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QUALIFICATION OF THE NIPPON INSTRUMENTATION FOR USE IN MEASURING MERCURY AT THE DEFENSE WASTE PROCESSING FACILITY

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EXECUTIVE SUMMARY

The Nippon Mercury/RA-3000 system installed in 221-S M-14 has been qualified for use. The qualification was a side-by-side comparison of the Nippon Mercury/RA-3000 system with the currently used Bacharach Mercury Analyzer. The side-by-side testing included standards for instrument calibration verifications, spiked samples and unspiked samples. The standards were traceable back to the National Institute of Standards and Technology (NIST). The side-by-side work included the analysis of Sludge Receipt and Adjustment Tank (SRAT) Receipt, SRAT Product, and Slurry Mix Evaporator (SME) samples. With the qualification of the Nippon Mercury/RA-3000 system in M-14, the DWPF lab will be able to perform a head to head comparison of a second Nippon Mercury/RA-3000 system once the system is installed.

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LIST OF ABBREVIATIONS

DWPF	Defense Waste Processing Facility
Hg	Mercury
HLW	High Level Waste
JMP	Statistical software package from SAS Institute, Inc. [3]
NIST	National Institute of Standards and Technology
ppm	Parts per million
SME	Slurry Mix Evaporator
SRAT	Slurry Receipt and Adjustment Tank
SRNL	Savannah River National Laboratory
TTR	Technical Task Request
TT&QA	Task Technical and Quality Assurance

1.0 INTRODUCTION

The Defense Waste Processing Facility (DWPF) analyzes receipt and product samples from the Sludge Receipt and Adjustment Tank (SRAT) to determine the mercury (Hg) concentration in the sludge slurry. The SRAT receipt is typically sampled and analyzed for the first ten SRAT batches of a new sludge batch to obtain an average Hg concentration. This average Hg concentration is then used to determine the amount of steam stripping required during the concentration/reflux step of the SRAT cycle to achieve a less than 0.6 wt% Hg in the SRAT product solids. After processing is complete, the SRAT product is sampled and analyzed for mercury to ensure that the mercury concentration does not exceed the 0.45 wt% limit in the Slurry Mix Evaporator (SME).

The DWPF Laboratory utilizes Bacharach Analyzers to support these Hg analyses at this facility. These analyzers are more than 10 years old, and they are no longer supported by the manufacturer. Due to these difficulties, the Bacharach Analyzers are to be replaced by new Nippon Mercury/RA-3000 systems. DWPF issued a Technical Task Request (TTR) [1] for the Savannah River National Laboratory (SRNL) to assist in the qualification of the new systems. SRNL prepared a task technical and quality assurance (TT&QA) plan [2] that outlined the activities that are necessary and sufficient to meet the objectives of the TTR. In addition, TT&QA plan also included a test plan that provided guidance to the DWPF Lab in collecting the data needed to qualify the new Nippon Mercury/RA-3000 systems.

2.0 EXPERIMENTAL PROCEDURE

The qualification of the Nippon Mercury/RA-3000 system installed in 221-S M-14 for use in the measurement of mercury for SRAT receipt and product samples will rely on side-by-side measurements that were conducted by the DWPF Lab. The side-by-side comparisons were performed for samples from SRAT batches and some limited samples of the SME product. In addition, during the processing of the SRAT batches (i.e., after the sampling of the SRAT Receipt but before the sampling of the SRAT Product), some intermediate samples were taken. These samples are labeled as "Conflux" samples.

Typically, four samples were taken from each batch of material for which the mercury content was measured. The samples were prepared for analysis: two samples "as-is" and two spiked with a mercury standard. The mercury concentrations of the two "as-is" samples were measured by the Bacharach Analyzer as done today. In addition, the same samples were measured by the Nippon Mercury/RA-3000 system. It should be noted that the Nippon instrument, which operates at lower Hg concentrations than the Bacharach, required an additional dilution before analysis. Sample identifiers were established and maintained so that the Bacharach and Nippon measurements for each sample may be paired appropriately. This is illustrated in Table 1, where the X's, Y's, A's and B's represent the measurements to be generated by DWPF Lab. Note that the X and Y values represent the mercury values in parts per million (ppm), the values of interest, from the "as-is" samples while the A and B values represent the percent (%) recoveries of mercury (Hg) for the spiked samples for the two instruments, respectively; % Hg recovery is the determination of interest that the DWPF Lab performs for the spiked samples.

Table 1. Illustration of Mercury (Hg) Measurements for a Given SRAT Batch

Sample ID	Type	Bacharach Hg (ppm)	Nippon/RA-3000 Hg (ppm)	Bacharach % Recovery	Nippon/RA-3000 % Recovery
Sample 1	As-is	X_1	Y_1	Not Appropriate	Not Appropriate
Sample 2	As-is	X_2	Y_2	Not Appropriate	Not Appropriate
Sample 3	Spiked	Not Reported	Not Reported	A_1	B_1
Sample 4	Spiked	Not Reported	Not Reported	A_2	B_2

Following this plan led to the generation by the DWPF Lab of the mercury measurements provided in Table A1 in the Appendix for the “as-is” samples and of the percent recovery values in Table A2 in the Appendix. Table 2 presents the spike recovery data from the two spiked samples analyzed along with the "as-is" samples. The established spike recovery limits for the method are 65 - 135%. While the spike recovery data show some variation, the recoveries are within the established range and are generally consistent between the instruments suggesting that spike recovery is not an instrument problem, but may reflect something inherent within the method. Recent mercury method work performed in SRNL and DWPF [3] concluded that "the current spike protocol for quality control does not appear to be effective. The spike failures do not correlate with the analyses of the unspiked samples for reasons that are still not known." Again, it is believed that the spike recovery problems are more of a method problem, rather than an instrument problem. Percent recoveries of check standards measured by the instruments are provided in Table A3 in the Appendix. The check standards were used in the block of measurements conducted by the DWPF Lab: a check standard early in the block (labeled as “first”) and a check standard late in the block (labeled as “second”). The standards were traceable back to the National Institute of Standards and Technology (NIST).

The measurements in Tables A1 through A3 provide the basis for the analysis presented in this report. The two rows of Table A1 corresponding to measurements of SME Product samples are shaded to highlight the limited amount of data from this type of material. Due to the limited data and the fact that the mercury content of this material is not typically measured, these data are not included in the statistical analyses that follow. A row corresponding to a spiked sample of SRAT Product Batch 525 is shaded in Table A2 to indicate the unacceptable value (-48.4%) generated by the Bacharach instrument. This row of data is not included in the analyses that follow.

3.0 Statistical Analysis

In this section, the statistical comparisons of the measurements generated by the two mercury instruments are presented. JMP Version 7.0.2 [4] was used to perform these analyses. Of primary interest are investigations into any relative bias between the two measurement systems and into a comparison of their precisions. Each of the three data tables is investigated in turn over the next three sections that follow to address the issues of concern.

3.1 Comparisons of the Two Instruments Based Upon the Hg Measurements of Samples

Exhibit A1 in the Appendix provides a plot of the Hg measurements of Table A1. The data are grouped by Type of Batch. The SME Product results are shown in this plot. While they suggest no problem in the performance of the two instruments, due to the limited amount of data for the type of material, these measurements are not included in the statistical analyses that follow. The groupings of the other measurement data provide the basis for the evaluation of the two instruments using a “paired-sample” analysis.

Exhibit A2 in the Appendix provides this comparison for the measurements from Conflux samples. Included in this exhibit is a plot of the Nippon measurements versus the Bacharach measurements along with a fitted model that assesses the relative bias between the measurements of the two instruments. That bias would be considered statistically significant if the "Prob>|t|" value in the "Parameter Estimates" table is 0.05 or smaller. For this analysis, the value is 0.0606, so it would not be considered significant at the 5% level. Also included in this exhibit are the results of a direct "Matched Pairs" analysis from JMP. That analysis indicates the same "Prob>|t|" value of 0.0606, but it also provides a 95% confidence interval for the average difference between the two instruments for this set of measurements. That interval is given by the two entries: Upper95% and Lower95%. These values yield the interval -204.22 to 5.26191, which indicates that the relative bias in ppm between the two instruments for these Conflux measurements is bounded by 204.22 ppm with at least 95% confidence.

Exhibits A3 and A4 in the Appendix provide similar results for the measurements of the SRAT Product and SRAT Receipt samples, respectively. There is no indication of a statistically significant bias (at the 5% significance level) between the two instruments for either of these two sets of measurements. For the SRAT Product samples, the relative bias between the two instruments is bounded by 68 ppm while the bias is bounded by 185.4 ppm for the SRAT Receipt samples.

One the aspect of the results from Exhibits A2 through A4 that is notable is the pattern of scatter around the fitted line for Exhibit A2. The smallest Hg measurements of this plot fall above this line while the two largest Hg values fall below this line. This indicates that for the Conflux samples, the Nippon yielded larger values than the Bacharach for lower Hg concentrations and smaller measurements than the Bacharach at higher Hg concentrations. This pattern was not evident in the SRAT Product and SRAT Receipt samples.

3.2 Comparisons of the Two Instruments Based Upon the Hg Recovery Values of Samples

Exhibits A5, A6, and A7 in the Appendix provide analyses of the percent recovery values of the Conflux, SRAT Product, and SRAT Receipt samples, respectively, that are similar to the analyses of the Hg measurements presented in the previous section. Based upon the results shown in these exhibits, the only indication of a statistically significant (at the 5% level) bias between the two instruments is for the SRAT Product sample measurements. Even so, the bias for this difference is bounded by 12.6% with at least 95% confidence. While not statistically significant at the 5% level, the bias between the two instruments is bounded by 20.6% for the Conflux samples and by 16.7% for the SRAT Receipt samples.

3.3 Comparisons of the Two Instruments Based Upon the Hg Recovery Values of the Check Standards

Exhibit A8 in the Appendix provides a plot of and some summary statistics for the percent recovery values of the check standards that are presented in Table A3. The check standard measurements are grouped by instrument and by their place in sequence of measurements (the first is labeled as % Rec and the second as % Rec 2). Box plots and 95% confidence interval diamonds are provided for each set of measurements. Summary statistics (i.e., the sample mean, the sample standard deviation, the standard error of the mean (Std Err Mean), the lower and upper limits of the confidence interval, the minimum value, the maximum value, and the number of observations (Obs) in the group are also provided as part of the exhibit. A closer look at these summary statistics is provided in the following discussion.

Exhibit A9 in the Appendix provides a comparison between the measurements of the first check standard for the two instruments. Included in this exhibit, is a series of statistical tests for equality of variances for these measurements between the two instruments. Relying on the results of Levene's test indicates that there is no indication of a statistically significant (at the 5% level) difference in the variances of the measurements from the two instruments. The t-test for equality of means also shows no indication of a statistically significant (at the 5% level) difference between the two instruments. The 95% confidence interval indicates that the difference or bias between the two instruments is bounded by 4.2%.

Exhibit A10 in the Appendix provides a similar comparison between the measurements of the second check standard for the two instruments. For these data, Levene's test indicates that there is indication a statistically significant (at the 5% level) difference in the variances of the measurements from the two instruments with the measurements from the Nippon yielding a smaller variance. While the t-test for equality of means shows a statistically significant (at the 5% level) difference between the two instruments. The 95% confidence interval indicates that the difference or bias between the two instruments is bounded by 7.4% with the Nippon yielding, on average, a smaller % recovery value.

4.0 Conclusions and Recommendations

Based upon the results presented in this report, it is recommended that the Nippon Mercury/RA-3000 system installed in 221-S M-14 has been qualified for use. The qualification was a side-by-side comparison of the Nippon Mercury/RA-3000 system with the currently used Bacharach Mercury Analyzer. The side-by-side testing included standards for instrument calibration verifications, spiked samples and unspiked samples. The standards were traceable back to NIST. The side-by-side work included the analysis of SRAT Receipt, SRAT Product, and SME samples. With the qualification of the Nippon Mercury/RA-3000 system in M-14, the DWPF lab will be able to perform a head to head comparison of a second Nippon Mercury/RA-3000 system once the system is installed.

References

[1] Brown, AY, "Technical Task Request: Develop Qualification Test Plan and Result Analysis for DWPF Lab New Mercury Analyzer RA-3000," HLW/DWPF/TTR-2009-0020, Revision 0, April, 2009.

[2] Edwards, TB, "Task Technical & QA Plan: Develop a Qualification Test Plan and Provide a Results Analysis for the New Mercury Analyzer RA-3000 Systems at DWPF Lab," SRNL-RP-2009-00971, Revision 0, July, 2009.

[3] Coleman, CJ, et al., "Evaluation of Sample Preparation Methods for Mercury Determinations in DWPF Sludge," SRNS-STI-2009-00315, Revision 0, August, 2009.

[4] JMP Version 7.0.2, SAS Institute, Inc., Cary NC, 1989-2007.

Appendix: Tables and Exhibits

Table A1. Mercury Measurements (ppm) of Samples by Instrument

Type of Batch	Batch	Bacharach Analyzer Hg (ppm)	Nippon/RA-3000 Hg (ppm)
SRAT Receipt	524	2562.7	2462.7
SRAT Receipt	524	2673.4	2534.3
SRAT Receipt	525	2419.7	2443.2
SRAT Receipt	525	2360.7	2312.2
SRAT Receipt	526	2235.1	2247
SRAT Receipt	526	2344	2303.4
SRAT Receipt	527	2070	2059.5
SRAT Receipt	527	1883.2	1898.5
SRAT Product	524	762.3	715
SRAT Product	524	694.4	722.6
SRAT Product	525	1415.3	1361
SRAT Product	525	1438.7	1366.4
SRAT Product	526	175.5	172.9
SRAT Product	526	173.3	170.5
SRAT Product	527	421.4	641.3
SRAT Product	527	436.6	617.5
SRAT Product	534	1488.2	1403.4
SRAT Receipt	535	3771.1	4150.8
Conflux*	535a	2714.3	2380.3
SRAT Product	535	891.7	895.3
SRAT Receipt	536	3442.1	3343.8
Conflux*	536a	3139.1	2747.0
Conflux*	536b	2607.5	2727.0
Conflux*	536c	2306.7	2121.2
Conflux*	536d	1286.1	1254.0
Conflux*	536e	1036.1	1076.9
SRAT Product	536	770.0	723.5
SRAT Product	536**	771.0	628.0
SME Product	535	518.7	485.7
SRAT Product	534	1491.5	1369.0
SRAT Receipt	535	3918.7	3327.1
Conflux*	535a	2707.3	2546.7
SRAT Product	535	881.6	837.9
SRAT Receipt	536	3350.1	3365.5
Conflux*	536a	3152.7	2904.6
Conflux*	536b	2624.7	2523.7
Conflux*	536c	2355.9	2377.7
Conflux*	536d	1307.6	1349.9
Conflux*	536e	1018.7	1054.1
SRAT Product	536	766.5	754.3
SRAT Product	536**	.	.
SME Product	535	499.9	529.9

Table A2. Percent Recoveries of Spiked Samples by Instrument

Type of Batch	Batch	Bacharach % Recovery	Nippon/RA-3000 % Recovery
SRAT Receipt	524	62.2	60.9
SRAT Receipt	524	100.4	99.7
SRAT Receipt	525	109.6	105
SRAT Receipt	525	46.2	81.4
SRAT Receipt	526	98.3	94.6
SRAT Receipt	526	108	104.8
SRAT Receipt	527	75.2	99.8
SRAT Receipt	527	77	86.9
SRAT Product	524	80.8	98.6
SRAT Product	524	84.1	110.7
SRAT Product	525	-48.4	107.4
SRAT Product	525	81.9	83
SRAT Product	526	66.1	70.8
SRAT Product	526	70	66.4
SRAT Product	527	52	73.6
SRAT Product	527	65.6	68.4
SRAT Product	534	111.8	103.0
SRAT Receipt	535	69.5	75.2
Conflux*	535a	36.7	76.0
SRAT Product	535	80.1	82.7
SRAT Receipt	536	66.1	79.1
Conflux*	536a	78.3	102.0
Conflux*	536b	72.5	64.2
Conflux*	536c	80.3	91.7
Conflux*	536d	59.5	64.2
Conflux*	536e	64.0	58.7
SRAT Product	536	21.6	23.3
SRAT Product	536**	18.4	11.1
SME Product	535	89.1	101.6
SRAT Product	534	119.8	116.9
SRAT Receipt	535	71.6	97.3
Conflux*	535a	77.4	92.6
SRAT Product	535	68.0	81.7
SRAT Receipt	536	102.4	92.5
Conflux*	536a	38.2	80.2
Conflux*	536b	112.6	108.3
Conflux*	536c	81.7	87.2
Conflux*	536d	61.6	61.2
Conflux*	536e	72.3	57.7
SRAT Product	536	41.2	47.3
SRAT Product	536**	22.6	43.8
SME Product	535	87.3	100.3

Table A3. Percent Recoveries of Check Standards by Instrument

Instrument/Ck Std	Instrument	Sequence of Ck Std	% Recovery
Bacharach/first	Bacharach	Check Std Bach % Rec	95.5
Bacharach/first	Bacharach	Check Std Bach % Rec	96.7
Bacharach/first	Bacharach	Check Std Bach % Rec	106.4
Bacharach/first	Bacharach	Check Std Bach % Rec	94.0
Bacharach/first	Bacharach	Check Std Bach % Rec	97.6
Bacharach/first	Bacharach	Check Std Bach % Rec	95.0
Bacharach/first	Bacharach	Check Std Bach % Rec	100.3
Bacharach/first	Bacharach	Check Std Bach % Rec	99.2
Bacharach/first	Bacharach	Check Std Bach % Rec	94.9
Bacharach/first	Bacharach	Check Std Bach % Rec	97.6
Bacharach/first	Bacharach	Check Std Bach % Rec	99.1
Bacharach/first	Bacharach	Check Std Bach % Rec	97.4
Bacharach/first	Bacharach	Check Std Bach % Rec	91.7
Bacharach/second	Bacharach	Check Std Bach % Rec 2	95.9
Bacharach/second	Bacharach	Check Std Bach % Rec 2	105.9
Bacharach/second	Bacharach	Check Std Bach % Rec 2	103.8
Bacharach/second	Bacharach	Check Std Bach % Rec 2	104.4
Bacharach/second	Bacharach	Check Std Bach % Rec 2	92.8
Bacharach/second	Bacharach	Check Std Bach % Rec 2	97.0
Bacharach/second	Bacharach	Check Std Bach % Rec 2	98.0
Bacharach/second	Bacharach	Check Std Bach % Rec 2	96.8
Bacharach/second	Bacharach	Check Std Bach % Rec 2	101.4
Bacharach/second	Bacharach	Check Std Bach % Rec 2	96.3
Bacharach/second	Bacharach	Check Std Bach % Rec 2	102.8
Bacharach/second	Bacharach	Check Std Bach % Rec 2	97.7
Bacharach/second	Bacharach	Check Std Bach % Rec 2	95.7
Nippon/first	Nippon	Check Std Nippon % Rec	97.4
Nippon/first	Nippon	Check Std Nippon % Rec	90.9
Nippon/first	Nippon	Check Std Nippon % Rec	99.1
Nippon/first	Nippon	Check Std Nippon % Rec	98.8
Nippon/first	Nippon	Check Std Nippon % Rec	96.3
Nippon/first	Nippon	Check Std Nippon % Rec	98.9
Nippon/first	Nippon	Check Std Nippon % Rec	96.4
Nippon/first	Nippon	Check Std Nippon % Rec	94.2
Nippon/first	Nippon	Check Std Nippon % Rec	95.1
Nippon/first	Nippon	Check Std Nippon % Rec	98.7
Nippon/first	Nippon	Check Std Nippon % Rec	93.4
Nippon/first	Nippon	Check Std Nippon % Rec	93.1
Nippon/first	Nippon	Check Std Nippon % Rec	92.5
Nippon/second	Nippon	Check Std Nippon % Rec 2	96.2
Nippon/second	Nippon	Check Std Nippon % Rec 2	100.4
Nippon/second	Nippon	Check Std Nippon % Rec 2	91.6
Nippon/second	Nippon	Check Std Nippon % Rec 2	99.1
Nippon/second	Nippon	Check Std Nippon % Rec 2	92.3
Nippon/second	Nippon	Check Std Nippon % Rec 2	93.1
Nippon/second	Nippon	Check Std Nippon % Rec 2	92.2
Nippon/second	Nippon	Check Std Nippon % Rec 2	92.1
Nippon/second	Nippon	Check Std Nippon % Rec 2	94.4
Nippon/second	Nippon	Check Std Nippon % Rec 2	94.7
Nippon/second	Nippon	Check Std Nippon % Rec 2	94.7
Nippon/second	Nippon	Check Std Nippon % Rec 2	94.4
Nippon/second	Nippon	Check Std Nippon % Rec 2	93.1

Exhibit A1. Variability Chart for Hg (ppm) Measurements

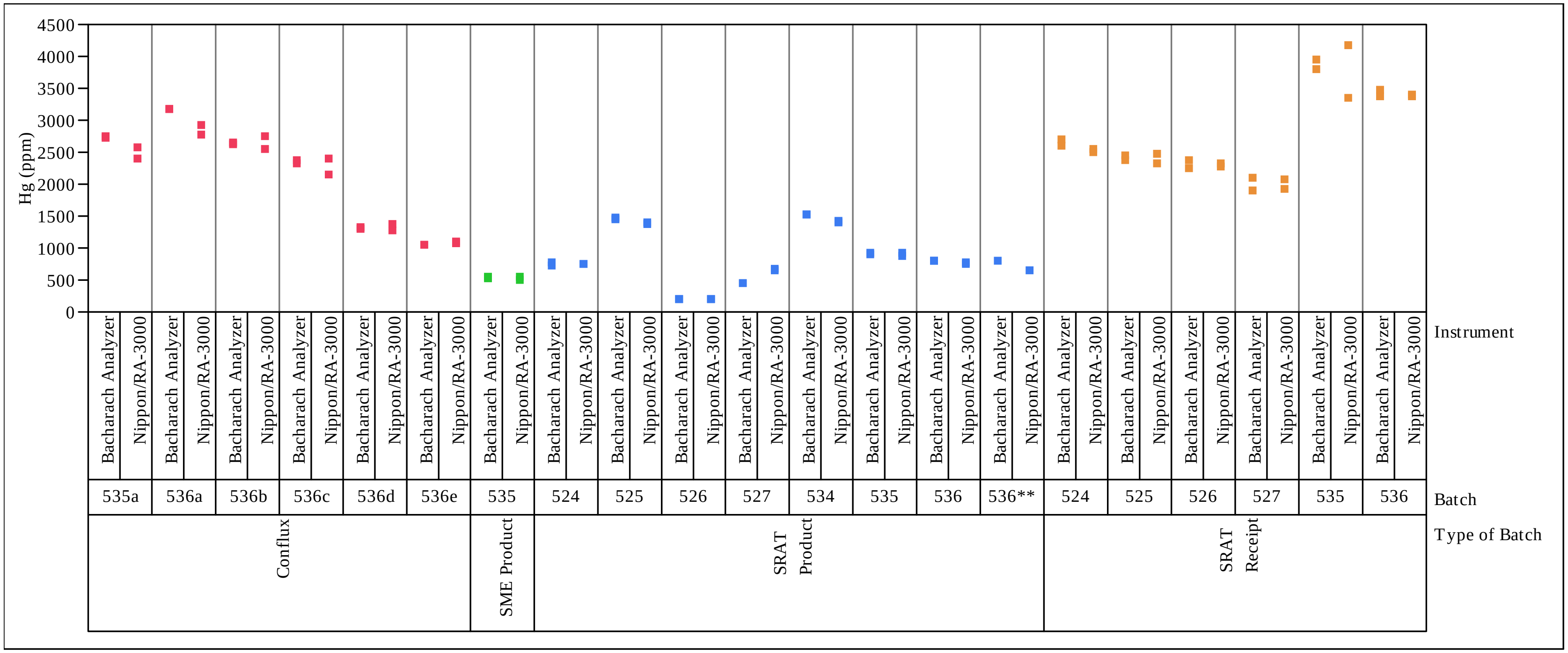
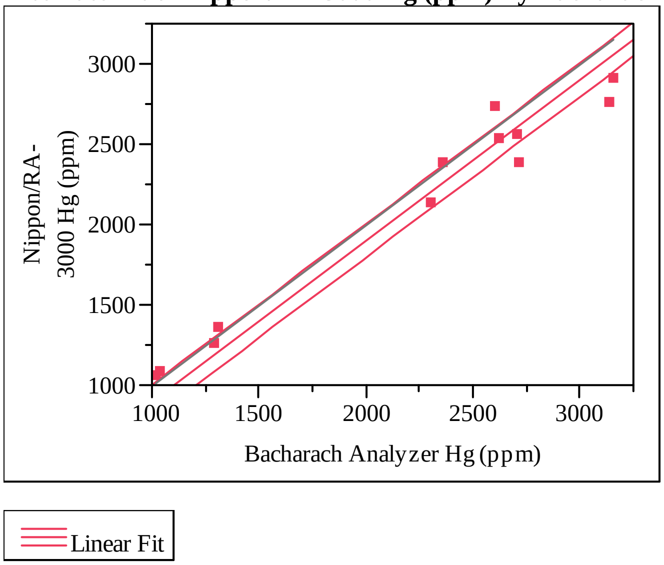


Exhibit A2. Paired Comparison of Hg Measurements for Conflux Samples

Bivariate Fit of Nippon/RA-3000 Hg (ppm) By Bacharach Analyzer Hg (ppm) Type of Batch=Conflux



Linear Fit
Nippon/RA-3000 Hg (ppm) = -99.477 + 1*Bacharach Analyzer Hg (ppm)

Summary of Fit

RSquare	.
RSquare Adj	.
Root Mean Square Error	164.8472
Mean of Response	2088.591
Observations (or Sum Wgts)	12

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	0	.	0.0	.
Error	11	298920.55	27174.6	**Prob > F**
C. Total	11	.		.

Parameter Estimates

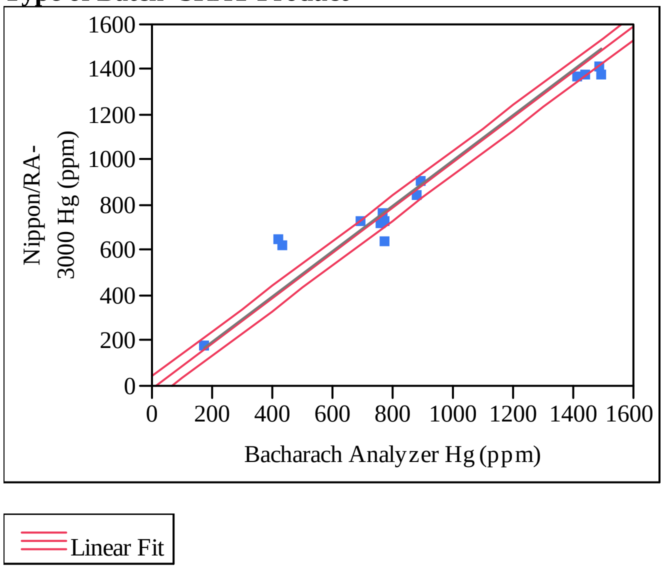
Term		Estimate	Std Error	t Ratio	Prob>|t|
Intercept		-99.477	47.58728	-2.09	0.0606
Bacharach Analyzer Hg (ppm)	Constrained	1	0	.	.

Matched Pairs Type of Batch=Conflux*
Difference: Nippon/RA-3000 Hg (ppm)-Bacharach Analyzer Hg (ppm)

Nippon/RA-3000 Hg (ppm)	2088.59	t-Ratio	-2.09041
Bacharach Analyzer Hg (ppm)	2188.07	DF	11
Mean Difference	-99.477	Prob > |t|	0.0606
Std Error	47.5873	Prob > t	0.9697
Upper95%	5.26191	Prob < t	0.0303
Lower95%	-204.22		
N	12		
Correlation	0.98482		

Exhibit A3. Paired Comparison of Hg Measurements for SRAT Product Samples

Bivariate Fit of Nippon/RA-3000 Hg (ppm) By Bacharach Analyzer Hg (ppm)
Type of Batch=SRAT Product



Linear Fit
Nippon/RA-3000 Hg (ppm) = -13.29064 + 1*Bacharach Analyzer Hg (ppm)

Summary of Fit

RSquare	.
RSquare Adj	.
Root Mean Square Error	98.75381
Mean of Response	825.25
Observations (or Sum Wgts)	15

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	0	.	0.00	.
Error	14	136532.42	9752.32	**Prob > F**
C. Total	14	.		.

Parameter Estimates

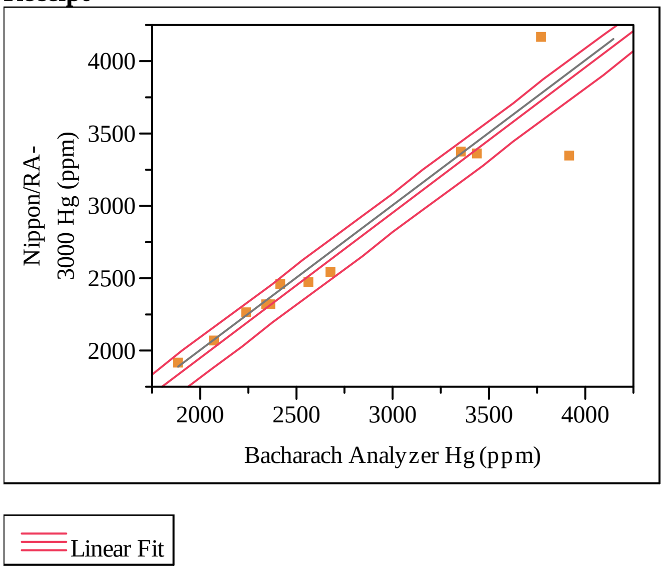
Term		Estimate	Std Error	t Ratio	Prob>|t|
Intercept		-13.29064	25.49813	-0.52	0.6103
Bacharach Analyzer Hg (ppm)	Constrained	1	0	.	.

Matched Pairs Type of Batch=SRAT Product
Difference: Nippon/RA-3000 Hg (ppm)-Bacharach Analyzer Hg (ppm)

Nippon/RA-3000 Hg (ppm)	825.25	t-Ratio	-0.52124
Bacharach Analyzer Hg (ppm)	838.541	DF	14
Mean Difference	-13.291	Prob > |t|	0.6103
Std Error	25.4981	Prob > t	0.6948
Upper95%	41.3974	Prob < t	0.3052
Lower95%	-67.979		
N	15		
Correlation	0.97942		

Exhibit A4. Paired Comparison of Hg Measurements for SRAT Receipt Samples

Bivariate Fit of Nippon/RA-3000 Hg (ppm) By Bacharach Analyzer Hg (ppm) Type of Batch=SRAT Receipt



Linear Fit
Nippon/RA-3000 Hg (ppm) = -48.57432 + 1*Bacharach Analyzer Hg (ppm)

Summary of Fit

RSquare	.
RSquare Adj	.
Root Mean Square Error	215.3453
Mean of Response	2703.995
Observations (or Sum Wgts)	12

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	0	.	0.0	.
Error	11	510109.49	46373.6	**Prob > F**
C. Total	11	.		.

Parameter Estimates

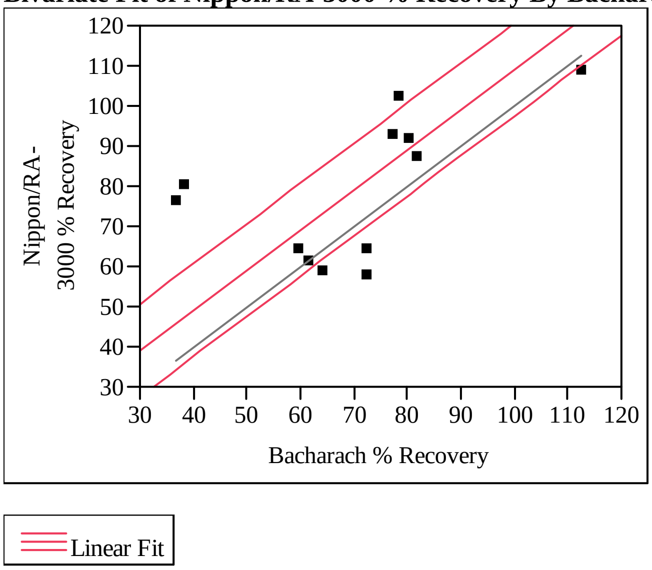
Term		Estimate	Std Error	t Ratio	Prob>|t|
Intercept		-48.57432	62.16483	-0.78	0.4511
Bacharach Analyzer Hg (ppm)	Constrained	1	0	.	.

Matched Pairs Type of Batch=SRAT Receipt
Difference: Nippon/RA-3000 Hg (ppm)-Bacharach Analyzer Hg (ppm)

Nippon/RA-3000 Hg (ppm)	2703.99	t-Ratio	-0.78138
Bacharach Analyzer Hg (ppm)	2752.57	DF	11
Mean Difference	-48.574	Prob > |t|	0.4511
Std Error	62.1648	Prob > t	0.7745
Upper95%	88.2495	Prob < t	0.2255
Lower95%	-185.4		
N	12		
Correlation	0.95037		

Exhibit A5. Paired Comparison of % Recovery Values for the Conflux Samples

Bivariate Fit of Nippon/RA-3000 % Recovery By Bacharach % Recovery Type of Batch=Conflux



Linear Fit
Nippon/RA-3000 % Recovery = 9.0855966 + 1*Bacharach % Recovery

Summary of Fit

RSquare	.
RSquare Adj	.
Root Mean Square Error	18.16991
Mean of Response	78.67035
Observations (or Sum Wgts)	12

Analysis of Variance

Source	**DF**	**Sum of Squares**	**Mean Square**	**F Ratio**
Model	0	.	0.000	.
Error	11	3631.6018	330.146	**Prob > F**
C. Total	11	.		.

Parameter Estimates

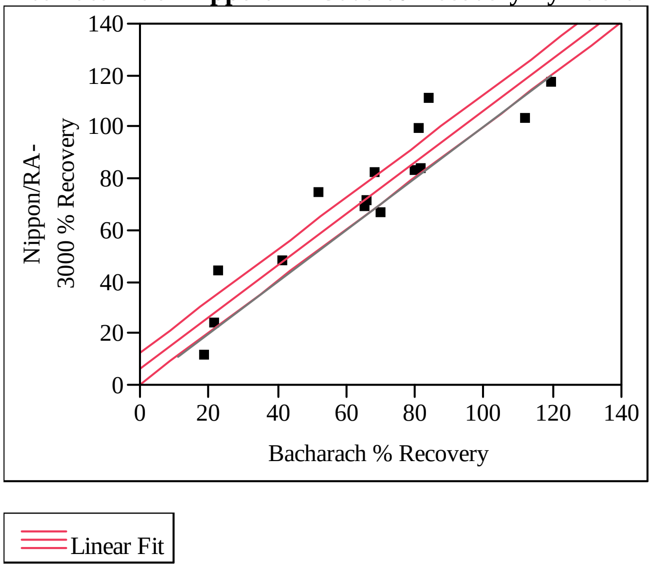
Term		**Estimate**	**Std Error**	**t Ratio**	**Prob>|t|**
Intercept		9.0855966	5.245201	1.73	0.1112
Bacharach % Recovery	Constrained	1	0	.	.

Matched Pairs Type of Batch=Conflux
Difference: Nippon/RA-3000 % Recovery-Bacharach % Recovery

Nippon/RA-3000 % Recovery	78.6703	t-Ratio	1.732173
Bacharach % Recovery	69.5848	DF	11
Mean Difference	9.0856	Prob > |t|	0.1112
Std Error	5.2452	Prob > t	0.0556
Upper95%	20.6302	Prob < t	0.9444
Lower95%	-2.459		
N	12		
Correlation	0.54908		

Exhibit A6. Paired Comparison of % Recovery Values for the SRAT Product Samples

Bivariate Fit of Nippon/RA-3000 % Recovery By Bacharach % Recovery Type of Batch=SRAT Product



Linear Fit
Nippon/RA-3000 % Recovery = 6.4778938 + 1*Bacharach % Recovery

Summary of Fit

RSquare	.
RSquare Adj	.
Root Mean Square Error	11.10362
Mean of Response	72.08514
Observations (or Sum Wgts)	15

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	0	.	0.000	.
Error	14	1726.0644	123.290	**Prob > F**
C. Total	14	.		.

Parameter Estimates

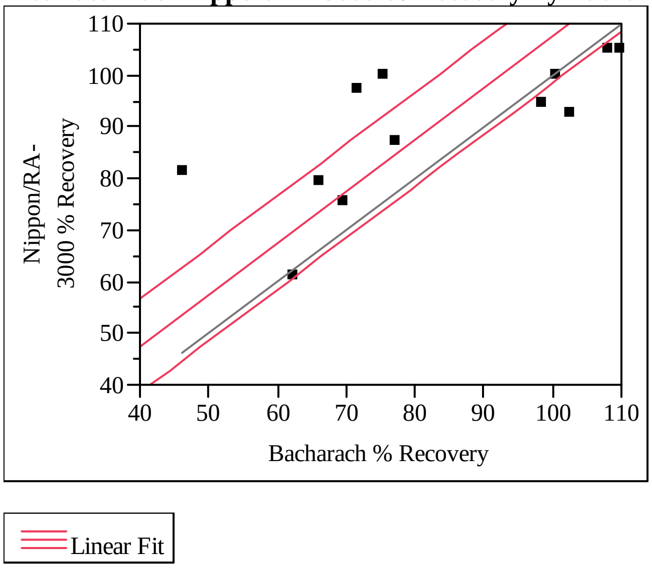
Term		Estimate	Std Error	t Ratio	Prob>|t|
Intercept		6.4778938	2.866942	2.26	0.0403
Bacharach % Recovery	Constrained	1	0	.	.

Matched Pairs Type of Batch=SRAT Product
Difference: Nippon/RA-3000 % Recovery-Bacharach % Recovery

Nippon/RA-3000 % Recovery	72.0851	t-Ratio	2.259514
Bacharach % Recovery	65.6072	DF	14
Mean Difference	6.47789	Prob > |t|	0.0403
Std Error	2.86694	Prob > t	0.0202
Upper95%	12.6269	Prob < t	0.9798
Lower95%	0.32892		
N	15		
Correlation	0.9336		

Exhibit A7. Paired Comparison of % Recovery Values for the SRAT Receipt Samples

Bivariate Fit of Nippon/RA-3000 % Recovery By Bacharach % Recovery Type of Batch=SRAT Receipt



Linear Fit

Nippon/RA-3000 % Recovery = 7.5645302 + 1*Bacharach % Recovery

Summary of Fit

RSquare	.
RSquare Adj	.
Root Mean Square Error	14.34575
Mean of Response	89.77153
Observations (or Sum Wgts)	12

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	0	.	0.000	.
Error	11	2263.8068	205.801	**Prob > F**
C. Total	11	.		.

Parameter Estimates

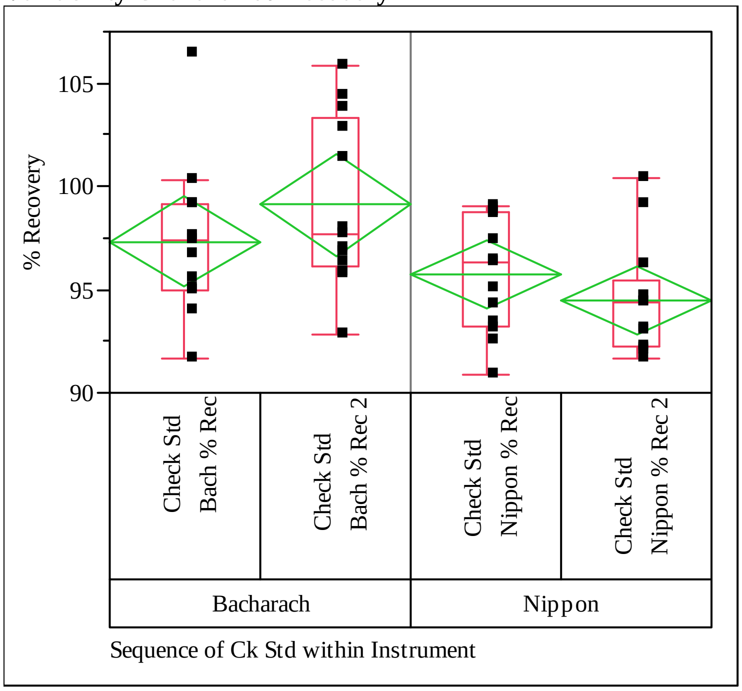
Term		Estimate	Std Error	t Ratio	Prob>|t|
Intercept		7.5645302	4.141262	1.83	0.0950
Bacharach % Recovery	Constrained	1	0	.	.

Matched Pairs Type of Batch=SRAT Receipt
Difference: Nippon/RA-3000 % Recovery-Bacharach % Recovery

Nippon/RA-3000 % Recovery	89.7715	t-Ratio	1.826624
Bacharach % Recovery	82.207	DF	11
Mean Difference	7.56453	Prob > |t|	0.0950
Std Error	4.14126	Prob > t	0.0475
Upper95%	16.6794	Prob < t	0.9525
Lower95%	-1.5503		
N	12		
Correlation	0.72557		

Exhibit A8. Evaluation of the % Recovery Values for the Check Standards

Variability Chart for % Recovery

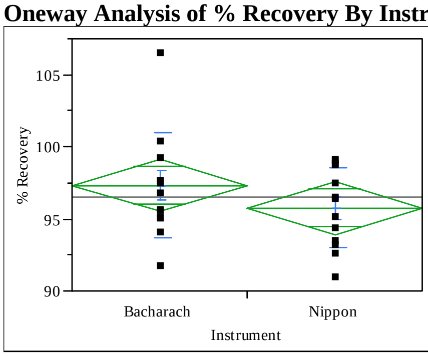


Variability Summary for % Recovery

	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%	Minimum	Maximum	Obs
Instrument[Bacharach]	98.22066	3.866269	0.758238	96.65904	99.78228	91.65	106.416	26
Instrument[Nippon]	95.11058	2.756131	0.540522	93.99735	96.2238	90.87	100.355	26
Instrument[Bacharach] Sequence of Ck Std[Check Std Bach % Rec]	97.33871	3.608543	1.00083	95.15809	99.51933	91.65	106.416	13
Instrument[Bacharach] Sequence of Ck Std[Check Std Bach % Rec 2]	99.10262	4.053989	1.124374	96.65281	101.5524	92.8	105.854	13
Instrument[Nippon] Sequence of Ck Std[Check Std Nippon % Rec]	95.75	2.763416	0.766434	94.08008	97.41992	90.87	99.05	13
Instrument[Nippon] Sequence of Ck Std[Check Std Nippon % Rec 2]	94.47115	2.702444	0.749523	92.83808	96.10422	91.635	100.355	13

Exhibit A9. Evaluation of the % Recovery Values for the First Check Standards

Oneway Analysis of % Recovery By Instrument First/Second=First



Oneway Anova
Summary of Fit

Rsquare	0.062073
Adj Rsquare	0.022992
Root Mean Square Error	3.21388
Mean of Response	96.54435
Observations (or Sum Wgts)	26

t Test
Nippon-Bacharach
Assuming equal variances

Difference	-1.5887	t Ratio	-1.26029
Std Err Dif	1.2606	DF	24
Upper CL Dif	1.0130	Prob > |t|	0.2197
Lower CL Dif	-4.1904	Prob > t	0.8902
Confidence	0.95	Prob < t	0.1098

Analysis of Variance

Source	DF	Sum of Squares	Mean Square	F Ratio	Prob > F
Instrument	1	16.40595	16.4059	1.5883	0.2197
Error	24	247.89661	10.3290		
C. Total	25	264.30256			

Means for Oneway Anova

Level	Number	Mean	Std Error	Lower 95%	Upper 95%
Bacharach	13	97.3387	0.89137	95.499	99.178
Nippon	13	95.7500	0.89137	93.910	97.590

Std Error uses a pooled estimate of error variance

Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Bacharach	13	97.3387	3.60854	1.0008	95.158	99.519
Nippon	13	95.7500	2.76342	0.7664	94.080	97.420

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Bacharach	13	3.608543	2.492346	2.495477
Nippon	13	2.763416	2.349231	2.315000

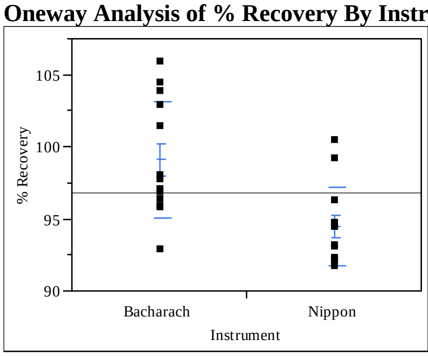
Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	0.5219	1	24	0.4770
Brown-Forsythe	0.0502	1	24	0.8247
Levene	0.0335	1	24	0.8563
Bartlett	0.8107	1	.	0.3679
F Test 2-sided	1.7052	12	12	0.3680

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
1.5883	1	22.473	0.2205

Exhibit A10. Evaluation of the % Recovery Values for the Second Check Standards

Oneway Analysis of % Recovery By Instrument First/Second=Second



Means and Std Deviations

Level	Number	Mean	Std Dev	Std Err Mean	Lower 95%	Upper 95%
Bacharach	13	99.1026	4.05399	1.1244	96.653	101.55
Nippon	13	94.4712	2.70244	0.7495	92.838	96.10

t Test
Nippon-Bacharach
Assuming unequal variances

Difference	-4.6315	t Ratio	-3.42742
Std Err Dif	1.3513	DF	20.90627
Upper CL Dif	-1.8205	Prob > |t|	0.0025
Lower CL Dif	-7.4424	Prob > t	0.9987
Confidence	0.95	Prob < t	0.0013

Tests that the Variances are Equal

Level	Count	Std Dev	MeanAbsDif to Mean	MeanAbsDif to Median
Bacharach	13	4.053989	3.496019	3.231231
Nippon	13	2.702444	1.957574	1.943462

Test	F Ratio	DFNum	DFDen	p-Value
O'Brien[.5]	2.7022	1	24	0.1132
Brown-Forsythe	2.0515	1	24	0.1649
Levene	4.8485	1	24	0.0375
Bartlett	1.8449	1	.	0.1744
F Test 2-sided	2.2504	12	12	0.1744

Welch Anova testing Means Equal, allowing Std Devs Not Equal

F Ratio	DFNum	DFDen	Prob > F
11.7472	1	20.906	0.0025

Distribution:

Name:	Location:
Sharon Marra	773-A
Connie Herman	999-W
Charles J. Coleman	773-A
Clint Gregory	773-A
Lori Chandler	773-A
Patricia Lee	703-41A
Gene Shine	703-41A
Damon R. Click	773-A
L. Curtis Johnson	773-A
Michael Stone	999-W
David Peeler	999-W
Tommy Edwards	999-W
Kevin Fox	999-W
Fabienne Johnson	999-W
Charles Crawford	773-42A
David Best	999-W
John Occhipinti	704-S
Jonathan Bricker	704-27S
John Iaukea	704-30S
Aaron Staub	704-27S
Jeff Ray	704-S
Robert Hinds	704-S
Terri Fellinger	704-26S
Michael J. Hart	210-S
Roger N. Mahannah	704-28S
Michael T. Feller	704-28S
Omar Cardona-Quiles	704-24S
Amanda Shafer	704-27S
Mason Clark	704-27S
Helen Pittman	704-27S
Hank Elder	704-24S
Bill Holtzscheiter	704-15S
Pat Vaughan	773-41A