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Title: Qualification of the Savannah River National Laboratories Coulometer, Model SRNL-Rev. 2 (Serial # SRNL-003 Coulometer) for use in Process 3401a, Plutonium Assay by Controlled Coulometer

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Intended for: Discussion at our next QA meeting with LASO.  
Report



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## **Abstract**

22 August 2012

Qualification of the Savannah River National Laboratories Coulometer, Model SRNL-Rev. 2 (Serial # SRNL-003 Coulometer) for use in Process 3401a, Plutonium Assay by Controlled Coulometer

Lav Tandon, Lisa Colletti, Randy Drake, Elmer Lujan, Kathy Garduno

This report discusses the process used to prove in the SRNL-Rev.2 coulometer for isotopic data analysis used in the special plutonium material project.

**Qualification of the Savannah River National Laboratories  
Coulometer, Model SRNL-Rev. 2 (Serial # SRNL-003 Coulometer)  
for use in Process 3401a, Plutonium Assay by Controlled  
Coulometry**

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**22 August 2012**

**Introduction:**

In May of 2012, the PAR 173 coulometer system that had been the workhorse of the Plutonium Assay team since the early 1970s became inoperable. A new coulometer system had been purchased from Savannah River National Laboratory (SRNL) and installed in August of 2011. Due to funding issues the new system was not qualified at that time. Following the failure of the PAR 173, it became necessary to qualify the new system for use in Process 3401a, *Plutonium Assay by Controlled Coulometry*.

A qualification plan similar to what is described in PQR -141a was followed. Experiments were performed to establish a statistical summary of the performance of the new system by monitoring the repetitive analysis of quality control sample, PEOL, and the assay of plutonium metals obtained from the **Plutonium Exchange Program**. The data for the experiments was acquired using work instructions ANC125 and ANC195. Figure 1 shows approximately 2 years of data for the PEOL material obtained using the PAR 173. The required acceptance criteria for the sample are that it returns the correct value for the quality control material of 88.00% within 2 sigma (95% Confidence Interval). It also must meet daily precision standards that are set from the historical data analysis of decades of data. The 2 sigma value that is currently used is 0.146 % as evaluated by the Statistical Science Group, CCS-6. The average value of the PEOL quality control material run in 10 separate days on the SRNL-03 coulometer is 87.98% with a relative standard deviation of 0.04 at the 95% Confidence interval. The date of data acquisition is between 5/23/2012 to 8/1/2012. The control samples are run every day experiments using the coulometer are carried out. It is also used to prove an instrument is in statistical control before any experiments are undertaken. The total number of replicate controls run with the new coulometer to date, is n=18. This value is identical to that calculated by the LANL statistical

group for this material from data produced by the PAR 173 system over the period of October 2007 to May 2011.

The final validation/verification test was to run a blind sample over multiple days. AAC participates in a plutonium exchange program which supplies blind Pu metal samples to the group on a regular basis. The Pu material supplied for this study was ran using the PAR 173 in the past and more recently with the new system. Table 1a contains the values determined through the use of the PAR 173 and Table 1b contains the values obtained with the new system. The Pu assay value obtained on the SRNL system is for paired analysis and had a value of 98.88+/-0.07% RSD at 95% CI. The Pu assay value (decay corrected to July 2012) of the material determined in prior measurements using the PAR173 is 99.05 +/- 0.06 % RSD at 95% CI. We believe that the instrument is adequate to meet the needs of the program.

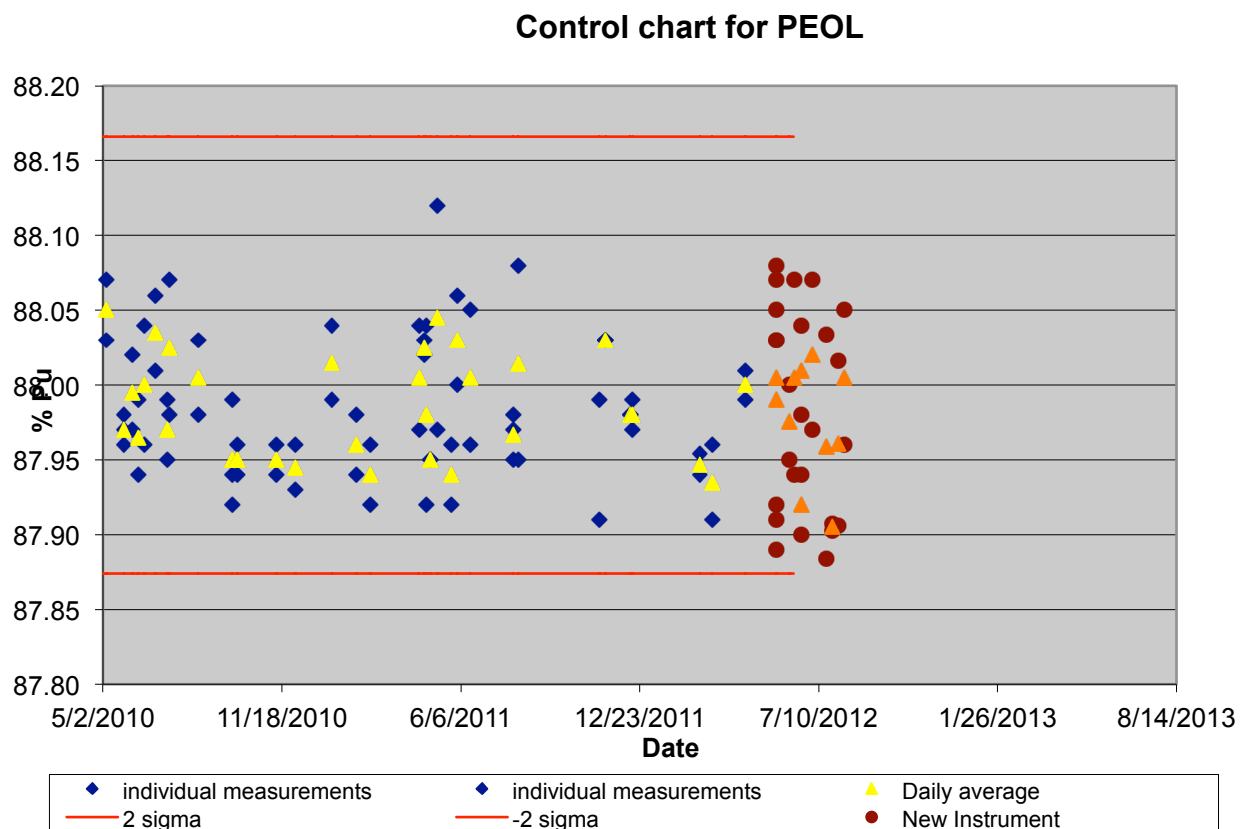


Figure 1. Control chart for Pu oxide control material. Red and orange represent data from the SRNL-03 Coulometer.

Table 1. Individual determinations for Pu (wt%) for Metal Exchange sample. Table 1a is for 4 years of data. All data was obtained with the PAR 173 coulometer. Table 1b contains the values obtained during the 2012 qualification using the SRNL coulometer.

PARR 173 Coulometer		
Sample ID	Pu (wt%)	Pu (wt%)
		Decay Corrected to 7/15/2012
Oct-07 1A	98.91	98.87
Oct-07 1B	98.92	98.88
Oct-07 2A	99.03	98.99
Oct-07 2B	99	98.96
Sep-08 1A	99.25	99.21
Sep-08 1B	99.26	99.22
Sep-08 2A	99.21	99.17
Sep-08 2B	99.19	99.15
Jul-09 1A	99.07	99.15
Jul-09 1B	99.04	99.03
Jul-09 2A	99.12	99.08
Jul-09 2B	99.1	99.06
Sep-11 1A	99.02	98.98
Sep-11 1B	98.95	98.91
Sep-11 2A	99.06	99.02
Sep-11 2B	99.09	99.05

SRNL-03 Coulometer		
Date of Analysis	Sample ID	Pu ( wt%)
7/18/12	A16143-1 A1	98.71
7/18/12	A16143-1 A2	98.66
7/18/12	A16143-1 A3	98.68
7/18/12	A16143-2 B1	98.76
7/18/12	A16143-2 B2	98.91
7/18/12	A16143-2 B3	98.90
7/18/12	A16143-2 B4	98.85
7/25/12	EJL-S C1	98.82
7/25/12	EJL-S C2	98.76
7/25/12	EJL-S C4	98.75
7/25/12	EJL-S D2	99.01
7/25/12	EJL-S D3	98.91
7/25/12	EJL-S D4	98.95
7/25/12	EJL-S D5	98.86
8/1/12	EJL-S E10	99.09
8/1/12	EJL-S E8	99.12
8/1/12	EJL-S F10	99.08
8/1/12	EJL-S F5	98.93

Table 2. Comparison of average Pu value for Pu Metal sample.

	Controlled Potential Coulometry Instrument	
	PAR173	SRNL-003
Mean Pu (wt%)	99.05	98.88
Std. Dev.	0.11	0.14
%RSD	0.11	0.14
L 95%CI	98.99	98.81
U 95% CI	99.11	98.94
N	16	18
DF	15	17
T-value	2.14	2.11
<b>F-Test</b>	<b>Passed</b>	