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Performance Testing of Software Upgrade for SRNL CPC instrument at the Nuclear Material Laboratory, Office of Safeguards Analytical Services, International Atomic Energy Agency

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PREFACE OR ACKNOWLEDGEMENTS

Thanks to the IAEA and the US Support Program for Sponsoring the Software Upgrade.

EXECUTIVE SUMMARY

This report summarizes the performance testing for the Software upgrade for the Savannah River National Laboratory (SRNL) Controlled Potential Coulometry (CPC) instrument. This upgrade is specifically to meet the needs of the Nuclear Material Laboratory, Office of Safeguards Analytical Services, International Atomic Energy Agency at Seibersdorf, Austria. The instrument hardware was upgraded in 2010, but the software continued to operate on a Windows platform using a High Tech Basic (HT Basic) application. HT Basic usage is declining. The IAEA requested an upgrade to the existing coulometer software to Laboratory Virtual Instrument Engineering Workbench (LabVIEW), an object-oriented programming language in wide use and one the NML staff is familiar with. The software was developed by SRNL Software Engineer, with assistance from Electrical Engineer and Scientist. Once developed, the new LabVIEW software was validated, tested with Iron (surrogate for plutonium) and plutonium. Once testing was completed, the software was installed at the International Atomic Energy Agency Nuclear Material Laboratory (IAEA NML) coulometer, and SRNL provided hands on training to NML staff on using the new software.

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LIST OF ABBREVIATIONS

SRNL	Savannah River National Laboratory
CPC	Controlled Potential Coulometry
IAEA	International Atomic Energy Agency
NML	Nuclear Material Laboratory
LabVIEW	Laboratory Virtual Instrument Engineering Workbench
High Tech Basic	HT Basic

1.0 Introduction

SRNL has been fabricating CPC instruments for over 30 years. The instrument hardware was upgraded in 2010, but the software continued to operate on a Windows platform using a High Tech Basic (HT Basic) application. HT Basic usage is declining. The IAEA requested an upgrade to the existing coulometer software to Laboratory Virtual Instrument Engineering Workbench (LabVIEW), an object-oriented programming language in wide use and one the NML staff is familiar with. The software was developed by SRNL Software Engineer, with assistance from Electrical Engineer and chemist. Once developed, the new LabVIEW software was validated, testing with Iron (surrogate for plutonium) and plutonium. Once testing was completed, the software was installed on the International Atomic Energy Agency Nuclear Material Laboratory (IAEA NML) coulometer, and SRNL provided hands on training to NML staff using the new software.

2.0 Scope

The performance testing of the LabVIEW software for SRNL CPC will demonstrate acceptable operation of the coulometer by performing:

System Automatic Continuous Calibration
Iron Blank/Sample Measurements
Plutonium Blank/Sample Measurements
Creation of files for data archiving
Installation and operation at NML CPC

The expected outcome is that the new software will produce results that will be equivalent to the results obtained using the HT Basic Program.

3.0 Procedure

3.1 System Automatic Continuous Calibration

Using the automatic calibration routine in the software, run at least three series of 10 calibration factors. The calibration file should appear similar to the calibration file for HT Basic. The system must be on for a minimum of 1 hour before starting calibration runs.

3.2 Iron Blank/Sample Measurements

Demonstrate Blank/Sample Measurement using Sulfuric acid solutions and Iron using the SRNL Cell. This test is not intended to test the performance of the cell assembly, acids, and iron. The purpose of this test is to determine if the software is functioning as expected. Note that system parameters may require changing to complete testing. This testing is only performed at SRNL.

3.2.1 Using a sulfuric or nitric acid with an Argon purge, demonstrate the running of a blank.

3.2.2 Using Iron, demonstrate the running of a sample.

3.3 Plutonium Blank/Sample Measurements

Demonstrate Blank/Sample Measurement using Sulfuric acid solutions and plutonium using the SRNL Cell. The purpose of this test is to determine if the software is functioning as expected and producing expected results, comparable to the HT Basic Software. Note that system parameters may require changing to complete testing.

3.3.1 Using a sulfuric or nitric acid with an Argon purge, demonstrate the running of a blank.

3.3.2 Using Plutonium, demonstrate the running of a sample.

3.4 Creation of files for data archiving

Demonstrate that the LabVIEW program creates the required txt and CXL files to archive data.

3.5 Installation and operation at NML CPC

Install the LabVIEW program on IAEA NML computer. Demonstrate Blank/Sample Measurement using Sulfuric acid solutions and plutonium using the IAEA NML Cell. The purpose of this test is to determine if the software is functioning as expected and producing expected results, comparable to the HT Basic Software. Note that system parameters may require changing to complete testing.

3.5.1 Perform continuous electrical calibrations

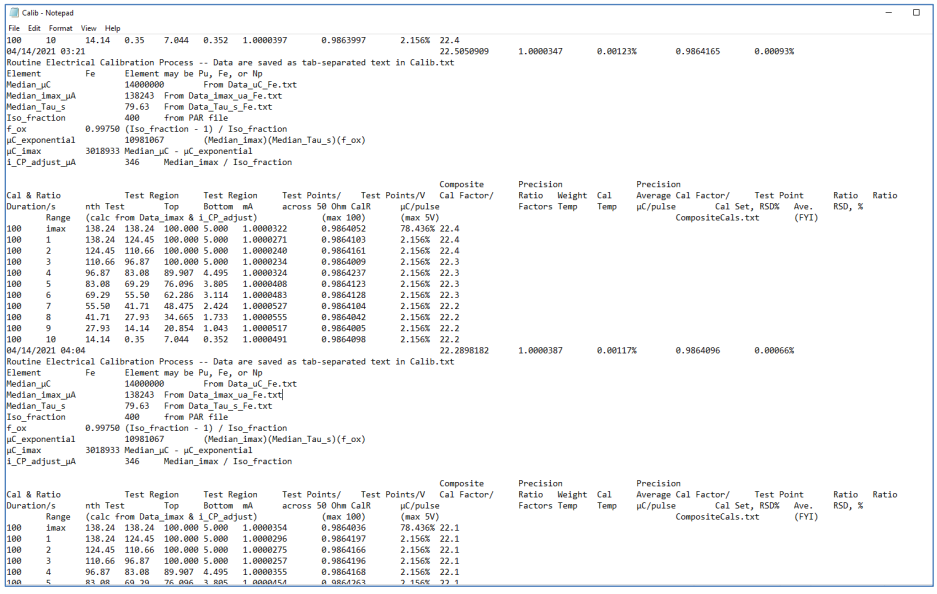
3.5.2 Using a sulfuric or nitric acid with an Argon purge, demonstrate the running of a blank.

3.5.3 Using Plutonium, demonstrate the running of a sample.

4.0 Results and Discussion

4.1 System Automatic Continuous Calibration

Using the automatic calibration routine in the software, the instrument was left running automatic continuous calibration. The calibration file produced was similar to the calibration file for HT Basic. See Figure 4-1.



Calib - Notepad

File Edit Format View Help

```
100     10      14.14   0.35    7.044   0.352   1.0000397       0.9863997       2.156%  22.4
04/14/2021 03:21                                                                        22.5050909      1.0000347       0.00123%        0.9864165       0.00093%
Routine Electrical Calibration Process -- Data are saved as tab-separated text in Calib.txt
Element         Fe      Element may be Pu, Fe, or Np
Median_µC               14000000        From Data_uC_Fe.txt
Median_imax_µA          138243  From Data_imax_ua_Fe.txt
Median_Tau_s            79.63   From Data_Tau_s_Fe.txt
Iso_fraction            400     from PAR file
f_ox            0.99750 (Iso_fraction - 1) / Iso_fraction
µC_exponential          10981067        (Median_imax)(Median_Tau_s)(f_ox)
µC_imax         3018933 Median_µC - µC_exponential
i_CP_adjust_µA          346     Median_imax / Iso_fraction

                                                                                Composite       Precision               Precision
Cal & Ratio             Test Region     Test Region     Test Points/    Test Points/V   Cal Factor/     Ratio   Weight  Cal     Average Cal Factor/     Test Point      Ratio   Ratio
Duration/s      nth Test        Top     Bottom  mA      across 50 Ohm CalR      µC/pulse                Factors Temp    Temp    µC/pulse        Cal Set, RSD%   Ave.    RSD, %
        Range   (calc from Data_imax & i_CP_adjust)              (max 100)       (max 5V)                                                CompositeCals.txt       (FYI)
100     imax    138.24  138.24  100.000 5.000   1.0000322       0.9864052       78.436% 22.4
100     1       138.24  124.45  100.000 5.000   1.0000271       0.9864103       2.156%  22.4
100     2       124.45  110.66  100.000 5.000   1.0000240       0.9864161       2.156%  22.4
100     3       110.66  96.87   100.000 5.000   1.0000234       0.9864009       2.156%  22.3
100     4       96.87   83.08   89.907  4.495   1.0000324       0.9864237       2.156%  22.3
100     5       83.08   69.29   76.096  3.805   1.0000408       0.9864123       2.156%  22.3
100     6       69.29   55.50   62.286  3.114   1.0000483       0.9864128       2.156%  22.3
100     7       55.50   41.71   48.475  2.424   1.0000527       0.9864104       2.156%  22.2
100     8       41.71   27.93   34.665  1.733   1.0000555       0.9864042       2.156%  22.2
100     9       27.93   14.14   20.854  1.043   1.0000517       0.9864005       2.156%  22.2
100     10      14.14   0.35    7.044   0.352   1.0000491       0.9864098       2.156%  22.2
04/14/2021 04:04                                                                        22.2898182      1.0000387       0.00117%        0.9864096       0.00066%
Routine Electrical Calibration Process -- Data are saved as tab-separated text in Calib.txt
Element         Fe      Element may be Pu, Fe, or Np
Median_µC               14000000        From Data_uC_Fe.txt
Median_imax_µA          138243  From Data_imax_ua_Fe.txt
Median_Tau_s            79.63   From Data_Tau_s_Fe.txt
Iso_fraction            400     from PAR file
f_ox            0.99750 (Iso_fraction - 1) / Iso_fraction
µC_exponential          10981067        (Median_imax)(Median_Tau_s)(f_ox)
µC_imax         3018933 Median_µC - µC_exponential
i_CP_adjust_µA          346     Median_imax / Iso_fraction

                                                                                Composite       Precision               Precision
Cal & Ratio             Test Region     Test Region     Test Points/    Test Points/V   Cal Factor/     Ratio   Weight  Cal     Average Cal Factor/     Test Point      Ratio   Ratio
Duration/s      nth Test        Top     Bottom  mA      across 50 Ohm CalR      µC/pulse                Factors Temp    Temp    µC/pulse        Cal Set, RSD%   Ave.    RSD, %
        Range   (calc from Data_imax & i_CP_adjust)              (max 100)       (max 5V)                                                CompositeCals.txt       (FYI)
100     imax    138.24  138.24  100.000 5.000   1.0000354       0.9864036       78.436% 22.1
100     1       138.24  124.45  100.000 5.000   1.0000296       0.9864197       2.156%  22.1
100     2       124.45  110.66  100.000 5.000   1.0000275       0.9864166       2.156%  22.1
100     3       110.66  96.87   100.000 5.000   1.0000257       0.9864196       2.156%  22.1
100     4       96.87   83.08   89.907  4.495   1.0000355       0.9864168       2.156%  22.1
100     5       83.08   69.29   76.096  3.805   1.0000454       0.9864363       2.156%  22.1
```

Figure 4-1. Example of output to Calibration text file

4.2 Iron Blank/Sample Measurements

Blank/Sample Measurements were performed using Sulfuric acid solutions and Iron at SRNL using the SRNL Cell. This test helped identified areas where the software was not functioning as expected. If an

unexpected outcome was obtained, the software engineer was notified. The software engineer performed changes to the program, as necessary, and testing was resumed. This process was repeated until the software produced expected outcomes. The testing progressed to testing with plutonium.

4.3 Plutonium Blank/Sample Measurements

Blank/Sample Measurement using Sulfuric acid solutions and plutonium using the SRNL Cell were performed. Plutonium standards were measured with HT Basic and LabVIEW software. The instrument and cell assembly used were the same. The only difference in the measurements is the software. All results with LabVIEW are within the laboratory limits.
Table 4-1 summarizes the data obtain. A t-test indicated the difference in the data is not statistically significant.

Table 4-1. Summary of plutonium measurements.

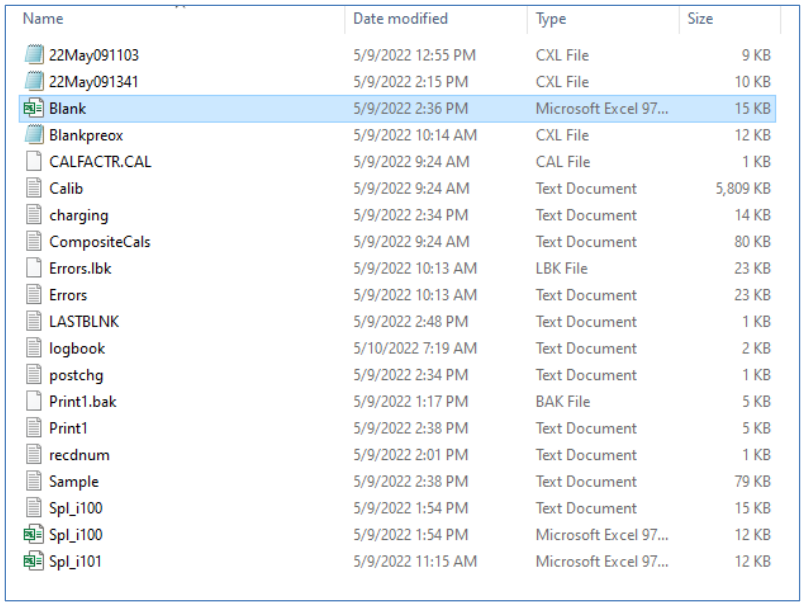
Software	Number of measurements	Average %RD (Pu corrected for Iron)	Std Deviation
HT Basic	10	-0.055	0.036
LabVIEW	10	0.050	0.067

4.4 Creation of files for data archiving

It was confirmed that the Data folder for the new software contained the required files. Table 4-2 summarizes the most important files. Figure 4-2 captures all the files generated by the software.

Table 4-2. Main files generated by software.

Filename	File content	Equivalent file in HT Basic
Blank.CXL	CXL file for the blank that contains the data collected during analysis	Blank.CXL
DateTime.CXL	CXL file for each ample analyzed with date and time stamp that contains the data collected during analysis	DateTime.CXL
Calib	Calibration txt file, results of continuous calibration	Calib
logbook	Text file that contains in chronological order blanks, samples, formal potentials	logbook
Sample	Text file that contains in chronological order blanks, samples, formal potentials with more detail information	samples



Name	Date modified	Type	Size
22May091103	5/9/2022 12:55 PM	CXL File	9 KB
22May091341	5/9/2022 2:15 PM	CXL File	10 KB
Blank	5/9/2022 2:36 PM	Microsoft Excel 97...	15 KB
Blankpreox	5/9/2022 10:14 AM	CXL File	12 KB
CALFACTR.CAL	5/9/2022 9:24 AM	CAL File	1 KB
Calib	5/9/2022 9:24 AM	Text Document	5,809 KB
charging	5/9/2022 2:34 PM	Text Document	14 KB
CompositeCals	5/9/2022 9:24 AM	Text Document	80 KB
Errors.lbk	5/9/2022 10:13 AM	LBK File	23 KB
Errors	5/9/2022 10:13 AM	Text Document	23 KB
LASTBLNK	5/9/2022 2:48 PM	Text Document	1 KB
logbook	5/10/2022 7:19 AM	Text Document	2 KB
postchg	5/9/2022 2:34 PM	Text Document	1 KB
Print1.bak	5/9/2022 1:17 PM	BAK File	5 KB
Print1	5/9/2022 2:38 PM	Text Document	5 KB
recdnum	5/9/2022 2:01 PM	Text Document	1 KB
Sample	5/9/2022 2:38 PM	Text Document	79 KB
Spl_i100	5/9/2022 1:54 PM	Text Document	15 KB
Spl_i100	5/9/2022 1:54 PM	Microsoft Excel 97...	12 KB
Spl_i101	5/9/2022 11:15 AM	Microsoft Excel 97...	12 KB

Figure 4-2. Files created by new LabVIEW program on Data folder.

4.5 Installation and operation at NML CPC

Before the installation of the new program at NML, the NML instrument was calibrated. See Appendix A for Calibration Report. The instrumetn was left overnight running continuous electrical calibrations. The next day a blank/sample determination was performed. Results were within the expected parameters. The formal potential was determined. All files for HT Basic were backed up.

A new computer was provided by IAEA personnel. The LabVIEW program was installed on IAEA NML computer. System parameters were adjusted for the instrument and cell configuration. The program was left running continuous electrical calibrations over lunch. Blank/Sample Measurement using Sulfuric acid solutions and plutonium using the IAEA NML cell were performed. Small adjustments to the program were performed. Final results displayed are total mg Pu. Results are within laboratory limit and agree with previous results. The software is functioning as expected and producing expected results, comparable to the HT Basic Software.
The IAEA NML personnel were trained on software use.

5.0 Conclusions

The IAEA NML CPC instrument was calibrated. The software was updated to LabVIEW. The software is functioning as expected and producing expected results, comparable to the HT Basic Software. Personnel were trained on the use of the new software.

6.0 Recommendations, Path Forward or Future Work

It is recommended that the personnel continue monitoring the performance of the new software and report any unexpected outcomes to SRNL personnel for evaluation.

Appendix A. **Calibration Report**

IAEA New Coulometer System, Calibrated on 2022-APR-25				
Lab Temperature 20.8 °C				
Aglient 34980A System Voltmeter Calibration				
Cross Check 34401A	**Agilent 34401A / V**	**Fluke 87**	**Relative Diff, %**	
IAEA Fluke Voltage Source				
0	0.0000700	0.0001000		
0.5	0.5000500	0.5000000	0.0100%	
1	1.0000300	1.0000000	0.0030%	
1.5	1.5000000	1.5000000	0.0000%	
2	2.0000000	2.0000000	0.0000%	
Cross Check 100 Ohm ASL Resitors at 22.6 Deg C (Use 23 Deg from Cal Report)				
34401A Four Wire Measurement	99.9980000	SRNL Cal Report	99.9982	0.00023
Condition of System prior to aligning the Digital Integrator				
K17 Relay Energized				
DAC 3001 = 2.5 V				
DAC 3002 = 1.0 V				
As Found Load Impedance and System Calibration Resistor Measurement				
R_{Std} resistor				
SRNL Calibration	Temp / °C	Resistance / Ω		
	20	99.998462		
	23	99.998225		
Temperature coefficient / Ω °C^{-1}			-0.00008	
Actual Temperature / °C	21			
Temperature Δ from 20 °C	1.00000			
Change in Resistance / ΔΩ		-0.00008		
Corrected Resistance / Ω		**99.99838**		
R_{Load} Measurement				
V_{Std} / V	0.994520			
V_{Load} / V	0.199750			
I_{Load} / A	0.0099454		previous	% shift
R_{Load} / Ω	20.08474		20.08437	0.00185

Appendix A. Calibration Report, continuation

R_{Cal} Measurement				
V_{Cal} / V	0.99034			
V_{Load} / V	0.39787			
I_{Cal} / A	0.019810		previous	% shift
R_{Cal} / Ω	**49.9930**		49.996000	-0.00596
Entered new R_{Load} and R_{Cal} into CPCInstr.par file on:		49.9929	(Entered at IAEA Using Calculator)	
Condtions of System prior to aligning the Digital Integrator				
DAC 3001 = 2.5 V				
DAC 3002 = see chart				
K17 Relay Energized				
Voltage-to-Frequency Convertor Alignment Check 09-14-2016, "As Found" same as "As left", i.e., no change				
DVM across load = 0.072e-3 V				
	As Found		**As Left**	
Readout	999.8	Hz	1000.0	Hz
Offset	1000.1	Hz	1000.1	Hz
Clock	10,000.11	Hz	10,000.11	Hz
34401A Load Voltage / V	**HP53132A / Hz**	**With offset subtracted / Hz**	**Theoretical (without offset) / Hz**	**% Non Linearity**
As Found				
2.0000	20996.7	19996.900	20000.000	-0.015
As Left				
2.00000	21000.3	20000.200	20000.000	0.001

Savannah River National Laboratory Measurement & Test Equipment			
1) ASL Standard Resistor Model RR100, SRNL M&TE# 54762, Expires 10/14/2022			
2) Agilent 34401A Digital Volt Meter, SRNL M&TE# 49750, Expires 2/3/23			
3) Agilent 53131A Universal Counter, SRNL M&TE# 53520, Expires 7/29/2022			
4) Omega HH41 Digital Thermometer, M&TE#49662, Exp 4/4/2023			
5) Fluke 87, M&TE #510137, Exp 3/10/2023			

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