	8746.5	2.1	-1.5	2.2	-0.4	1.2
F-151 SED	2395.5	1.7	0.1	0.9	0.3	0.3
F-003 from F-002	2059.8	0.7	-0.3	0.3	0.1	0.1
F-151 SED 2	2431.5	1.5	-2.4	0.4	0.1	0.2
F-003 from F-091	33154.5	4.1	-4.6	8.6	-11.8	8.4
F-091 glovebox 10	22705.7	3.4	118.6	5.9	2.0	4.8

We do not show any of the γ-ray spectra we obtained. Each of the important ones are displayed in reference 21. The spectra from F-091 glovebox 10 and from a ^{252}Cf source displayed multiple transitions at 177-, 227-, 253-, 333-, and 388-keV that originate from the isotopes ^{249,251}Cf co-produced with ^{252}Cf. None of these were observed in the spectra from outside F-003 or from outside the SED facility. The SED facility spectrum contained a very strong signal from multiple γ-rays associated with ^{239}Pu decay, and the spectrum obtained from outside F-003 contained a transition at 153-keV from decay of ^{238}Pu.

We finally obtained neutron acquisitions from the Analytical Development Section ^{252}Cf neutron activation facility to confirm that we would observe passive measurable doubles rates from a ^{252}Cf source external to our well counter. Table 8 summarizes those acquisitions where with each successive acquisition we have brought the source closer to the portable active well instrument. Acquisition b003source1 was obtained from 9 feet, the next two from approximately five feet on successive days, B-003 Cf 4 was obtained from 3 feet, and B-003 Cf 5 was obtained with the source inside the chamber of the active well counter. It is clear that a ^{252}Cf source produces a doubles rate that rapidly drops off with distance. This decrease in doubles rate can be attributed to the simple geometric distance effect as well as to the increased scatter which eliminates the coincidence relationship between correlated neutrons.

Table 8. Neutron acquisitions in Californium neutron activation facility obtained in experiments with Cf-252 source.

Acquisition	Singles	uncert	doubles	uncert	triples	uncert
B-003bg328	24.31	0.24	0.026	0.019	0	0.003
b003source1	1351.09	1.80	0.818	0.751	0.008	0.156
B003source2	4100	400	9.2	8.0	0.2	2.5
B-003 Cf 3	4748.8	3.4	6.47	2.65	1.614	0.879
B-003 Cf 4	66101.54	13.54	2585.80	41.80	16.87	52.48
B-003 Cf 5	460641.604	49.4	127709.193	497.0	12525.339	911.582

From the passive data acquired with the neutron well counter in SRNL F-Wing associated with the passive γ-PHA data and associated with the passive data acquired with the neutron well counter with a known ^{252}Cf source, we were able to determine conclusively that the SRNL F-Wing neutron background was not composed exclusively of ^{252}Cf activity. We were also able to identify the two main sources of non-^{252}Cf neutron activity. Upon our evaluation and recommendation, the PuBe source that we "discovered" in F-003 was removed. This substantially reduced the contemporary neutron background in F-Wing and transformed the existing background to one that is decaying as expected with the 2.6-year ^{252}Cf half-life. Module F-003 has been renovated into a productive actinide chemistry laboratory.

247-F Activation Analyses

The last use of the Aquila active well counter that we discuss is one in which we evaluated the HEU content of two incompletely labeled 55-gallon drums discovered in the deactivation and decommissioning of the 247-F Naval Fuels (NF) Facility at SRS. The two drums were discovered inside of a warehouse used by the NF facility. The drums were not adequately labeled but appeared to be spaced apart with fixed geometry to ensure criticality safety. The apparent fixed geometry raised criticality concerns with regard to movement of the two drums. Since the facility was constructed with the intention to manufacture nuclear propulsion fuel for the navy, a criticality concern was a credible event.[22-24] The Facilities Disposition Department (FDD) requested the authors to conduct a nondestructive assay to determine if these drums contained fissile materials and in what quantity.

Our initial evaluation of these two drums utilized segmented transmission corrected γ-PHA of each of the two drums. These acquisitions and the calculations are described in reference 25. A photograph of one of the acquisitions is shown in Figure 15. For the transmission correction of each drum segment we measured the direct transmission of the 661.6-keV γ-ray using a ^{137}Cs transmission source. From each direct measurement of transmission of the ^{137}Cs we calculated an areal density for the segment and from there calculated the absorption of the 185.7-keV γ-ray from decay of ^{235}U. From the corrected detection rate we determined limits of content for each segment in both of the two drums. Our measured data established limits of 1 g and 0.4 g of ^{235}U in the two drums.

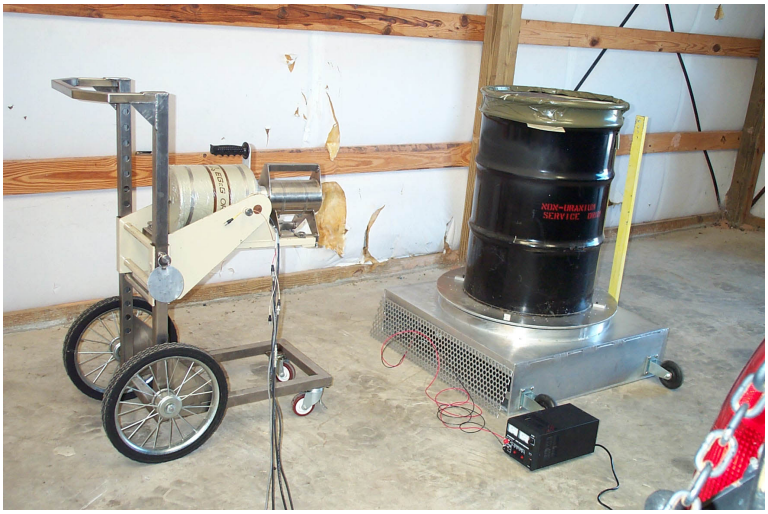

Figure 15. Photograph of the Naval Fuels gamma PHA setup.

While these γ-ray measurements appeared to completely satisfy the criticality concerns of FDD, the calculated transmission corrections for the six segments of the two drums ranged from 1.07 to 12.91 and clearly demonstrated a non-uniform distribution of mass inside the drums. The correction factors of 9.6, 12.9, and 7.6 in one of the drums demonstrated a content of very highly absorbing items. We believed that a neutron measurement was required to confirm and reinforce our passive γ-ray measurements.

To obtain neutron measurements of the ^{235}U content of these two drums it was necessary to devise a method to activate any HEU that might be present in the drums and to count the fission neutrons generated. Since it was not possible to place the 55-gallon drums inside of our active well detector, it was necessary to activate and measure the drums externally. An external efficiency calibration was required. A slab detector would have been the ideal solution. SRNL now possesses just such a detector[26], but we did not at the time.

The external activation efficiency calibration was performed on the loading dock of the M-Area Reactor Fuel Fabrication Facility using a ^{252}Cf source, scratch polyethylene moderation and reflection, and with fourteen HEU sources. The fourteen separate sources allowed us to perform the calibration with multiple combinations of HEU mass that ranged from less than one gram up to 400 grams. We obtained activation data for increments of approximately 50 g from 0 to 400. The ^{252}Cf source strength was approximately 10^5 neutrons/sec (reference 27). In each case the separate HEU sources were placed inside of an empty 55-gallon drum and the ^{252}Cf was rested on top of the drum as shown in Figure 16. Polyethylene reflection and moderation were set up as shown in Figure 16.

The measured singles and doubles rates are listed in Table 9, where the active background rates of ^{252}Cf only have been subtracted from the measured data using the INCC software. While the activated doubles rates are not conclusive, it is clear that the measured singles rates correlate with ^{235}U mass in the range 150 g – 300 g. Using this

technique of neutron activation, we believe that our measurement system is sensitive to HEU masses down to 150 g ^{235}U. We performed neutron activation measurements on the two drums.

Figure16. Photograph of the Cf-252 activation setup for the AWCC.

Table 9. Measured neutron singles and doubles rates in the external activation calibration.

Acquisition Name	Item ID	Singles (cps)	sigma singles	Doubles (cps)	sigma Doubles
cf in poly	Background	450.81	1.38	1.8	0.35
300g u activated	all u 300 g	110.6	2.99	4.07	1.15
allminus100	u 200 g	31.21	3.28	0.96	1.23
allbut150g	u 150g	19.78	3.27	1.54	1.23
allbut200	u 100g	20.3	3.27	2.01	1.22
50gram	u 50g	24.74	3.27	-0.17	1.23

The AWCC measurements allowed us to qualitatively search for chunks of material. The AWCC is designed to use observed neutron coincidence rates to measure HEU content of samples that are placed *inside* the well of the instrument. For those samples it has a sensitivity of approximately 50 g when used in the fast mode, as our U-Al and metal U results above demonstrate. In this adoption we have utilized the instrument to count neutrons from samples that have been activated external to the instrument. This greatly reduces our detection efficiency, so that measuring coincidence events was not practical. We have attempted to measure neutron singles rates to search for evidence of activated ^{235}U.

Our activation measurements of the two drums in the Naval Fuels warehouse facility yielded null results. Neither the measured singles nor doubles rates were above active background rates of 1190 singles/sec and 15 doubles/sec. While the AWCC results we obtained did not allow us to set limits of HEU chunks inside the drums, we were able to state that we had negative qualitative evidence of any such chunks being inside the drums.

Passive Neutron Multiplicity Measurements in SRNL

In August and December of 2005 the Actinide Technology Section of SRNL received separate items of special nuclear material that appeared to be mislabeled. On each occasion ADS was requested to perform a confirmatory measurement of the items to confirm content. For both receipts we performed a passive γ-PHA analysis to confirm HEU content in the August receipt and to confirm ^{238,239}Pu content in the December receipt. In each case the γ-ray measurement was adequate for confirmatory identification of the nuclear material, but in neither case was the γ-ray measurement unambiguously acceptable for quantitative assay. The HEU measurements was made difficult be the strongly absorbing material, and the Pu-isotopics assay was made difficult by the very high rate coming from the ^{238}Pu component. We quickly recognized the advantage that a portable neutron multiplicity counter would have given us.

Subsequent to the ^{238,239}Pu γ-ray acquisitions we tested the Aquila instrument for its capabilities as a passive neutron multiplicity assay device for Pu content. Our nuclear material stored in the SRNL nuclear NDA laboratory was especially well suited for this test. We have two Pu sources of varying isotopics and multiple HEU sources that allow us to compile incremental sources of HEU of up to 400 grams in ^{235}U mass. One of the Pu sources has contents of 0.65 g ^{239}Pu and 0.13 g ^{240}Pu and the other has contents of 6.92 g ^{239}Pu and 0.26 g ^{240}Pu. The latter item also has a content of 1.04 g ^{235}U and 40.75 g ^{238}U. Together these items can make up a composite sample of 7.57 g ^{239}Pu and 0.39 g ^{240}Pu. They will yield three test samples that we could use to qualify the active for passive Pu NMC verification measurements.

The results of the passive neutron multiplicity analyses of these “three” samples are listed in Table 10. Note we have two separate acquisitions of the first sample 500411 to demonstrate the difference obtained between comparatively short and comparatively long acquisition times. From the data of Table 10 we believe the active well counter has demonstrated excellent capabilities to perform passive neutron multiplicity analyses of Pu in the mass range 0.8 – 10 g.

Table 10. Measured neutron event rates and verification results for the passive multiplicity assays of the Pu nuclear material in the SRNL NDA facility.

Acquisition Name	acquisition time parameters	declared Pu mass (g)	Singles (cps)	Doubles (cps)	Triples (cps)	verification Pu-240 mass (g)	Total Pu verification mass (g)
Background	25 cycles x 21 seconds	0	41.2±0.3	0.28±0.22	0.000±0.000	N/A	N/A
Null sample(1)	6 cycles x 37 seconds	0	21.2±1.1	-0.11±0.18	-0.01±0.07	-0.003±0.024	-0.056±0.487
500411	21 cycles x 26 seconds	0.78	164.9±0.7	1.73±0.12	0.106±0.019	0.058±0.010	0.829±0.145
500411	36 cycles x 32 seconds	0.78	163.2±0.5	1.60±0.08	0.139±0.016	0.035±0.008	0.500±0.090
M15197	18 cycles x 50 seconds	7.91	120.6±0.6	14.9±0.2	1.49±0.06	0.353±0.007	9.88±0.19
combined	14 cycles x 46 seconds	8.69	322.0±0.9	18.14±0.26	1.810±0.084	0.401±0.015	8.196±0.312
M15197 + 200 g HEU	15 cycles x 42 seconds	7.91	336.9±0.6	18.08±0.27	1.719±0.081	0.415±0.016	8.476±0.318

We subsequently performed NMC verification measurements on receipted HEU samples from FB-Line and from the F-Area Material Storage facility so that we could treat them as HEU unknowns for our AWCC monitor. The measured values for these samples are shown in Table 11. Note all five appear to have null or very low Pu content. Item M015283 was measured as non-zero, but trace Pu content even with a measured NMC alpha value of 21.965 ± 8.891. With this very large (α,n) singles component we would expect the active well measurements for HEU to be biased high. Similarly item M015237 had an NMC alpha value of 63.850 ± 64.505, yet we were able to demonstrate a total Pu content of less than measurable. Item M015284 had an alpha value of 56.127 ± 109.442 with a measured Pu content of 0.133 ± 0.256 (i.e. less than detectable). Each of the acquisitions in Table 11 confirmed that the samples were eligible for active well analysis for HEU content.

Table 11. Measured neutron event rates and verification results for the passive multiplicity assays of the ATS HEU nuclear material.

Acquisition Name	acquisition time parameters	declared Pu mass (g)	Singles (cps)	Doubles (cps)	Triples (cps)	verification Pu-240 mass (g)	Total Pu verification mass (g)
Background ATS Lab 1	17 cycles x 46 seconds	0	17.666± 0.151	0.026± 0.009	0.000±0.000	N/A	N/A
M015237	20 cycles x 20 seconds	0	152.04± 0.67	0.280± 0.100	0.019±0.010	0.009±0.009	0.188±0.188
Background ATS Lab 2	34 cycles x 450 seconds	0	10.543± 0.026	0.043± 0.002	-0.005±0.013	N/A	N/A
M015284	8 cycles x 13 seconds	0	93.892± 1.002	0.038± 0.007	-0.005±0.013	0.006±0.013	0.133±0.256
M015283	18 cycles x 20 seconds	0	91.420± 0.535	0.200± 0.067	-0.001±0.007	0.016±0.006	0.323±0.127
Background ATS Lab 3	17 cycles x 38 seconds	0	22.813± 0.188	0.039± 0.013	-0.000±0.000	N/A	N/A
M015273	20 cycles x 20 seconds	0	17.205± 0.191	0.028± 0.013	0.006±0.000	-0.002±0.000	-0.039±0.006
M015287	95 cycles x 500 seconds	0	93.892± 1.002	0.038± 0.007	-0.005±0.013	0.006±0.013	0.133±0.256

In Table 12 we show measurements of four samples of special nuclear material using passive neutron multiplicity counting to demonstrate the capabilities of the active well counter for Pu verification measurements. For none of these four samples did we have a certified Pu-240 isotopics content, so we used a constant value of 6.0% in each of the analyses. The multiplication and calculated alpha values for each are listed in Table 13, which also contains the multiplication and alpha values for the very successful NMC verification acquisitions of Table 10. Note in three of the four tests of Table 12 we were able to demonstrate very good agreement within the uncertainty of the unknown Pu-240 isotopics. For sample 50014671 the instrument calculated a large multiplication and a very large and negative alpha value with a very large uncertainty. (Note the huge singles rate of 52782 cps.) This assists an operator to note when multiplicity counting fails. However even in the event of failure, the analytical software projected very large uncertainties in the Pu-240 and total Pu verification masses. These uncertainties assure that the operator would not "believe" and accept the measured values.

Table 12. Measured neutron event rates and verification results for the passive multiplicity assays of Pu special nuclear material.

Acquisition Name	acquisition time parameters	declared Pu mass (g)	Singles (cps)	Doubles (cps)	Triples (cps)	verification Pu-240 mass (g)	Total Pu verification mass (g)
Background ATS Lab 4	7 cycles x 67 seconds	0	141.454± 0.550	0.1109± 0.076	0.006±0.006	N/A	N/A
Null sample(2)	44 cycles x 141 seconds	0	-0.115 ±0.128	-0.002± 0.011	-0.001± 0.001	0.057±0.025	0.403±0.064
50014668	8 cycles x 78 seconds	101.40	1339.256 ±1.964	238.342± 1.241	28.657± 0.534	5.260±0.047	87.708±0.779
50014499	10 cycles x 70 seconds	14.71	879.373± 1.377	30.237± 0.526	2.789± 0.147	0.682±0.040	11.370±0.662
50014671	8 cycles x 116 seconds	35.	52781.52 ±6.09	199.025± 16.119	30.926± 19.334	-3.561±10.319	-59±172
20015077	13 cycles x 120 seconds	0	0.216± 0.627	0.031± 0.087	0.004± 0.004	0.001±0.002	0.010±0.027

Table 13. Measured neutron event rates and verification results for the passive multiplicity assays of Pu special nuclear material.

Acquisition Name	Acquisition time parameters	Multiplication	Alpha
Null Sample (2)	44 cycles x 141 seconds	-1.074±2.330	-0.772±0.254
50014668	8 cycles x 78 seconds	1.057±0.002	-0.056±0.007
50014499	10 cycles x 70 seconds	1.017±0.004	3.974±0.27
50014671	8 cycles x 116 seconds	1.020±0.023	-57.988±166.440
20015077	13 cycles x 120 seconds	1.075±0.226	0.262±3.362
500411	21 cycles x 26 seconds	0.993±0.006	10.147±1.892
500411	36 cycles x 32 seconds	1.006±0.005	17.349±3.901
M15197	18 cycles x 50 seconds	1.032±0.004	0.298±0.022
combined	14 cycles x 46 seconds	1.027±0.004	2.063±0.105
M15197 + 200 g HEU	15 cycles x 42 seconds	1.022±0.004	2.109±0.106

CONCLUSIONS

We have demonstrated six distinct applications of the SRNL Aquila active well neutron coincidence counter for measurement of ^{235}U, ^{240}Pu, and for evaluation of ^{252}Cf and $^{238,239}Pu$ contributions to observed ambient neutron backgrounds. The neutron activation measurements of ^{235}U content utilized both the fast mode and the thermal mode of neutron measurement to demonstrate nondestructive assay in three distinct mass ranges and for three distinct chemical forms of HEU. Use of the active well counter as a portable passive neutron monitor allowed us to determine the composition of the SRNL F-wing neutron background to explain why the background was not decaying with the appropriate half-life of a pure ^{252}Cf composition. The observations yielded identification of a $^{238}Pu(Be)$ source that was removed. The module was subsequently renovated for use as a productive chemical laboratory. We have also demonstrated the use of the active well instrument with external activation analysis to evaluate criticality concerns of two 55-gallon drums stored in an apparently critically safe geometry in the SRS Naval Fuels facility. Finally we have demonstrated the use of the active well monitor in the passive neutron multiplicity mode for ^{240}Pu and total Pu assays.

The quantitative nondestructive assay measurements include determination U metal, U-Al, and Desicooler uranium waste. Nondestructive assay of U metal in the mass range of 0 – 5000 g were demonstrated using the fast mode of activation at the Argonne West National Laboratory. Nondestructive assay of U-Al in the mass range of 50 – 1300 g were demonstrated using the fast mode of activation at the SRS K-Area Material Storage facility. These nondestructive assay measurements have been used for seven successive years of verification measurements in the DOE annual inventory of U-Al fuel ingots. Finally nondestructive assay of waste Desicooler U material using the thermal activation mode in the mass range of 5 – 35 g were demonstrated at the SRS HB-Line facility. These measurements provided HB-Line the capability for waste processing verification measurements that satisfied DOE requirement for discard of 4.3 kg of HEU as Attractiveness Level E material.

The thermal and fast modes of neutron activation were applied for measurement of HEU in the regime of < 4 g to assist with book value measurements of ^{235}U in the Actinide Technology Section of SRNL. To confirm each of the four samples assayed by activation were pure of Pu contributions, we performed a neutron multiplicity analysis on each of them. Null results were obtained, thus clearing the samples for activation analysis of HEU. For six of seven other SNM samples and composite SNM samples we have demonstrated accurate verification measurement capabilities using the passive neutron multiplicity measurement mode. These samples had Pu contents in the range 0.8 g – 100 g with interfering HEU contents of up to 200 g.

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