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AN ANALYSIS OF SHIPPER-RECEIVER DATA
FOR PLUTONIUM NITRATE AND
PLUTONIUM OXIDE

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AN ANALYSIS OF SHIPPER-RECEIVER DATA
FOR PLUTONIUM NITRATE AND PLUTONIUM OXIDE

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January 15, 1973

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AN ANALYSIS AND DESCRIPTION OF SHIPPER-RECEIVER DATA
FOR PLUTONIUM NITRATE AND PLUTONIUM DIOXIDE

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AN ANALYSIS OF SHIPPER-RECEIVER DATA FOR PLUTONIUM NITRATE AND PLUTONIUM OXIDE

I. INTRODUCTION

In recent years, a number [1] - [4] of nuclear materials safeguards studies have been aimed at assessing the sensitivity with which materials accounting could detect a loss of material from the accounting records. In these studies, heavy emphasis was given to estimating the measurement errors associated with the accounting terms "material unaccounted for" (MUF) and "shipper-receiver difference" (S/R difference). The measurement uncertainties associated with those terms define the limiting sensitivity of materials accounting as a safeguards tool. In this sense, numerical values for measurement uncertainties form the "building blocks" in systems studies aimed at evaluating and/or improving the safeguards effectiveness of materials accounting.

Two materials of high strategic value whose associated measurement errors play an important role in the accountancy of nuclear materials are plutonium nitrate and plutonium oxide. Because of the increasing importance of these materials to nuclear materials safeguards, a study was undertaken to assess recent (1969-1971) shipper-receiver measurement experience for these materials. Although such experience does not yield the same detailed insight as a controlled experiment, it does provide an estimate of the actual measurement performance.

The purpose of this report is to summarize recent shipper-receiver experience for those materials. The intent is to represent that experience by "pooled" precisions and ranges of precisions for the various types of measurements which are made on $\text{Pu}(\text{NO}_3)_4$ and PuO_2 for accountability purposes. The data are coded to direct attention to a population of facilities rather than particular facilities. Comparisons are made "between" and "within" data sets to explain discrepancies and to determine if the precisions shown by various measurers are significantly different. However, the aim is to present the results simply as historical experience and no endeavor is made to draw technical conclusions as to whether the results represent the capability (best state of the art) of measurement or even what might be judged good performance.

II. SUMMARY

Recent (1969-1971) shipper-receiver measurement data for plutonium nitrate and plutonium oxide were obtained from various sources at the Hanford Complex. These included official transfer forms (741's), contractor and licensee measurement reports, and field office records. A one-to-one pairing was made of all available measurement data reported by a shipper and receiver on the same transfer unit, e.g., Pu assay, specific gravity, gross and net weight on a bottle of $\text{Pu}(\text{NO}_3)_4$. An analysis of the data was performed. The main aspects of the data analysis and a summary of results are given in Table I for each material and measurement type.

TABLE I

SUMMARY OF S/R VARIABILITY
FOR $\text{Pu}(\text{NO}_3)_4$ AND PuO_2

Material	Measurement	Pooled % s.d.	Range--% s.d. of Precision	Significant Difference in % s.d.'s of Precision	Normality Results--(No. Sig.)/(No. Tested)	% s.d. of Bias	% s.d. of Process Variation	No. of Cases	Total S/R's
$\text{Pu}(\text{NO}_3)_4$	Specific Gravity	0.43%	0.26%-0.46%	--	0/2	0.14%	2.20%	2	71
	Pu Assay	0.49%	0.16%-0.61%	yes	6/8	0.21%	10.70%	9	245
	Net weights	0.11%	0.04%-0.15%	yes	0/3	0.60%*	5.60%	5	85
	^{238}Pu wt. %	10.9 %	1.0% -17.4%	yes	1/4	2.60%	25.2 %	5	205
	^{239}Pu wt. %	0.08%	0.07%-0.13%	brdrln	1/4	0.04%	1.5 %	5	190
	^{240}Pu wt. %	0.34%	0.05%-0.41%	yes	0.4	0.03%	1.1 %	5	186
	^{241}Pu wt. %	0.77%	0.50%-1.21%	brdrln	1/4	0.43%	6.3 %	5	179
	^{242}Pu wt. %	2.27%	0.46%-3.60%	yes	0/4	0.42%	10.7 %	5	147
PuO_2	Pu weight %	0.12%	0.06%-0.20%	no	0/6	0.63%*	0.28%	6	37
	Tare weights	0.26%*	0.08%-0.41%	--	0/2	0.56%	45.0 %	2	38
	Net weights	0.20%	0.02%-0.54%	yes	1/6	0.52%	23.7 %	7	139
	Total Pu	0.10%*	0.04%-0.28%	yes	2/4	0.29%	14.1 %	4	36
General	Gross weights	0.03%*	0.01%-1.05%	yes	3/8	0.16%	25.1 %	10	324
									1882

*See qualifying remarks on page 4 of this report.

The methods of data analysis are given in the Appendix, definitions are given in Section VI and the methods of pooling are given and discussed in Section V.

The results shown in Table I which are marked by an asterisk represent the removal of a nontypical result from the pooled precision or the case where a particular problem appeared to cause a relatively large bias. For example, the bias of 0.63 percent standard deviation on Pu weight percent for PuO_2 is believed to be largely due to moisture pick up by PuO_2 . In other cases, apparent biases reflect differences in operational handling procedure, such as the inclusion or noninclusion of protective coverings in weighing, rather than true measurement differences. In many cases, the prompt evaluation of shipper-receiver data can pinpoint the causes of such discrepancies so that corrective action can be initiated at an early date.

The pooled precisions shown for individual shipper or receiver measurements reflect the variability of individual statements about a measured property of a single transfer unit, e.g., the plutonium assay of a can of PuO_2 or a bottle of $\text{Pu}(\text{NO}_3)_4$ solution. Precisions shown for specific gravity, Pu assay of $\text{Pu}(\text{NO}_3)_4$ and Pu weight percent of PuO_2 include both analytical and sampling errors. In general, those precisions reflect the measurement of a single sample with either a single or more generally a duplicate analysis of the sample. Assuming sampling errors to be small, the values shown for measurement precisions could be multiplied by the $\sqrt{2}$ to approximate an upper limit for the precision of an individual analytical measurement.

The values shown for the limits of the range of precisions for a given measurement can be used as an estimate of currently attainable precisions. The lower limit would indicate "best" precision attained by some facilities whereas the upper limit would indicate a precision attainable by all facilities.

The precisions shown for isotopic composition generally reflect a single filament loading with 10 scans for each shipper or receiver mass concentration. The range of precisions reflected by the range of plutonium isotopes reflects the abundance of those isotopes rather than a true measurement variability. The precisions for the different mass concentrations are, as expected, inversely proportional to their abundance, e.g., ^{238}Pu and ^{242}Pu precisions reflect abundances of 0.03 and 0.19 weight percent, respectively. These and other aspects of the results are discussed in detail in Section III for each data set.

The measurement methods for the range of measurements used are described in Table II. No effort was made in this study to evaluate individual methods.

III. RESULTS IN DETAIL

The results for each of the 13 different material, measurement types given in Section II are given in more detail here. This includes a discussion of the results and the following;

1. the facilities involved where the facilities are coded as to identity (the first mentioned facility is always facility 1),
2. the number of S/R differences used in the set,
3. the average of facility 1,

TABLE II
MEASUREMENT METHODS USED FOR PLUTONIUM
NITRATE AND PLUTONIUM OXIDE S/R MEASUREMENTS

<u>Material</u>	<u>Measurement Component</u>	<u>Description of Method</u>
$\text{Pu}(\text{NO}_3)_4$	Specific Gravity	Weighed volume, e.g., 100 μ liter pipet.
"	Pu Assay	Volumetric titration after nitrate removal or after ion exchange separation.
"	Weighing	Precision balance to nearest 0.1 gram.
"	Sampling	Thief sample or circulating sampling after transfer of solution.
"	Isotopic Composition	Surface ionization mass spectrometry, including ^{238}Pu .
PuO_2	Weight %	Controlled potential coulometry or volumetric titration after dissolution and pretreatment.
"	Sampling	Core sampling of powder.
"	Weighing	Precision analytical type balance weighing to 0.1 gram or better.
"	Isotopic Composition	Same as above.

4. the average of facility 2,
5. the average of the two facilities,
6. the difference between the two facility averages $D = Av_1 - Av_2$,
7. the percent difference-- $100\% D/Av.$ where $Av. = 1/2(Av_1 + Av_2)$,
8. Student's t to test for significant differences in average values,
9. estimated standard deviation of measurement precision for a shipper or receiver value,
10. estimated standard deviation of measurement precision in percent for a shipper or receiver value,
11. the W value to test for significant departures from normally distributed values (these are given for $n \leq 25$),
12. the 95% critical value of W --(note that the 99 percentile value of W is smaller than the 95 percentile value),
13. the estimated bias standard deviation,
14. the estimated bias standard deviation in percent,
15. the estimated process standard deviation,
16. the estimated process standard deviation in percent,
17. the number of outliers,

and

18. the percent of S/R differences which are outliers.

$Pu(NO_3)_4$ - Specific Gravity

There are two sets of data from the same two facilities. The results are as follows.

1. There is a significant difference between the measurement precisions of the two sets of data. The percent standard deviations of precision for the individual shipper or receiver measurement are

	<u>% s.d.</u>	<u>n</u>
1	0.46%	55
2	0.26%	16

2. The pooled percent standard deviation of measurement precision is 0.43% so that the observed performance value here for measurement precision is about 1/2 percent or somewhat less.
3. Based on the second set of data, it appears that 1/4 percent is an attainable level for the relative standard deviation of precision.
4. The W statistic for one set of S/R Sp G differences indicates that there is no reason to assume that the differences are not normally distributed. No W statistic was computed for the other data set.
5. The percent standard deviation of bias is estimated at 0.14%.
6. The pooled process variability as estimated by the percent standard deviation is 2.2%, a range of 1.8-3.2%.
7. The results in more detail for the individual data sets are as follows.

Type of Material PuNO₃Characteristic Measured Specific GravityFacilities A,BData Sets

Facilities	A,B	A,B		
No. S/R Comparisons	55	16		
Ave. Facility 1	1.45725	1.4485		
Ave. Facility 2	1.45853	1.4527		
Ave. (1) & (2)	1.45789	1.4506		
Diff. (1) - (2)	-0.00127	-0.0042		
% Difference	-0.087%	-0.29%		
Student's t	-1.0	-3.21		
Est. Meas. s.d.	6.68×10^{-3}	3.69×10^{-3}		
Meas. s.d. in %	0.46%	0.26%		
W Value	--	0.917		
95% W Critical Value	--	0.887		
Est. Bias s.d.	0	2.81×10^{-3}		
Bias s.d. in %	0	0.194%		
Est. Process s.d.	2.64×10^{-2}	4.67×10^{-2}		
Process s.d. in %	1.81%	3.22%		
No. of Outliers	3	0		
% of Outliers	5.0	0		

Pu(NO₃)₄ - Pu Assay

Nine sets of data were analyzed involving 11 different facilities. The main conclusions are as follow.

1. The % standard deviations for measurement precision are significantly different from one data set to another. This is not due to a particularly large or particularly small standard deviation, but is due to a general lack of similarity. An ordering of % standard deviations of measurement precision from largest to smallest is

	<u>% s.d.</u>	<u>n</u>
1	0.612%	7
2	0.539%	12
3	0.538%	166
4	0.490%	6
5	0.288%	14
6	0.238%	8
7	0.221%	3
8	0.221%	19
9	0.162%	10

2. The pooled estimate of the measurement rel-standard deviation of precision is 0.488% so that the observed performance value here for measurement precision is about 1/2%.
3. Smaller rel-measurement standard deviations of 0.16% to 0.29% were attained for data sets of fairly good size and this range seems to represent an attainable level of measurement precision.
4. These differences, in general, tend to be non-normally distributed as gauged by the W test. Six out of the eight data sets where the W value was calculated were borderline cases of being non-normally distributed when tested at the 5% level. This

fact should cause no restrictions on the use of average-type statistics but should serve as a cautionary note when dealing with statistical tolerance statements and with other statistics which depend closely on the form of the distribution of the individual measurements. Other instances of closely distribution-dependent statistics are Bartlett's test and the tests for outliers.

5. For the observed biases, the pooled rel-bias standard deviation is 0.21%. Note that this value is similar to that of the capability level of the rel-standard deviation of measurement precision.
6. The pooled process variability rel-standard deviation is 10.7%. This indicates that care must be exercised in the mixing and sampling of product batches.
7. The results in more detail are as follows.

Type of Material PuNO₃
 Characteristic Measured Pu Assay
 Facilities A, B, C, D, E, F, G, H, I, J, K

	<u>Data Sets</u>			
Facilities	<u>A,B</u>	<u>B,C</u>	<u>B,D</u>	<u>E,F</u>
No. S/R Comparisons	<u>6</u>	<u>12</u>	<u>7</u>	<u>19</u>
Ave. Facility 1	<u>0.1250</u>	<u>0.13087</u>	<u>0.12986</u>	<u>0.1398</u>
Ave. Facility 2	<u>0.1252</u>	<u>0.13150</u>	<u>0.13060</u>	<u>0.1401</u>
Ave. (1) & (2)	<u>0.1251</u>	<u>0.131185</u>	<u>0.13023</u>	<u>0.13995</u>
Diff. (1) - (2)	<u>-0.0002</u>	<u>-0.00063</u>	<u>-0.00074</u>	<u>-0.00025</u>
% Difference	<u>-0.16%</u>	<u>-0.48%</u>	<u>-0.57%</u>	<u>-0.18%</u>
Student's t	<u>-0.5</u>	<u>-2.2</u>	<u>-1.75</u>	<u>-2.5</u>
Est. Meas. s.d.	<u>6.13×10^{-4}</u>	<u>7.05×10^{-4}</u>	<u>7.95×10^{-4}</u>	<u>3.09×10^{-4}</u>
Meas. s.d. in %	<u>0.490%</u>	<u>0.539%</u>	<u>0.612%</u>	<u>0.221%</u>
W Value	<u>0.781</u>	<u>0.934</u>	<u>0.955</u>	<u>0.870</u>
95% W Critical Value	<u>0.788</u>	<u>0.859</u>	<u>0.803</u>	<u>0.901</u>
Est. Bias s.d.	<u>0</u>	<u>3.99×10^{-4}</u>	<u>4.31×10^{-4}</u>	<u>1.6×10^{-4}</u>
Bias s.d. in %	<u>0</u>	<u>0.305%</u>	<u>0.332%</u>	<u>0.114%</u>
Est. Process s.d.	<u>5.77×10^{-3}</u>	<u>4.83×10^{-3}</u>	<u>4.03×10^{-3}</u>	<u>1.55×10^{-2}</u>
Process s.d. in %	<u>4.61</u>	<u>3.66%</u>	<u>3.1%</u>	<u>11.1%</u>
No. of Outliers	<u>1</u>	<u>0</u>	<u>0</u>	<u>1</u>
% of Outliers	<u>14.2%</u>	<u>0</u>	<u>0</u>	<u>5%</u>

Type of Material PuNO₃

Characteristic Measured Pu Assay

Facilities _____

Data Sets

Facilities	<u>G,B</u>	<u>H,B</u>	<u>J,E</u>	<u>K,B</u>
No. S/R Comparisons	<u>3</u>	<u>14</u>	<u>8</u>	<u>10</u>
Ave. Facility 1	<u>0.1476</u>	<u>0.13916</u>	<u>0.14334</u>	<u>0.1350</u>
Ave. Facility 2	<u>0.1476</u>	<u>0.13864</u>	<u>0.14292</u>	<u>0.1346</u>
Ave. (1) & (2)	<u>0.1476</u>	<u>0.13890</u>	<u>0.14313</u>	<u>0.1348</u>
Diff. (1) - (2)	<u>0</u>	<u>0.00052</u>	<u>0.00041</u>	<u>3.9×10^{-4}</u>
% Difference	<u>0</u>	<u>0.37%</u>	<u>0.29%</u>	<u>0.29%</u>
Student's t	<u>0</u>	<u>3.5</u>	<u>2.4</u>	<u>1.6</u>
Est. Meas. s.d.	<u>3.27×10^{-4}</u>	<u>4.00×10^{-4}</u>	<u>3.41×10^{-4}</u>	<u>5.43×10^{-4}</u>
Meas. s.d. in %	<u>0.221</u>	<u>0.288%</u>	<u>0.238%</u>	<u>0.162%</u>
W Value	<u>0.750</u>	<u>0.873</u>	<u>0.819</u>	<u>0.862</u>
95% W Critical Value	<u>0.767</u>	<u>0.874</u>	<u>0.818</u>	<u>0.842</u>
Est. Bias s.d.	<u>0</u>	<u>3.53×10^{-4}</u>	<u>2.65×10^{-4}</u>	<u>2.16×10^{-4}</u>
Bias s.d. in %	<u>0</u>	<u>0.254%</u>	<u>0.185%</u>	<u>0.160%</u>
Est. Process s.d.	<u>1.9×10^{-4}</u>	<u>2.19×10^{-2}</u>	<u>5.67×10^{-3}</u>	<u>1.25×10^{-2}</u>
Process s.d. in %	<u>0.13</u>	<u>15.7%</u>	<u>4.0%</u>	<u>9.3%</u>
No. of Outliers	<u>0</u>	<u>2</u>	<u>0</u>	<u>1</u>
% of Outliers	<u>0</u>	<u>12.5</u>	<u>0</u>	<u>9.1</u>

Type of Material PuNO₃

Characteristic Measured Pu Assay

Facilities A,B

Data Sets

Facilities	A,B			
No. S/R Comparisons	166			
Ave. Facility 1	0.14371			
Ave. Facility 2	0.14309			
Ave. (1) & (2)	0.14340			
Diff. (1) - (2)	0.00062			
% Difference	0.43%			
Student's t	7.3			
Est. Meas. s.d.	7.73×10^{-4}			
Meas. s.d. in %	0.54%			
W Value	--			
95% W Critical Value	--			
Est. Bias s.d.	4.32×10^{-4}			
Bias s.d. in %	0.30%			
Est. Process s.d.	0.016			
Process s.d. in %	11.1%			
No. of Outliers	4			
% of Outliers	2.3%			

Pu(NO₃)₄ - Net Weights

The following results are based on five sets of S/R differences from four facilities. The main conclusions pertinent to the variability aspects of the data are as follows.

1. The percent standard deviations for measurement precision are significantly different between the data sets. This does not seem to be due to one especially large or small percent standard deviation. The percent standard deviations, arranged according to magnitude, are as follows.

	<u>% s.d.</u>	<u>n</u>
1	0.148%	23
2	0.104%	32
3	0.095%	5
4	0.045%	16
5	0.036%	9

2. The pooled estimate of the percent standard deviation of precision is 0.106% so that the observed performance level for measurement precision is about 1/10%.
3. Smaller percent standard deviations of 0.045% and 0.036% have been observed based on 25 observations. Thus, a capability level of performance around 1/20% to 1/30% is demonstrated.
4. None of the three tests on which the W statistic was calculated indicates that the differences are not normally distributed.
5. The relative bias standard deviation estimate is 0.60% which is quite large compared to the measurement precisions.

Improper treatment of the weight of bags, tape and security seals have been observed as common causes of biases between facility weights. Leakage of solution from the shipping bottles poses an especially difficult problem for the shipper to reconcile. Evaporation and condensation may be bias contributors.

6. The pooled process variability as expressed by the percent standard deviation is 5.6%.
7. The results in more detail for the individual data sets are as follows.

Type of Material PuNO₃

Characteristic Measured Net Wt. PuNO₃/Bottle

Facilities A, B, C, D

Data Sets

Facilities	<u>A,B</u>	<u>C,B</u>	<u>A,B</u>	<u>D,B</u>
No. S/R Comparisons	<u>16</u>	<u>23</u>	<u>32</u>	<u>9</u>
Ave. Facility 1	<u>12672.5</u>	<u>12419.5</u>	<u>12703.2</u>	<u>11830.7</u>
Ave. Facility 2	<u>12650.1</u>	<u>12571.0</u>	<u>12666.0</u>	<u>12000.2</u>
Ave. (1) & (2)	<u>12616.0</u>	<u>12495.2</u>	<u>12684.6</u>	<u>11915.45</u>
Diff. (1) - (2)	<u>22.4</u>	<u>-151.5</u>	<u>37.2</u>	<u>-169.6</u>
% Difference	<u>0.177%</u>	<u>-1.21%</u>	<u>0.293%</u>	<u>-1.42%</u>
Student's t	<u>11.2</u>	<u>-27.9</u>	<u>11.3</u>	<u>-84</u>
Est. Meas. s.d.	<u>5.64</u>	<u>18.4</u>	<u>13.2</u>	<u>4.3</u>
Meas. s.d. in %	<u>0.0445%</u>	<u>0.148%</u>	<u>0.104%</u>	<u>0.036</u>
W Value	<u>0.961</u>	<u>--</u>	<u>--</u>	<u>0.952</u>
95% W Critical Value	<u>0.887</u>	<u>--</u>	<u>--</u>	<u>0.829</u>
Est. Bias s.d.	<u>15.8</u>	<u>107</u>	<u>26.2</u>	<u>119.9</u>
Bias s.d. in %	<u>0.125%</u>	<u>0.862</u>	<u>0.043</u>	<u>1.01</u>
Est. Process s.d.	<u>617</u>	<u>765</u>	<u>359</u>	<u>80.4</u>
Process s.d. in %	<u>4.87%</u>	<u>6.1%</u>	<u>2.83%</u>	<u>0.674%</u>
No. of Outliers	<u>0</u>	<u>1</u>	<u>0</u>	<u>0</u>
% of Outliers	<u>0</u>	<u>4.2%</u>	<u>0</u>	<u>0</u>

Type of Material PuNO₃

Characteristic Measured Net Wt. PuNO₃/Bottle

Facilities

Data Sets

Facilities	<u>D,B</u>	<u></u>	<u></u>	<u></u>
No. S/R Comparisons	<u>5</u>	<u></u>	<u></u>	<u></u>
Ave. Facility 1	<u>10791.6</u>	<u></u>	<u></u>	<u></u>
Ave. Facility 2	<u>10801.2</u>	<u></u>	<u></u>	<u></u>
Ave. (1) & (2)	<u>10796.4</u>	<u></u>	<u></u>	<u></u>
Diff. (1) - (2)	<u>-9.6</u>	<u></u>	<u></u>	<u></u>
% Difference	<u>-0.089%</u>	<u></u>	<u></u>	<u></u>
Student's t	<u>-1.5</u>	<u></u>	<u></u>	<u></u>
Est. Meas. s.d.	<u>10.2</u>	<u></u>	<u></u>	<u></u>
Meas. s.d. in %	<u>0.095</u>	<u></u>	<u></u>	<u></u>
W Value	<u>0.959</u>	<u></u>	<u></u>	<u></u>
95% W Critical Value	<u>0.762</u>	<u></u>	<u></u>	<u></u>
Est. Bias s.d.	<u>5.02</u>	<u></u>	<u></u>	<u></u>
Bias s.d. in %	<u>0.0465%</u>	<u></u>	<u></u>	<u></u>
Est. Process s.d.	<u>1778</u>	<u></u>	<u></u>	<u></u>
Process s.d. in %	<u>16.5%</u>	<u></u>	<u></u>	<u></u>
No. of Outliers	<u>0</u>	<u></u>	<u></u>	<u></u>
% of Outliers	<u>0</u>	<u></u>	<u></u>	<u></u>

$\text{Pu}(\text{NO}_3)_4$ - ^{238}Pu - Weight Percent

Five sets of data from five facilities are included in the analysis of the weight percent of ^{238}Pu in PuNO_3 material. The main results are as follows.

1. The Bartlett's test indicates that the variances due to measurement precision are significantly different. The percent standard deviations, as arranged in magnitude from the largest to the smallest, are as follows.

	<u>% s.d.</u>	<u>n</u>
1	17.4%	14
2	11.0%	162
3	6.8%	4
4	5.8%	9
5	1.0%	16

2. The pooled estimate of the percent standard deviation due to measurement precision is 10.9%. Needless to say, this isotopic weight percent is not measured very precisely at this low abundance of 0.03-0.08 weight percent ^{238}Pu .
3. One of the four sets for which the W statistics were calculated indicates a lack of normality. The other three sets indicated good agreement with normality assumptions.
4. The percent standard deviation due to bias is estimated as 2.57%.
5. The percent standard deviation due to process variation is estimated at 25.2%.
6. Additional results for the individual data sets are as follows.

Type of Material PuNO₃
 Characteristic Measured ²³⁸Pu Wt. %
 Facilities A, B, C, D, E

	<u>Data Sets</u>			
Facilities	<u>A,B</u>	<u>C,B</u>	<u>B,D</u>	<u>E,B</u>
No. S/R Comparisons	<u>14</u>	<u>4</u>	<u>9</u>	<u>16</u>
Ave. Facility 1	<u>0.02936</u>	<u>0.11475</u>	<u>0.06544</u>	<u>0.79456</u>
Ave. Facility 2	<u>0.03079</u>	<u>0.10875</u>	<u>0.07077</u>	<u>0.79475</u>
Ave. (1) & (2)	<u>0.03007</u>	<u>0.11175</u>	<u>0.06810</u>	<u>0.79465</u>
Diff. (1) - (2)	<u>-0.00143</u>	<u>0.0060</u>	<u>-0.0053</u>	<u>-0.00019</u>
% Difference	<u>-4.75%</u>	<u>5.4%</u>	<u>-7.8%</u>	<u>-0.024%</u>
Student's t	<u>-0.74</u>	<u>1.1</u>	<u>-3.0</u>	<u>-0.1</u>
Est. Meas. s.d.	<u>0.0051</u>	<u>0.0078</u>	<u>0.0038</u>	<u>0.0080</u>
Meas. s.d. in %	<u>17.4%</u>	<u>6.8%</u>	<u>5.8%</u>	<u>1.01%</u>
W Value	<u>0.718</u>	<u>0.958</u>	<u>0.925</u>	<u>0.963</u>
95% W Critical Value	<u>0.874</u>	<u>0.748</u>	<u>0.829</u>	<u>0.887</u>
Est. Bias s.d.	<u>0</u>	<u>0.0017</u>	<u>0.0036</u>	<u>0%</u>
Bias s.d. in %	<u>0</u>	<u>1.45%</u>	<u>5.4%</u>	<u>0%</u>
Est. Process s.d.	<u>0.0048</u>	<u>0.0122</u>	<u>0.017</u>	<u>0.296</u>
Process s.d. in %	<u>15.9%</u>	<u>10.9%</u>	<u>2.5%</u>	<u>37.2%</u>
No. of Outliers	<u>1</u>	<u>1</u>	<u>1</u>	<u>2</u>
% of Outliers	<u>6.7%</u>	<u>20%</u>	<u>10.0%</u>	<u>11.1%</u>

Type of Material PuNO₃
 Characteristic Measured ²³⁸Pu Wt. %
 Facilities A,B

Data Sets

Facilities	<u>A,B</u>			
No. S/R Comparisons	<u>162</u>			
Ave. Facility 1	<u>0.0548</u>			
Ave. Facility 2	<u>0.0535</u>			
Ave. (1) & (2)	<u>0.05415</u>			
Diff. (1) - (2)	<u>0.00123</u>			
% Difference	<u>2.27%</u>			
Student's t	<u>1.83</u>			
Est. Meas. s.d.	<u>0.0060</u>			
Meas. s.d. in %	<u>11.00</u>			
W Value	<u>--</u>			
95% W Critical Value	<u>--</u>			
Est. Bias s.d.	<u>.0007</u>			
Bias s.d. in %	<u>1.33</u>			
Est. Process s.d.	<u>.0137</u>			
Process s.d. in %	<u>25.3</u>			
No. of Outliers	<u>5</u>			
% of Outliers	<u>2.9</u>			

Pu(NO₃)₄ - ²³⁹Pu - Weight Percent

Five sets of data from five facilities are included in the analysis. The main results as regard to the precision, bias, and process sources of variation are as follows.

1. There is some indication that the measurement precisions are different for the different sets as gauged by the Bartlett's test of the homogeneity of variances. The percent standard deviations of measurement precision are as follows.

	<u>% s.d.</u>	<u>n</u>
1	0.131%	5
2	0.113%	20
3	0.084%	14
4	0.076%	9
5	0.071%	142

2. The pooled percent standard deviation of measurement precision is 0.078%. Since the 0.071% value is based on 142 values, it appears that a measurement precision of 0.075% has been and is a realizable target.
3. In three of the four sets of data, there are no indicated departures from normality as tested by the W statistic.
4. The percent standard deviation of bias is estimated as 0.040% which is about 1/2 of the precision percent standard deviation.
5. The process variation is indicated by the estimated percent standard deviation of 1.51%.
6. Other and more detailed results on the individual data sets are as follows.

Type of Material PuNO₃
 Characteristic Measured ²³⁹Pu Wt. %
 Facilities A, B, C, D, E

Facilities	Data Sets			
	A,B	C,B	B,D	B,E
No. S/R Comparisons	14	5	9	20
Ave. Facility 1	89.828	82.634	86.092	71.282
Ave. Facility 2	89.833	82.749	86.041	71.260
Ave. (1) & (2)	89.830	82.691	86.066	71.271
Diff. (1) - (2)	-0.0049	-0.115	0.051	0.023
% Difference	-0.0055%	-0.14%	0.059%	0.03%
Student's t	-0.2	-1.67	1.67	0.88
Est. Meas. s.d.	0.076	0.108	0.065	8.08 x 10 ⁻²
Meas. s.d. in %	0.084%	0.131%	0.076	0.113%
W Value	0.946	0.714	0.927	0.937
95% W Critical Value	0.874	0.762	0.829	0.904
Est. Bias s.d.	0	0.0648	0.0290	0
Bias s.d. in %	0%	0.0754%	0.0336%	0%
Est. Process s.d.	0.683	3.65	0	3.0
Process s.d. in %	0.760	4.42	0	4.20%
No. of Outliers	1	0	1	1
% of Outliers	6.7	0%	10.0	4.8%

Type of Material PuNO₃
 Characteristic Measured ²³⁹Pu Wt. %
 Facilities A,B

Data Sets

Facilities	A,B			
No. S/R Comparisons	142			
Ave. Facility 1	86.566			
Ave. Facility 2	86.532			
Ave. (1) & (2)	86.549			
Diff. (1) - (2)	0.034			
% Difference	0.040%			
Student's t	4.7			
Est. Meas. s.d.	0.062			
Meas. s.d. in %	0.071%			
W Value	--			
95% W Critical Value	--			
Est. Bias s.d.	0.0238			
Bias s.d. in %	0.0274			
Est. Process s.d.	0.069			
Process s.d. in %	0.080			
No. of Outliers	1			
% of Outliers	0.7			

Pu(NO₃)₄ - ²⁴⁰Pu - Weight Percent

Five facilities and five sets of S/R values are involved in the analysis here. The main results pertaining to measurement precision and bias and process variability are as follows.

1. There is a significant difference between the percent standard deviations from the different data sets. This is probably due to one particularly low percent standard deviation. The percent standard deviations arranged in order of magnitude are as follows.

	<u>% s.d.</u>	<u>n</u>
1	0.41%	9
2	0.40%	4
3	0.36%	139
4	0.24%	20
5	0.05%	14

2. The pooled percent s.d. of measurement precision is 0.34%. This would appear to be a good bench mark for a percent standard deviation of measurement performance at these abundance levels as it is based on a considerable number of observations.
3. None of the W statistics are significant at the 95% level which means that there is no reason to assume that S/R differences for the weight percent ²⁴⁰Pu are not normally distributed.
4. The percent standard deviation of bias is estimated as 0.028%.
5. The percent standard of process variance is estimated as 1.14%.
6. Further and more detailed results on individual data sets are as follows.

Type of Material PuNO₃
 Characteristic Measured ²⁴⁰Pu Wt. %
 Facilities A, B, C, D, E, F

	<u>Data Sets</u>			
Facilities	<u>A,B</u>	<u>C,B</u>	<u>B,D</u>	<u>E,F</u>
No. S/R Comparisons	<u>14</u>	<u>4</u>	<u>9</u>	<u>20</u>
Ave. Facility 1	<u>9.0178</u>	<u>14.776</u>	<u>11.638</u>	<u>17.357</u>
Ave. Facility 2	<u>9.0558</u>	<u>14.702</u>	<u>11.671</u>	<u>17.393</u>
Ave. (1) & (2)	<u>9.0368</u>	<u>14.739</u>	<u>11.6545</u>	<u>17.375</u>
Diff. (1) - (2)	<u>-0.0379</u>	<u>0.074</u>	<u>-0.033</u>	<u>-0.036</u>
% Difference	<u>-0.42%</u>	<u>0.50%</u>	<u>-0.28%</u>	<u>-0.21%</u>
Student's t	<u>-2.26</u>	<u>1.78</u>	<u>-1.5</u>	<u>-2.7</u>
Est. Meas. s.d.	<u>0.0442</u>	<u>0.0589</u>	<u>0.0473</u>	<u>0.0421</u>
Meas. s.d. in %	<u>0.49%</u>	<u>0.399%</u>	<u>0.406%</u>	<u>0.242%</u>
W Value	<u>0.945</u>	<u>0.938</u>	<u>0.875</u>	<u>0.913</u>
95% W Critical Value	<u>0.874</u>	<u>0.748</u>	<u>0.829</u>	<u>0.904</u>
Est. Bias s.d.	<u>0.0241</u>	<u>0.0432</u>	<u>0.0176</u>	<u>0.0240</u>
Bias s.d. in %	<u>0.266%</u>	<u>0.293%</u>	<u>0.152%</u>	<u>0.0138</u>
Est. Process s.d.	<u>0.602</u>	<u>1.92</u>	<u>0.021</u>	<u>3.41</u>
Process s.d. in %	<u>6.67%</u>	<u>13.0%</u>	<u>0.2%</u>	<u>19.6%</u>
No. of Outliers	<u>1</u>	<u>1</u>	<u>1</u>	<u>1</u>
% of Outliers	<u>6.7%</u>	<u>20.0%</u>	<u>10.0%</u>	<u>4.8%</u>

Type of Material PuNO₃

Characteristic Measured ²⁴⁰Pu Wt. %

Facilities

Data Sets

Facilities	A,B			
No. S/R Comparisons	139			
Ave. Facility 1	11.533			
Ave. Facility 2	11.565			
Ave. (1) & (2)	11.5492			
Diff. (1) - (2)	-0.0321			
% Difference	-0.28%			
Student's t	-6.5			
Est. Meas. s.d.	0.041			
Meas. s.d. in %	0.359			
W Value	--			
95% W Critical Value	--			
Est. Bias s.d.	0.022			
Bias s.d. in %	0.195			
Est. Process s.d.	0.0197			
Process s.d. in %	0.0291			
No. of Outliers	5			
% of Outliers	3.4%			

Pu(NO₃)₄ - ²⁴¹Pu - Weight Percent

Five data sets from five facilities were involved in this analysis.

The main results pertaining to variability are as follows.

1. The test for a difference between measurement precisions for the different data sets is of borderline significance. The percent standard deviations of the measurement precision arranged in magnitude are as follows.

	<u>% s.d.</u>	<u>n</u>
1	1.214%	9
2	0.769%	131
3	0.763%	14
4	0.693%	5
5	0.496%	20

2. The pooled percent standard deviation of measurement precision is 0.77%. A suitable percent standard deviation for performance as far as measurement precision is concerned is (1/2--3/4)% at this abundance level.
3. The data do not reveal too much here in regard to the ultimate measurement capability for precision.
4. One of the four data sets tested for normality showed a variance with expected results under the hypothesis of normality, and another is of borderline significance.
5. The standard deviation due to bias is estimated at 0.43% which is similar in magnitude to the percent standard deviation of measurement precision.
6. The percent standard deviation of process variation is estimated at 6.27%

7. Additional and more detailed results for the individual data sets are as follows.

Type of Material PuNO₃
 Characteristic Measured ²⁴¹Pu Wt. %
 Facilities A, B, C, D, E, F

Data Sets

Facilities	<u>A,B</u>	<u>C,B</u>	<u>D,E</u>	<u>B,F</u>
No. S/R Comparisons	<u>14</u>	<u>5</u>	<u>20</u>	<u>9</u>
Ave. Facility 1	<u>0.9838</u>	<u>3.1566</u>	<u>8.782</u>	<u>2.035</u>
Ave. Facility 2	<u>0.9747</u>	<u>3.1308</u>	<u>8.770</u>	<u>2.014</u>
Ave. (1) & (2)	<u>0.9792</u>	<u>3.1437</u>	<u>8.776</u>	<u>2.0245</u>
Diff. (1) - (2)	<u>0.0091</u>	<u>0.0258</u>	<u>0.012</u>	<u>0.021</u>
% Difference	<u>0.93%</u>	<u>0.82%</u>	<u>0.137%</u>	<u>1.04%</u>
Student's t	<u>3.22</u>	<u>1.55</u>	<u>0.88</u>	<u>1.78</u>
Est. Meas. s.d.	<u>0.0075</u>	<u>0.0262</u>	<u>0.0436</u>	<u>.0247</u>
Meas. s.d. in %	<u>0.763</u>	<u>0.693</u>	<u>0.496</u>	<u>1.214</u>
W Value	<u>0.892</u>	<u>0.747</u>	<u>0.943</u>	<u>0.938</u>
95% W Critical Value	<u>0.874</u>	<u>0.762</u>	<u>0.905</u>	<u>0.829</u>
Est. Bias s.d.	<u>0.0062</u>	<u>.0139</u>	<u>0</u>	<u>.0122</u>
Bias s.d. in %	<u>0.625%</u>	<u>0.442%</u>	<u>0%</u>	<u>0.598%</u>
Est. Process s.d.	<u>0.0972</u>	<u>0.625</u>	<u>1.22</u>	<u>.0270</u>
Process s.d. in %	<u>9.9%</u>	<u>19.9%</u>	<u>13.9%</u>	<u>1.8%</u>
No. of Outliers	<u>1</u>	<u>0</u>	<u>1</u>	<u>1</u>
% of Outliers	<u>6.7</u>	<u>0</u>	<u>4.8</u>	<u>10.0</u>

Type of Material PuNO₃

Characteristic Measured ²⁴¹Pu Wt. %

Facilities _____

Data Sets

Facilities	A,B			
No. S/R Comparisons	131			
Ave. Facility 1	1.6628			
Ave. Facility 2	1.6613			
Ave. (1) & (2)	1.6620			
Diff. (1) - (2)	0.0015			
% Difference	0.090%			
Student's t	0.9			
Est. Meas. s.d.	.01278			
Meas. s.d. in %	0.769			
W Value	--			
95% W Critical Value	--			
Est. Bias s.d.	0%			
Bias s.d. in %	0%			
Est. Process s.d.	.0258			
Process s.d. in %	1.55			
No. of Outliers	7			
% of Outliers	4.8%			

Pu(NO₃)₄ - ²⁴²Pu - Weight Percent

Six facilities were involved in the five data sets of S/R comparisons. The main conclusions pertinent to variability are as follows.

1. The differences between the percent standard deviations of measurement precision is of high statistical significance. The percent standard deviations of measurement precision arranged in order of magnitude are as follows.

	<u>% s.d.</u>	<u>n</u>
1	3.6%	14
2	2.7%	8
3	2.2%	102
4	0.95%	4
5	0.46%	19

2. The pooled percent standard deviation of measurement precision is 2.27%. The values 1/2% and 1.0% represented by the last two data sets, however, would seem to represent attainable target values. This range of precision appears reasonable in view of the low isotopic abundances of the samples, e.g., 0.04-1.7 weight percent ²⁴²Pu.
3. None of the four W tests for normality are statistically significant so that the S/R differences here, and indeed the random measurement components of the individual measurements can be assumed to be normally distributed.
4. The percent standard deviation of bias is estimated at 0.42%.
5. The percent standard deviation of process variation is estimated at 10.7%.

6. Additional and detailed results pertaining to the individual data sets are as follows.

Type of Material PuNO₃
 Characteristic Measured ²⁴²Pu Wt. %
 Facilities A, B, C, D, E, F

	<u>Data Sets</u>			
Facilities	<u>A,B</u>	<u>C,B</u>	<u>B,D</u>	<u>E,F</u>
No. S/R Comparisons	<u>14</u>	<u>4</u>	<u>8</u>	<u>19</u>
Ave. Facility 1	<u>0.1249</u>	<u>.040525</u>	<u>0.1749</u>	<u>1.7603</u>
Ave. Facility 2	<u>0.1245</u>	<u>.040625</u>	<u>0.1779</u>	<u>1.7657</u>
Ave. (1) & (2)	<u>0.1247</u>	<u>0.040575</u>	<u>0.1764</u>	<u>1.7630</u>
Diff. (1) - (2)	<u>3.6×10^{-4}</u>	<u>-0.0001</u>	<u>-0.0030</u>	<u>-0.0054</u>
% Difference	<u>0.28%</u>	<u>-0.25%</u>	<u>-1.7%</u>	<u>-0.31%</u>
Student's t	<u>0.2</u>	<u>-0.4</u>	<u>-1.27</u>	<u>-2.05</u>
Est. Meas. s.d.	<u>.0045</u>	<u>.0039</u>	<u>.0047</u>	<u>.0081</u>
Meas. s.d. in %	<u>3.6</u>	<u>0.95</u>	<u>2.7</u>	<u>0.46</u>
W Value	<u>0.919</u>	<u>0.989</u>	<u>0.990</u>	<u>0.973</u>
95% W Critical Value	<u>0.874</u>	<u>0.748</u>	<u>0.818</u>	<u>0.901</u>
Est. Bias s.d.	<u>0</u>	<u>0</u>	<u>.0013</u>	<u>.0034</u>
Bias s.d. in %	<u>0%</u>	<u>0%</u>	<u>0.75%</u>	<u>0.19</u>
Est. Process s.d.	<u>.0292</u>	<u>0.113</u>	<u>.0039</u>	<u>0.339</u>
Process s.d. in %	<u>23.5</u>	<u>27.8</u>	<u>2.2%</u>	<u>19.2</u>
No. of Outliers	<u>1</u>	<u>1</u>	<u>2</u>	<u>2</u>
% of Outliers	<u>6.7</u>	<u>20.0</u>	<u>20.0</u>	<u>9.5</u>

Type of Material PuNO₃

Characteristic Measured ²⁴²Pu Wt. %

Facilities

Data Sets

Facilities	A,B			
No. S/R Comparisons	102			
Ave. Facility 1	0.18902			
Ave. Facility 2	0.19063			
Ave. (1) & (2)	0.18982			
Diff. (1) - (2)	-0.00161			
% Difference	-0.85%			
Student's t	-2.7			
Est. Meas. s.d.	0.0042			
Meas. s.d. in %	2.24			
W Value	--			
95% W Critical Value	--			
Est. Bias s.d.	.00106			
Bias s.d. in %	0.559			
Est. Process s.d.	.00119			
Process s.d. in %	0.628			
No. of Outliers	4			
% of Outliers	3.6			

PuO₂ - Pu Content - Weight Percent

The following results are based on six sets of S/R data on the Pu content of PuO₂ from five facilities. The main results as regards variability are as follows.

1. There is no significant difference in the measurement precision standard deviations when tested at the 95% level. The percent standard deviations of precision, arranged according to magnitude, are as follows.

	<u>% s.d.</u>	<u>n</u>
1	0.197%	7
2	0.116%	5
3	0.103%	7
4	0.084%	7
5	0.084%	7
6	0.057%	4

2. The pooled percent standard deviation of precision is 0.120% which serves as a bench mark of comparison for both performance and capability values since there are no significant differences between standard deviations.
3. There is no reason to suspect that the data are not normally distributed. None of the W statistics computed for the six data sets lie beyond the 95% point for the statistic.
4. The % standard deviation of bias is estimated at 0.63%. This may be due to real differences in what was measured at the time of the measurement rather than to biases of this magnitude in the measurements themselves. That is to say, the material concentration may have changed due particularly to moisture

pickup either in the PuO_2 shipped or in the sample chosen for analysis. Dissolution problems have also been observed to be causes of bias in sample analysis.

5. The process variance is 0.28% in percent standard deviation terms.
6. Additional results for the individual data sets are as follows.

Type of Material PuO₂

Characteristic Measured Pu Content Wt. %

Facilities A, B, C, D, E

Data Sets

Facilities	<u>B,C</u>	<u>B,C</u>	<u>D,B</u>
No. S/R Comparisons	<u>5</u>	<u>4</u>	<u>7</u>
Ave. Facility 1	<u>87.802</u>	<u>87.65</u>	<u>87.836</u>
Ave. Facility 2	<u>87.946</u>	<u>88.20</u>	<u>87.879</u>
Ave. (1) & (2)	<u>87.874</u>	<u>87.925</u>	<u>87.858</u>
Diff. (1) - (2)	<u>-0.132</u>	<u>-0.55</u>	<u>-0.043</u>
% Difference	<u>-0.16%</u>	<u>-0.63</u>	<u>-0.049</u>
Student's t	<u>-2.24</u>	<u>-15.7</u>	<u>-1.1</u>
Est. Meas. s.d.	<u>0.102</u>	<u>0.0496</u>	<u>0.074</u>
Meas. s.d. in %	<u>0.116</u>	<u>0.0566</u>	<u>0.084</u>
W Value	<u>0.832</u>	<u>0.862</u>	<u>0.933</u>
95% W Critical Value	<u>0.762</u>	<u>0.748</u>	<u>0.803</u>
Est. Bias s.d.	<u>0.0910</u>	<u>0.388</u>	<u>0.0117</u>
Bias s.d. in %	<u>0.1037</u>	<u>0.443</u>	<u>0.0134</u>
Est. Process s.d.	<u>0.0712</u>	<u>0.104</u>	<u>0.062</u>
Process s.d. in %	<u>0.0810</u>	<u>0.1187</u>	<u>0.071</u>
No. of Outliers	<u>1</u>	<u>0</u>	<u>1</u>
% of Outliers	<u>16.7</u>	<u>0</u>	<u>12.5</u>

Type of Material PuO₂
 Characteristic Measured Pu Content Wt %
 Facilities A, B, C, D, E

	<u>Data Sets</u>			
Facilities	<u>B,A</u>	<u>D,B</u>	<u>B,E</u>	
No. S/R Comparisons	<u>7</u>	<u>7</u>	<u>7</u>	
Ave. Facility 1	<u>87.882</u>	<u>87.836</u>	<u>87.88</u>	
Ave. Facility 2	<u>86.276</u>	<u>87.879</u>	<u>86.98</u>	
Ave. (1) & (2)	<u>87.079</u>	<u>87.857</u>	<u>87.43</u>	
Diff. (1) - (2)	<u>1.61</u>	<u>-0.043</u>	<u>0.90</u>	
% Difference	<u>1.85%</u>	<u>-0.049%</u>	<u>1.00%</u>	
Student's t	<u>17.3</u>	<u>-1.08</u>	<u>18.6</u>	
Est. Meas. s.d.	<u>0.173</u>	<u>0.074</u>	<u>0.091</u>	
Meas. s.d. in %	<u>0.197</u>	<u>0.084</u>	<u>0.103</u>	
W Value	<u>0.936</u>	<u>0.933</u>	<u>0.951</u>	
95% W Critical Value	<u>0.803</u>	<u>0.803</u>	<u>0.803</u>	
Est. Bias s.d.	<u>1.133</u>	<u>.01175</u>	<u>0.64</u>	
Bias s.d. in %	<u>1.29</u>	<u>.01338</u>	<u>0.73</u>	
Est. Process s.d.	<u>0</u>	<u>0.621</u>	<u>0.027</u>	
Process s.d. in %	<u>0</u>	<u>0.707</u>	<u>0.031</u>	
No. of Outliers	<u>0</u>	<u>1</u>	<u>0</u>	
% of Outliers	<u>0</u>	<u>12.5</u>	<u>0%</u>	

PuO₂ - Tare Weights

The following comparisons of S/R tare weights for PuO₂ containers originated from three facilities and two sets of data. The main conclusions pertaining to variability are as follows.

1. The difference in the percent standard deviation of precisions of the two sets is statistically significant, the difference in the absolute standard deviations is not statistically significant, however. The values here are

	<u>% s.d.</u>	<u>abs. s.d.</u>	<u>n</u>
1	0.410%	0.215%	15
2	0.078%	0.275%	23

2. It is hard to generalize about performance and capability aspects of measurement precision due to the limited number of data sets and data points here. Thus, these results in themselves are about as far as it is possible to go at this stage.
3. The W tests indicate that there is no reason to assume that the net weights are not normally distributed.
4. The bias percent standard deviation is estimated as 0.056%.
5. The process variability percent standard deviation is estimated as 45.0%.
6. Additional results for individual data sets are as follows.

Type of Material PuO₂

Characteristic Measured Tare Weights

Facilities A, B, C

	<u>Data Sets</u>			
Facilities	<u>A,B</u>	<u>C,B</u>		
No. S/R Comparisons	<u>15</u>	<u>23</u>		
Ave. Facility 1	<u>52.611</u>	<u>352.61</u>		
Ave. Facility 2	<u>52.620</u>	<u>353.02</u>		
Ave. (1) & (2)	<u>52.6155</u>	<u>352.815</u>		
Diff. (1) - (2)	<u>-0.009</u>	<u>-0.41</u>		
% Difference	<u>-0.017%</u>	<u>-0.12%</u>		
Student's t	<u>-0.1</u>	<u>-5.0</u>		
Est. Meas. s.d.	<u>0.215</u>	<u>0.275</u>		
Meas. s.d. in %	<u>0.410</u>	<u>0.078</u>		
W Value	<u>0.969</u>	<u>0.954</u>		
95% W Critical Value	<u>0.881</u>	<u>0.914</u>		
Est. Bias s.d.	<u>0</u>	<u>0.283</u>		
Bias s.d. in %	<u>0</u>	<u>0.080</u>		
Est. Process s.d.	<u>37.9</u>	<u>4.11</u>		
Process s.d. in %	<u>72.2</u>	<u>1.16</u>		
No. of Outliers	<u>1</u>	<u>2</u>		
% of Outliers	<u>6.7</u>	<u>8.0</u>		

PuO₂ Powder - Net Weights - Grams

These results are based on seven data sets from five different facilities. The main results in the variability aspects of the S/R values are as follows.

1. The differences between the percent standard deviations of the different data sets are statistically significant. The percent and absolute standard deviations are given as follows where the percent standard deviations are arranged according to magnitude.

	<u>% s.d.</u>	<u>s.d.</u>	<u>n</u>
1	0.537%	2.400	14
2	0.382%	0.799	9
3	0.144%	1.360	14
4	0.079%	0.810	13
5	0.063%	0.650	13
6	0.024%	0.242	24
7	0.021%	0.198	52

2. The graph on page 53 shows that the percent standard deviation is a rather poor way here to characterize the total effects of these measurement precision standard deviations. There does not in fact seem to be a good way to do this although in this instance the absolute standard deviation seems to be somewhat better than the relative standard deviation.

3. One test out of six indicates that the S/R differences are not normally distributed. For the other five W tests, the results are not significant. This seems to indicate that generally, one will have no difficulty under the assumption of normality but occasionally a non-normally distributed set of PuO_2 powder S/R differences will occur.
4. The percent standard deviation of bias is estimated as 0.52%.
5. The pooled process variances are characterized by the estimated percent deviation of 23.7%.
6. More detailed results on the individual data sets are as follows.

Type of Material PuO₂ Powder
 Characteristic Measured Weight (g) Net
 Facilities A, B, C, D, E, F, G

	<u>Data Sets</u>			
Facilities	A,B	C,B		
No. S/R Comparisons	14	9		
Ave. Facility 1	943.1	209.27		
Ave. Facility 2	955.7	210.15		
Ave. (1) & (2)	949.4	209.71		
Diff. (1) - (2)	-12.6	-0.88		
% Difference	-1.33%	-0.42%		
Student's t	-24.4	-2.4		
Est. Meas. s.d.	1.360	0.799		
Meas. s.d. in %	0.144	0.3815		
W Value	0.957	0.804		
95% W Critical Value	0.874	0.829		
Est. Bias s.d.	8.88	0.556		
Bias s.d. in %	0.942%	0.267%		
Est. Process s.d.	133.2	89.1		
Process s.d. in %	14.0%	42.5		
No. of Outliers	0	0		
% of Outliers	0	0		

Type of Material PuO₂ Pu Oxide PowderCharacteristic Measured Net WeightFacilities A, B, C, D, E, F, GData Sets

Facilities	D,B	B,E	A,B	E,B
No. S/R Comparisons	24	52	13	14
Ave. Facility 1	1022.6	945.93	1029.4	447.16
Ave. Facility 2	1023.5	945.57	1028.2	450.86
Ave. (1) & (2)	1023.05	945.75	1028.8	449.01
Diff. (1) - (2)	-0.87	0.36	1.2	-3.70
% Difference	-0.085%	0.038%	0.117%	-0.82%
Student's t	-1.2	9.3	4.5	-4.1
Est. Meas. s.d.	0.242	0.198	0.650	2.40
Meas. s.d. in %	0.0236%	0.0209%	0.0632	0.537
W Value	0.934	--	0.952	0.951
95% W Critical Value	0.916	--	0.866	0.874
Est. Bias s.d.	0.614	0.254	0.801	2.539
Bias s.d. in %	0.0600	0.0269	0.0778	0.568
Est. Process s.d.	135.3	214.0	106.1	212.0
Process s.d. in %	13.2%	22.6%	10.3%	47.3%
No. of Outliers	1	1	0	0
% of Outliers	4%	1.9%	0%	0%

Type of Material PuO₂ Powder

Characteristic Measured Net Weight

Facilities A, B, C, D, E, F, G

Data Sets

Facilities	F,B			
No. S/R Comparisons	13			
Ave. Facility 1	1029.3			
Ave. Facility 2	1028.4			
Ave. (1) & (2)	1028.85			
Diff. (1) - (2)	0.9			
% Difference	0.087%			
Student's t	2.87			
Est. Meas. s.d.	0.81			
Meas. s.d. in %	0.0789			
W Value	0.951			
95% W Critical Value	0.866			
Est. Bias s.d.	0.607			
Bias s.d. in %	0.0589			
Est. Process s.d.	106.1			
Process s.d. in %	10.3%			
No. of Outliers	0%			
% of Outliers	0%			

PuO₂ Total Pu - grams

Four sets of data were analyzed involving four different facilities.

The results are as follows.

1. The percent standard deviations of measurement precision are significantly different from one data set to another. This is apparently due to one rather high percent standard deviation. From largest to smallest, the results are as follows.

	<u>% s.d.</u>	<u>n</u>
1	0.276%	6
2	0.106%	14
3	0.099%	12
4	0.037%	4

2. The pooled percent standard deviations of precision before and after eliminating the largest value are 0.141% and 0.098%. Thus, 0.1% represents a reasonable performance percent standard deviation value.
3. There are only four values in the data set with the lowest percent standard deviation of 0.037%. It is difficult on the basis of just these data to assert what a capability level here is; it does seem that a capability level of about 1/10% is attainable.
4. In two of the data sets the differences appear to give results which contradict the hypothesis of normality. Thus, statistical measures which are heavily distribution-dependent should be used with some caution as regards this type of measurement.
5. The percent standard deviation of bias is estimated as 0.29% which is very high relative to the measurement precision.

6. The pooled process variance in relative terms is 14.1%.
7. More details on the individual data sets are as follows.

Type of Material PuO₂
 Characteristic Measured Weight Pu
 Facilities A, B, C, D

Facilities	<u>Data Sets</u>			
	A,B	A,B	C,A	D,A
No. S/R Comparisons	6	4	12	14
Ave. Facility 1	7311.3	9142.40	906.17	834.28
Ave. Facility 2	7268.7	9130.45	905.0	839.14
Ave. (1) & (2)	7290.0	9136.42	905.58	836.71
Diff. (1) - (2)	42.6	11.95	1.17	-4.86
% Difference	0.58%	0.13%	0.13%	-0.58%
Student's t	3.65	5.33	3.2	-14.5
Est. Meas. s.d.	20.2	3.17	0.896	0.885
Meas. s.d. in %	0.276%	0.0347	0.0988	0.106%
W Value	0.919	0.837	0.857	0.866
95% W Critical Value	0.788	0.748	0.859	0.874
Est. Bias s.d.	29.0	8.30	0.783	3.43
Bias s.d. in %	0.396	0.091	0.086	0.41%
Est. Process s.d.	1448	1270	97.2	117.5
Process s.d. in %	19.9	13.9	10.7	14.0%
No. of Outliers	0	2	1	0
% of Outliers	0	33%	7.7%	0%

Gross Weights

The following is taken from ten sets of S/R differences involving nine different facilities. The gross weights of the various cans and containers vary from 65g to 16,000g. If one plots $\ln \sigma_w$ versus $\ln$ weight, the following relationship is obtained.

For these data sets, excluding set 4, the slope is near one, (it is actually estimated as 0.996 ± 0.26) so that there is no reason to believe that the assumption of a constant percent standard deviation is inappropriate. In other cases, as for example the PuO_2 powder net weights, this assumption is not as good. The conclusions are as follows.

1. The % s.d. arranged from largest to smallest are as follows.

	<u>% s.d.</u>	<u>n</u>
1	1.050%	15
2	0.068%	5
3	0.057%	20
4	0.035%	24
5	0.027%	25
6	0.027%	147
7	0.024%	7
8	0.015%	16
9	0.014%	14
10	0.006%	51

The standard deviations in absolute terms for measurement precisions arranged from largest to smallest are as follows.

	<u>Absolute s.d.</u>	<u>% s.d.</u>	<u>Average</u>	<u>n</u>
1	36.9	0.024%	156401.	7
2	9.23	0.057%	16234.5	20
3	4.04	0.027%	15054.7	147
4	3.56	1.500%	339.0	15
5	2.34	0.015%	15598.0	16
6	1.35	0.068%	1995.2	5
7	0.49	0.035%	1376.0	24
8	0.35	0.014%	2467.8	14
9	0.068	0.006%	1176.2	51
10	0.018	0.027%	65.5	25

It is apparent that with the exception of set 4 that even with some shortcomings the percent standard deviation is a more sensible way of summarizing, displaying and combining these data than is the absolute standard deviation. When the average values are all of the same magnitude the relative and absolute standard deviations will give comparable results.

2. The pooled percent standard deviation of measurement precision neglecting the largest standard deviation is 0.028%. Since weighing equipment varies considerably over this range of average values, it is hard to be dogmatic about performance levels. The general range from 0.01% to 0.07%, however, may be useful when assessing the quality of a particular weighing process.
3. Since three out of eight of the W statistics indicate a tendency toward non-normality this suggests that caution should be exercised here in using tests or percentile values associated with the normal distribution.

4. The bias percent standard deviation estimate is 0.165% which is very high compared to the precision standard deviation. However, one may be seeing true differences here rather than biases in the weight measurements due to actual change in the weights of the materials.
5. The pooled process variability, as expressed in percent standard deviation is 25.1%.
6. Added and more detailed results on the individual data sets are as follows.

Type of Material Various Cans and Containers

Characteristic Measured Gross Weights

Facilities A, B, C, D, E, F, G, H, I

Data Sets

Facilities	<u>A,B</u>	<u>B,C</u>	<u>D,B</u>	<u>E,B</u>
No. S/R Comparisons	<u>15</u>	<u>5</u>	<u>14</u>	<u>51</u>
Ave. Facility 1	<u>337.68</u>	<u>1995.5</u>	<u>2467.5</u>	<u>1176.2</u>
Ave. Facility 2	<u>340.31</u>	<u>1994.9</u>	<u>2468.1</u>	<u>1176.2</u>
Ave. (1) & (2)	<u>338.995</u>	<u>1995.2</u>	<u>2467.8</u>	<u>1176.2</u>
Diff. (1) - (2)	<u>-2.63</u>	<u>0.62</u>	<u>-0.6</u>	<u>0</u>
% Difference	<u>-0.78%</u>	<u>0.03</u>	<u>-0.02</u>	<u>0%</u>
Student's t	<u>-2.0</u>	<u>0.72</u>	<u>-4.8</u>	<u>0.4</u>
Est. Meas. s.d.	<u>3.56</u>	<u>1.351</u>	<u>0.3516</u>	<u>0.0684</u>
Meas. s.d. in %	<u>1.05</u>	<u>0.0677</u>	<u>0.0142</u>	<u>0.0058</u>
W Value	<u>0.790</u>	<u>0.945</u>	<u>0.62</u>	<u>--</u>
95% W Critical Value	<u>0.881</u>	<u>0.762</u>	<u>0.87</u>	<u>--</u>
Est. Bias s.d.	<u>1.61</u>	<u>0</u>	<u>0.445</u>	<u>0</u>
Bias s.d. in %	<u>0.478</u>	<u>0</u>	<u>0.0180</u>	<u>0</u>
Est. Process s.d.	<u>320</u>	<u>242.3</u>	<u>231.7</u>	<u>243.7</u>
Process s.d. in %	<u>94.7</u>	<u>12.1</u>	<u>9.39</u>	<u>20.7</u>
No. of Outliers	<u>1</u>	<u>2</u>	<u>0</u>	<u>1</u>
% of Outliers	<u>6.7</u>	<u>28%</u>	<u>0%</u>	<u>1.9%</u>

Type of Material Various Cans and Containers

Characteristic Measured Gross Weight

Facilities A, B, C, D, E, F, G, H, I

	<u>Data Sets</u>			
Facilities	<u>F,B</u>	<u>G,B</u>	<u>C,B</u>	<u>C,B</u>
No. S/R Comparisons	<u>25</u>	<u>24</u>	<u>16</u>	<u>147</u>
Ave. Facility 1	<u>65.546</u>	<u>1375.4</u>	<u>15600.6</u>	<u>15059.1</u>
Ave. Facility 2	<u>65.491</u>	<u>1376.5</u>	<u>15596.1</u>	<u>15050.3</u>
Ave. (1) & (2)	<u>65.5185</u>	<u>1375.95</u>	<u>15598</u>	<u>15054.7</u>
Diff. (1) - (2)	<u>0.0547</u>	<u>-1.1</u>	<u>4.4</u>	<u>8.8</u>
% Difference	<u>0.0835%</u>	<u>-0.08%</u>	<u>0.028</u>	<u>0.058</u>
Student's t	<u>10.84</u>	<u>-7.7</u>	<u>5.4</u>	<u>18.5</u>
Est. Meas. s.d.	<u>0.0178</u>	<u>0.487</u>	<u>2.34</u>	<u>4.04</u>
Meas. s.d. in %	<u>0.0272%</u>	<u>0.0354%</u>	<u>0.0150</u>	<u>0.0268</u>
W Value	<u>0.939</u>	<u>0.949</u>	<u>0.970</u>	<u>--</u>
95% W Critical Value	<u>0.918</u>	<u>0.915</u>	<u>0.890</u>	<u>--</u>
Est. Bias s.d.	<u>0.0385</u>	<u>0.757</u>	<u>3.08</u>	<u>6.17</u>
Bias s.d. in %	<u>0.0588</u>	<u>0.055</u>	<u>0.020</u>	<u>0.041</u>
Est. Process s.d.	<u>27.0</u>	<u>136</u>	<u>525</u>	<u>60.0</u>
Process s.d. in %	<u>41.3%</u>	<u>9.9%</u>	<u>3.4%</u>	<u>4.0%</u>
No. of Outliers	<u>0</u>	<u>1</u>	<u>0</u>	<u>2</u>
% of Outliers	<u>0%</u>	<u>4.0%</u>	<u>0</u>	<u>1.3%</u>

Type of Material Various Cans and Containers
 Characteristic Measured Gross Weights
 Facilities A, B, C, D, E, F, G, H, I

Facilities	<u>Data Sets</u>			
	<u>B,H</u>	<u>I,B</u>		
No. S/R Comparisons	<u>7</u>	<u>20</u>		
Ave. Facility 1	<u>156476</u>	<u>16253.1</u>		
Ave. Facility 2	<u>156326</u>	<u>16213.5</u>		
Ave. (1) & (2)	<u>156401</u>	<u>16233.3</u>		
Diff. (1) - (2)	<u>149.7</u>	<u>39.55</u>		
% Difference	<u>0.10%</u>	<u>0.24%</u>		
Student's t	<u>7.6</u>	<u>13.5</u>		
Est. Meas. s.d.	<u>36.9</u>	<u>9.23</u>		
Meas. s.d. in %	<u>0.024%</u>	<u>0.057%</u>		
W Value	<u>0.882</u>	<u>0.833</u>		
95% W Critical Value	<u>0.803</u>	<u>0.905</u>		
Est. Bias s.d.	<u>104.9</u>	<u>27.9</u>		
Bias s.d. in %	<u>0.067%</u>	<u>0.172%</u>		
Est. Process s.d.	<u>5705</u>	<u>2050</u>		
Process s.d. in %	<u>3.65%</u>	<u>12.7%</u>		
No. of Outliers	<u>0</u>	<u>1</u>		
% of Outliers	<u>0%</u>	<u>4.8</u>		

IV. DEVELOPMENT OF THE DATA BASE FOR THIS STUDY

A. Data Acquisition

The basic data source for this study was the standard shipping document (AEC Form 741) utilized for recording nuclear materials transactions between facilities. In many cases, supplementary data were acquired from laboratory, quality control, and product specification reports. The aggregation of data in shipping documents and the use of shipper's data by the receiver were major reasons why additional data were required. Because the shipping document does not provide for the reporting of the complete span of isotopic composition, laboratory reports were used extensively as a source of isotopic data.

B. Comparability of Data

All measurement data had to be carefully screened for accuracy and completeness. Great care had to be exercised in the sorting of data to match like units of material and measurement data. The tracing of data through laboratory, quality control, and shipping records was complicated by identification changes made at different steps in the operation. It was observed that material receipts were sometimes measured in unit quantities different from those utilized by the shippers. In some instances, the comparative measurement data were reported in different quantitative units. A standardization of identification and reporting practices would greatly facilitate the comparison of S/R data.

C. Data Reporting and Analysis

The most complete and effective analysis of shipper-receiver data can be performed when measurement data are reported and tested on an individual container or lot basis. Measurement errors, shipping misstatements, and material losses associated with an individual container can be hid in the analysis of S/R differences on an overall shipment basis. When feasible, it is desirable that S/R comparisons be made individually for tare, gross, and net weights, element assay, and isotopic composition. As supplementary data, the reporting of density, impurity, moisture, and acid concentration results would be invaluable to the technical assessment of S/R differences. Because the AEC Form 741 cannot accomodate such reporting requests, it is recommended that a new measurement data form be designed for attachment to the present AEC Form 741. The standard format should be designed to facilitate computer treatment of recorded measurement data. To the degree that the desired data would already be available in production control and quality assurance records, the request for additional information in a new format would require little additional effort.

I. DATA ANALYSIS, DATA SUMMARIZATION, DATA EXPOSURE AND DISCUSSION

A. Discussion

The study was undertaken to obtain estimates of the essential variability aspect of historical S/R data for perspective and for possible use in the safeguarding of special nuclear material by the

statistics of mass balance accounting. The data give insights into the magnitude of the different error sources, into the type of measurement performances that have been carried out in the past and thus they serve as guides to the required, attainable, and desired performance parameters for the future.

The calculation of the statistics of MJF and S/R differences requires estimates of various measurement precisions, measurement bias variances and sometimes even the process variances. For this reason data which are capable of filling present gaps in such required information are useful.

While the methodology of statistics is exact, the decisions which affect the applications of statistics to practical matters involve understanding and skill in order to get the best results. For example, the matter of pooling variances is not a straightforward business. One needs to decide when pooling should be done, what are the possible gains and drawbacks and these considerations in turn are dependent upon the inspectorate's objectives. Reference [8] discusses pooling and this article indicates that the consequences of pooling are not, in general, easily portrayed nor easily evaluated.

The main impact from this study is that the results give perspective on the order of magnitude of the different variance components, i.e., the measurement precision, the bias and the process variances. If, for example, the study indicates that biases tend to be present in significant amounts, the attainment of a desired degree of sensitivity in a statistic may be severely delimited by the magnitude of this bias.

The measurement precisions for different sets of S/R data may be significantly different but these precisions may still be most revealing. Suppose, for example, that all the relative standard deviations of a characteristic in question based on fairly extensive sampling, are in the range of 0.05% to 0.10%. This tells quite a bit about the order of magnitude of the measurement precision characteristic whether or not the precisions are significantly different.

The treatment of the data falls into the broad category of data analysis rather than into statistical analysis per se [7]. The analysis attempts to give exposure to the main aspects of the data by summarizing the results in ways which can be of considerable help to prospective users. Some statistical techniques are used in certain cases which can be criticized on the grounds that the data do not obey all the required assumptions but again here the objective is not to provide a rigorous treatment but to be of help in summarizing data. Intermediate results of summarization are also presented so that the prospective user can go to these sources in order to use the data in other ways.

B. Combining Values

1. Precisions - If s_i^2 , the measurement variances with ν_i degrees of freedom, $i = 1, 2, \dots, n$, are based on normally distributed numbers, Bartlett's test may be used to test for the homogeneity of variances, i.e., that the true variances are the same. The Bartlett's test statistic has a χ^2 distribution approximately with $(k-1)$ degrees of freedom if the true variances are the same, where k is the number of sets of measurements which are involved. The

Bartlett's test is very sensitive to departures from normality. Since, in most cases, the W test of normality is included, one can use these W test results to gauge the appropriateness of the Bartlett's test.

The measurement variances are combined by using the relative standard deviations of measurements in the following way. Let r_i and v_i denote respectively for the i th class the estimate of the standard deviation of measurement and the degrees of freedom therein. The value

$$r = \sqrt{\sum_{i=1}^k v_i r_i^2 / \sum_{i=1}^k v_i}$$

is the pooled or composite relative standard deviation of measurement estimate. The choice of the relative standard deviation of measurement rather than the absolute standard deviation is based on the consideration that measurement variances tend to be a function of magnitude and if the magnitudes of the variable under consideration are similar from set to set, it is rather immaterial whether absolute or relative estimates are used. If the Bartlett's test shows the relative variances to be different, the largest variance is deleted and the composite relative standard deviation is then recomputed. These are all done for the convenience of the reader. The original standard deviations are also presented so that a user may composite the results in any desired manner. A lack of homogeneity of the variances may be due as well to a variance that is too small, or just due to a general lack of agreement. The rationale behind excluding the largest variance is that the composite value using the remaining measurement standard deviation estimates may be useful in gauging what is an attainable range of measurement performance values.

It was also felt that it would be more useful to the reader to present the results in terms of the relative standard deviations expressed in percent. This procedure makes the results from different areas more easily related.

2. Biases - Since one set of data generally estimates the bias variance with one degree of freedom and since this is independent of the number of values in the set, the variance component due to systematic error is represented as a relative standard deviation of bias expressed in percent and is obtained by taking the square root of the mean of the squares of the individual relative standard deviations of the different sets.

3. Process Variances - For the process variances, the overall process variance is obtained by pooling the results according to the number of degrees of freedom as with measurement precision and expressing the results as the percent standard deviations.

C. Additional Comments on Biases

The subject of the bias variance components can be rather complicated since biases can be introduced by people, instruments and environmental conditions. Thus, to say that different S/R differences from two facilities are going to have a constant bias $b = b_1 - b_2$ for the time period of concern assumes that the facility conditions are constant over the time period. What actually happens in treating

$$\hat{\sigma}_m^2 = 1/2 \sum_{i=1}^n (d_i - \bar{d})^2 / (n-1)$$

an estimate of measurement precision and

$$\hat{\sigma}_b^2 = \frac{\bar{d}^2}{2} - \frac{\hat{\sigma}_m^2}{n}$$

as an estimate of the bias variance is that local biases in time get pooled with the precision estimate and these are treated as averaging to zero in the bias variance estimate.

D. Some Statistics of Shipper-Receiver Variances

The following exposition of the statistical variation in S/R differences is introduced to show the relevance of some of the variance components given in this document. It is not to be regarded as an overall model for such studies as there are many pertinent characteristics which should be considered in a particular case.

The amount of Pu in a container is given by

$$x = (w_g - w_t)y = w_n y$$

where

w_g is the gross weight of the container and material

w_t is the container tare weight

w_n is the net weight, i.e., the estimate of the material weight

y is the Pu concentration (g/g)

Let a capped symbol denote an estimate. For example, $\hat{x}$ is an estimate of x . Then since

$$\frac{dx}{x} = \frac{dw_n}{w_n} + \frac{dy}{y}$$

the relative variance of the estimate of the amount of Pu is given by

$$\left(\frac{\hat{\sigma}_x}{\hat{x}}\right)^2 = \left(\frac{\hat{\sigma}_{w_n}}{w_n}\right)^2 + \left(\frac{\hat{\sigma}_y}{y}\right)^2$$

or

$$V_{\hat{x}} = V_{\hat{w}} + V_{\hat{y}} .$$

This assumes that $\hat{w}_n$ and $\hat{y}$ are statistically independent. Thus, if one wishes to test a S/R difference

$$d = \hat{x}_S - \hat{x}_R$$

one can use the approximate standard deviation

$$\sigma_d \approx \sqrt{\hat{x}_S^2 V_{\hat{x}_S} + \hat{x}_R^2 V_{\hat{x}_R}}$$

in the evaluation, assuming $\hat{x}_R$ and $\hat{x}_S$ are statistically independent. Generally, $\hat{x}_S$ and $\hat{x}_R$ will be close enough in value to use

$$\sigma_d = \hat{x} \cdot \sqrt{V_{\hat{x}_S} + V_{\hat{x}_R}}$$

where $\hat{x} = (\hat{x}_R + \hat{x}_S)/2$. If there is reason to believe that σ_{x_S} and σ_{x_R} are the same, due to theoretical and/or empirical evidence, then

$$\sigma_d = \hat{x} \cdot \sqrt{2V_{\hat{x}}} = \hat{\sigma}_{\hat{x}} \sqrt{2} .$$

If the d values are normally distributed and σ_d is known, d/σ_d will have a $N(\mu=0, \sigma=1)$ distribution if the S/R differences are due only to the measurement error sources which constitute σ_x^2 . This can be extended to cover averages of S/R differences where $d/\sigma_d =$

$\frac{\bar{d}\sqrt{n}}{\sigma_d}$ also has a $N(0,1)$ distribution under the null hypothesis that the difference is due only to measurement sources which constitute σ_x^2 . Student's t can be used in the situation where $\hat{\sigma}_x$ is estimated from the observed S/R data.

VI. DEFINITIONS AND FORMULAE

Symbol	Name	Meaning
μ_i	True amount	The true value of the amount measured in ith unit
b_j	Bias	The bias in the estimates which are made by the jth facility
ϵ_{ij}	Random measurement error	The random measurement error in the jth unit made by the ith facility
σ_m^2	Measurement precision variance	The variance which would occur under unlimited repetition of independent measurements of the same item by the same facility
μ	Process average	As used herein it is the true average of the quantities in the data set
$100\left(\frac{\sigma_m}{\mu}\right)\%$	Percent standard deviation of precision	The standard deviation of precision normalized in percentage terms
σ_b^2	Bias variance	The elements b_j are considered to be sampled from a population of facilities where the overall average of the b_j values is zero so that σ_b^2 is the variance of these bias elements
$100\left(\frac{\sigma_b}{\mu}\right)\%$	Percent standard deviation of bias	The bias standard deviation normalized for comparison and pooling purposes
σ_p^2	Process variance	The variance of the μ_i values, i.e., the variance in the true amounts of the items
$100\left(\frac{\sigma_p}{\mu}\right)\%$	Percent standard deviation of the process variance	The standard deviation of the distribution of the true amounts which are measured by the shipper and the receiver
W	W statistic	A statistic calculated for the purpose of testing whether the observations come from a normal distribution

<u>Estimate</u>	<u>Parameter Estimated</u>	<u>Formula</u>
$\bar{x}_i$	μ_i	$(x_{1i} + x_{2i})/2$
$\bar{x}$	μ	$\Sigma \bar{x}_i / n$
d_i	$b_1 - b_2$	$x_{1i} - x_{2i}$
$\bar{d}$	$b_1 - b_2$	$\Sigma d_i / n$
$\hat{\sigma}_m^2 = s_m^2$	σ_m^2	$\frac{\Sigma d_i^2 - (\Sigma d_i)^2 / n}{2(n-1)}$
$100 \left(\frac{s_m}{\bar{x}} \right) \%$	$100 \left(\frac{\sigma_m}{\mu} \right) \%$	
$\hat{\sigma}_b^2 = s_b^2$	σ_b^2	$\frac{\bar{d}^2}{2} - \frac{s_m^2}{n}$
$100 \left(\frac{s_b}{\bar{x}} \right) \%$	$100 \left(\frac{\sigma_b}{\mu} \right) \%$	
$\hat{\sigma}_p^2 = s_p^2$	σ_p^2	$s_x^2 - \frac{s_m^2}{2}$ where
		$s_x^2 = \frac{\Sigma \bar{x}_i^2 - (\Sigma \bar{x}_i)^2 / n}{n - 1}$
$100 \left(\frac{s_p}{\bar{x}} \right) \%$	$100 \left(\frac{\sigma_p}{\mu} \right) \%$	

VII. REFERENCES

The role of material accountability in the safeguarding of special nuclear materials is discussed in [1] - [4]. The efficiency of material accountability is limited by the accuracy and the precision of measurement, and there are situations where the process variability plays a role in safeguards. References [1] - [4] provide the basic reasons for the need for such estimates as are given in this document.

Background material for the use of the W statistic in testing whether a variable is normally distributed is given in [5].

Supplementary textual material on variance components, testing for differences in statistical averages and variances are given in [6]. In addition, there is an excellent discussion of measurement biases and precisions.

Reference [7] discusses data analysis per se, the relationship between data analysis and statistics, and the techniques of data analysis in summarizing data and in presenting data in useful ways. The authors treat statistical analysis as a useful tool in data analysis but as one which imposes a rather tight formal structure on the data. In the unexceptional case one wishes to examine data in almost all of its aspects and to give, in the author's words, "exposure" to the data.

- [1] Schneider, R. A., K. B. Stewart and D. P. Granquist, BNW Evaluation of the U.S. Resident Inspection Experiment, Official Use Only, BNWL-1360, June, 1970.
- [2] Schneider, R. A., and D. P. Granquist, Capability of a Typical Material Balance Accounting System for a Chemical Processing Plant, BNWL-1384, UC-15, May, 1970.
- [3] Wirfs, L. F., A Detailed Analysis of the Safeguards Capabilities of Material Balance Accounting System in the Low Enriched UO₂ Conversion and Fabrication Sectors of the Commercial Fuel Cycle, BNWL-1336, UC-15, January, 1970.
- [4] Wirfs, L. F., Safeguards Capabilities of the Material Balance Accounting Systems of the Plutonium Processing and Scrap Recovery Operations at Hanford, BNWL-1399, UC-15, June, 1970.
- [5] Hahn, G. J., and S. S. Shapiro, Statistical Models in Engineering, John Wiley and Sons, Inc., New York, 1967, pp. 294-302
- [6] Bennett, C. A., and N. L. Franklin, Statistical Analysis in Chemistry and the Chemical Industry, John Wiley and Sons, Inc., New York, 1954, pp. 176-208.
- [7] Tukey, J. W., and M. B. Wilk, Data Analysis and Statistics, Techniques and Approaches, Proceedings of the Symposium on Information Processing in Sight Sensory Systems, Nov., 1965, California Institute of Technology, Pasadena, California.
- [8] Mosteller, Frederick, On Pooling Data, Journal of the American Statistical Association, June, 1948, Vol. 43, pp. 231-242.

APPENDIX

A. Description of the Statistical Procedures

Following is a description of the techniques which are used in analyzing the S/R data. Some of the assumptions, derivations and interpretations of procedures are given in the situations where these procedures are not straightforward. An example is also included. Let

x_{1i} denote the i th measurement by the first facility

x_{2i} denote the i th measurement by the second facility

$d_i = x_{1i} - x_{2i}$ be a shipper-receiver difference

$\bar{d} = \sum_{i=1}^n d_i / n$ be the average difference

$s_d^2 = \left[\sum_{i=1}^n d_i^2 - \left(\sum_{i=1}^n d_i \right)^2 / n \right] / (n-1)$ be the estimated variance of the differences

s_d be the estimated standard deviation of the differences

$s_{\bar{d}} = s_d / \sqrt{n}$ be the estimated standard deviation of the average difference of n S/R comparisons

$t = \bar{d} / s_{\bar{d}}$ be the Student's t value for comparing the shipper and receiver values for a significant difference.

B. The Statistical Model

The model for an observation is given as

$$x_{ji} = \mu_i + b_j + \varepsilon_{ji}$$

where

μ_i is the true value

b_j is the bias of the j th facility

ε_{ji} is the random measurement error for the i th measurement, j th facility.

Then the model for d_i is

$$d_i = x_{1i} - x_{2i} = b_1 - b_2 + \varepsilon_{1i} - \varepsilon_{2i}$$

$$\sum_{i=1}^n d_i = nb_1 - nb_2 + \sum_{i=1}^n \varepsilon_{1i} - \sum_{i=1}^n \varepsilon_{2i}$$

$$\bar{d} = \sum_{i=1}^n d_i / n = b_1 - b_2 + \sum_{i=1}^n \varepsilon_{1i} / n - \sum_{i=1}^n \varepsilon_{2i} / n$$

$$d_i - \bar{d} = \varepsilon_{1i} - \varepsilon_{2i} + \sum_{j \neq i}^n \varepsilon_{1j} / n - \sum_{j \neq i}^n \varepsilon_{2j} / n = \varepsilon_{1i} - \frac{\sum_{j \neq i}^n \varepsilon_{1j}}{n} + \varepsilon_{2i} - \frac{\sum_{j \neq i}^n \varepsilon_{2j}}{n} .$$

C. Estimating the Measurement Variance

$$\text{Consider the value } \varepsilon_{1i} - \frac{\sum_{j \neq i}^n \varepsilon_{1j}}{n} = \varepsilon_{1i} \left(\frac{n-1}{n} \right) - \frac{\sum_{j \neq i}^n \varepsilon_{1j}}{n} .$$

Then its expected variance is

$$\sigma_1^2 \left[\left(\frac{n-1}{n} \right)^2 + \frac{n-1}{n^2} \right] = \sigma_1^2 \left(\frac{n-1}{n^2} \right) (n-1 + 1) = \sigma_1^2 \left(\frac{n-1}{n} \right) ,$$

and

$$E \left(\frac{\sum_{i=1}^n (\varepsilon_{1i} - \bar{\varepsilon}_{1i})^2}{n-1} \right) = \sigma_1^2 .$$

If we assume that $\sigma_m^2 = \sigma_1^2 = \sigma_2^2$, then

$$E \left(\frac{\sum_{i=1}^n (d_i - \bar{d})^2}{n-1} \right) = \sigma_1^2 + \sigma_2^2 = 2\sigma_m^2 \text{ say where } \sigma_m^2 \text{ is the measurement}$$

precision variance, and σ_m^2 is estimated by

$$\hat{\sigma}_m^2 = 1/2 \left(\frac{\sum_{i=1}^n (d_i - \bar{d})^2}{n-1} \right) = 1/2 s_d^2 = 1/2 \left(\frac{\sum_{i=1}^n d_i^2 - \left(\sum_{i=1}^n d_i \right)^2 / n}{n-1} \right) .$$

D. Estimating the Bias Variance Component

In order to obtain an estimate of σ_b^2 , the bias variance component, one proceeds as follows. Since

$$E(\bar{d}^2) = E(b_1 - b_2 + \bar{\varepsilon}_1 - \bar{\varepsilon}_2)^2 = \sigma_{b_1}^2 + \sigma_{b_2}^2 + 2\sigma_m^2/n$$

where b_1 and b_2 are assumed to be sampled out of a population with mean zero and variance σ_b^2 , and if we let $\sigma_b^2 = \sigma_{b_1}^2 = \sigma_{b_2}^2$ one has the estimate

$$\hat{\sigma}_b^2 = \frac{\bar{d}^2}{2} - \frac{\hat{\sigma}_m^2}{n} = 1/2 \left(\bar{d}^2 - \frac{s_d^2}{n} \right)$$

If $\hat{\sigma}_b^2 \leq 0$, then $\hat{\sigma}_b^2$ is estimated as zero. Since $\bar{d}^2 - \frac{s_d^2}{n} = 0$ implies that

$$\left| \frac{\bar{d}}{s_d/\sqrt{n}} \right| = 1, \text{ the bias variance component has a non-zero estimate when}$$

|Student's t | is larger than 1.

E. Estimating the Process Variance

Let

$$X_i = (x_{1i} + x_{2i})/2.$$

The model for X_i is

$$X_i = (\mu_i + b_1 + \varepsilon_{1i} + \mu_i + b_2 + \varepsilon_{2i})/2 = \mu_i + \frac{b_1 + b_2}{2} + \frac{\varepsilon_{1i} + \varepsilon_{2i}}{2}$$

$$\sum_{i=1}^n X_i = \sum_{i=1}^n \mu_i + n \left(\frac{b_1 + b_2}{2} \right) + 1/2 \sum_{i=1}^n (\varepsilon_{1i} + \varepsilon_{2i})$$

$$\bar{X} = \frac{\sum_{i=1}^n X_i}{n} = \mu. + \frac{b_1 + b_2}{2} + \frac{1}{2n} \sum_{i=1}^n (\varepsilon_{1i} + \varepsilon_{2i})$$

$$X_i - \bar{X} = \mu_i - \mu. + 1/2(\epsilon_{1i} + \epsilon_{2i} - \epsilon_{1.} - \epsilon_{2.})$$

$$= \mu_i - \mu. + 1/2 \left[\epsilon_{1i} \left(\frac{n-1}{n} \right) - \frac{\sum_{j \neq i}^n \epsilon_{1j}}{n} + \epsilon_{2i} \left(\frac{n-1}{n} \right) - \frac{\sum_{j \neq i}^n \epsilon_{2j}}{n} \right]$$

$$\begin{aligned} E(X_i - \bar{X})^2 &= \sigma_p^2 \left[\left(\frac{n-1}{n} \right)^2 + \frac{n-1}{n^2} \right] + (1/4)(2) \left[\left(\frac{n-1}{n} \right)^2 + \frac{n-1}{n^2} \right] \sigma_m^2 \\ &= \sigma_p^2 \left(\frac{n-1}{n} \right) + 1/2 \left(\frac{n-1}{n} \right) \sigma_m^2 \end{aligned}$$

where σ_p^2 is the process variance. Then

$$E(s_X^2) = \frac{E \sum (X_i - \bar{X})^2}{n-1} = \frac{n}{n-1} \left[\sigma_p^2 \left(\frac{n-1}{n} \right) + \frac{\sigma_m^2}{2} \left(\frac{n-1}{n} \right) \right] = \sigma_p^2 + \frac{\sigma_m^2}{2}$$

Thus

$$s_p^2 = s_X^2 - \frac{s_m^2}{2} = s_X^2 - \frac{s_d^2}{4}$$

provides an estimate of the process variance.

F. RELATIVE VARIANCES AND STANDARD DEVIATIONS

The results are also put in terms of the relative variances and relative standard deviations as percentages. The estimated relative standard deviation of measurement in percent is for example

$$(RS_m) = \frac{s_m \times 100\%}{\bar{\bar{x}}}$$

where $\bar{\bar{x}} = (\bar{x}_1 + \bar{x}_2)/2$. The relative variance in $(\%)^2$ terms is then

$$RV_m = (RS_m)^2 = \frac{(s_m \times 100\%)^2}{(\bar{\bar{x}})^2}$$

similar expressions are used for the bias and process variance components.

G. OUTLIERS

r_i , the residual of d_i , can be written in the form

$$r_i = d_i - \bar{d} = d_i - \frac{\sum_{j=1}^n d_j}{n} = d_i - \frac{d_i}{n} - \frac{\sum_{j \neq i}^n d_j}{n} = d_i \left(\frac{n-1}{n} \right) - \frac{\sum_{j \neq i}^n d_j}{n} .$$

Then

$$\begin{aligned} \sigma_{r_i}^2 &= \sigma_d^2 \left[\left(\frac{n-1}{n} \right)^2 + \frac{\sum_{j \neq i}^n \frac{1}{n^2}}{n^2} \right] = \sigma_d^2 \left[\frac{(n-1)^2}{n^2} + \frac{n-1}{n^2} \right] = \sigma_d^2 \left[\frac{(n-1)}{n^2} (n-1+1) \right] \\ &= \sigma_d^2 \left(\frac{n-1}{n} \right) . \end{aligned}$$

Then

$$s_r = s_d \sqrt{\frac{n-1}{n}} \text{ estimates } \sigma_r$$

and

$$\frac{r_i}{s_r} = \frac{r_i}{s_d} \sqrt{\frac{n}{n-1}}$$

is a normalized residual.

The outliers were defined as the values of the differences $d_i - \bar{d}$ which exceed $C\sigma_d$, or where

$$\frac{d_i - \bar{d}}{s_r} > C .$$

H. THE TEST FOR NORMALITY

The W value to test for normality is computed as follows.

1. The differences are arranged from the smallest to the largest so that Z_1 is the smallest difference, Z_n is the largest difference, and Z_j is the j th smallest.

2. The value

$$b = A_n(Z_n - Z_1) + A_{n-1}(Z_{n-1} - Z_2) + \dots + A_{n-k+1}(Z_{n-k+1} - Z_k)$$

where $k = n/2$ or $(n-1)/2$ according as n is even or odd, is calculated.

These are given in (5), pp. 330-331.

3. The statistic

$$W = \frac{b^2}{S_{dd}} = \frac{b^2}{(n-1)s_d^2} = \frac{b^2}{\left[\frac{n}{\sum d_i^2} - \left(\frac{n}{\sum d_i} \right)^2 / n \right]}$$

is calculated and compared with the $100(1-\alpha)$ percentile value of the statistic under the assumption of normality which is also obtained from (5), page 332. In this test values which are smaller than the significance level indicate a departure from normality.

There is a useful theorem on normality which is applicable here. The theorem states that if

$$y = a_1x_1 + a_2x_2$$

is normally distributed and x_1 and x_2 are statistically independent then x_1 and x_2 are normally distributed. The relevance here is that if the d values are normally distributed then the measurement errors of x_1 and x_2 are normally distributed.

Non-rejection of the null hypothesis in tests of normality does not prove normality, however, it only indicates that the data are consistent with the hypothesis of normality. Thus, if there is no reason to suspect that differences are not normally distributed, the same holds for the precision component of the individual x values.

I. EXAMPLE

The original data include 13 pairs of S/R Pu assay values.

<u>i</u>	<u>x_{1i}</u>	<u>x_{2i}</u>	<u>d_i</u>	<u>$d_i - \bar{d}$</u>	<u>$(d_i - \bar{d}) / \sqrt{(d_i - \bar{d})}$</u>
1	0.1317	0.1310	.0007	-.00016	0.2
2	0.1396	0.1410	-.0014	-.00194	-2.7
3	0.1204	0.1211	-.0007	-.00124	-1.7
4	0.1354	0.1354	.0000	-.00054	-0.8
5	0.1375	0.1348	.0027	.00216	3.0
6	0.1382	0.1376	.0006	.00006	0.1
7	0.1444	0.1444	.0004	-.00014	-0.2
8	0.1449	0.1432	.0017	.00116	1.6
9	0.1556	0.1549	.0007	.00016	0.2
10	0.1559	0.1553	.0006	.00006	0.1
11	0.0717	0.0720	-.0003	-.00084	-1.2
12	0.1478	0.1472	.0006	.00006	0.1
13	0.1430	0.1423	.0007	.00016	0.2
14	0.1553	0.1544	.0009	.00036	0.5
15	0.1468	0.1461	.0007	.00016	0.2
16	0.1572	0.1565	.0007	.00016	0.2

On the basis of the outlier test, observations 2 and 5 are deemed to be outliers. The new set of data then does not include these values. The new set of data is as follows.

<u>i</u>	<u>x_{1i}</u>	<u>x_{2i}</u>	<u>d_i</u>	<u>$d_i - \bar{d}$</u>	<u>$\frac{d_i - \bar{d}}{s_{d_i - \bar{d}}}$</u>
1	0.1317	0.1310	.0007	.0000	.0
2	0.1204	0.1211	-.0007	-.0014	-1.9
3	0.1354	0.1354	.0000	-.0007	-1.0
4	0.1382	0.1376	.0006	.0001	0.1
5	0.1444	0.1440	.0004	-.0003	-0.4
6	0.1449	0.1432	.0017	.0010	1.5
7	0.1556	0.1549	.0007	.0000	0.0
8	0.1559	0.1553	.0006	-.0001	-0.1
9	0.0717	0.0720	-.0003	-.0010	-1.4
10	0.1478	0.1472	.0006	-.0001	-0.1
11	0.1430	0.1423	.0007	.0000	0.0
12	0.1553	0.1544	.0009	.0002	0.3
13	0.1468	0.1461	.0007	.0000	0.0
14	0.1572	0.1565	.0007	.0000	0.0

J. MEASUREMENT VARIANCE

$$\bar{d} = 0.0073/14 = 0.00052$$

$$\Sigma d_i^2 = 797 \times 10^{-8}$$

$$\Sigma d_i = 73 \times 10^{-4}$$

$$(\Sigma d_i)^2/n = 380.64 \times 10^{-8}$$

$$Sdd = \Sigma d_i^2 - (\Sigma d_i)^2/n = 416.357 \times 10^{-8}$$

$$s_d^2 = Sdd/(n-1) = 32.03 \times 10^{-8}$$

$$s_d = 5.66 \times 10^{-4}$$

$$s_{\bar{d}} = s_d/\sqrt{14} = 1.5125 \times 10^{-4}$$

$$t = \bar{d}/s_{\bar{d}} = \frac{5.2 \times 10^{-4}}{1.5125 \times 10^{-4}} = 3.44$$

$$s_m^2 = \frac{s_d^2}{2} = 16.01 \times 10^{-8} = 1.601 \times 10^{-7}$$

$$s_m = \sqrt{16.01} \times 10^{-4} = 4.00 \times 10^{-4}$$

$$\bar{x}_1 = 0.13916, \bar{x}_2 = 0.13864, \bar{\bar{x}} = 0.13890$$

$$Rs_m = \frac{(0.0004) \times 100\%}{0.13890} = 0.288\%$$

$$RV_m = (Rs_m)^2 = 8.29 \times 10^{-2}(\%)^2$$

K. TEST OF NORMALITY

The differences arranged from smallest to largest are

-0.0007

-0.0003

0.0000

0.0004

0.0006

0.0006

0.0006

0.0007

0.0007

0.0007

0.0007

0.0007

0.0009

0.0017

Then b is

$$\begin{aligned} b &= 0.5251[0.0017 - (-0.0007)] \\ &\quad + 0.3318[0.0009 - (-0.0003)] \\ &\quad + 0.2460[0.0007 - 0.0000] \\ &\quad + 0.1802[0.0007 - 0.0004] \\ &\quad + 0.1240[0.0007 - 0.0006] \\ &\quad + 0.0727[0.0007 - 0.0006] \\ &\quad + 0.0240[0.0007 - 0.0007] \\ &= 0.00190433, \end{aligned}$$

and

$$W = \frac{b^2}{S_{dd}} = \frac{(0.00190433)^2}{0.000004164} = 0.87$$

Since the 95 percentile point of W under a normal distribution is 0.874, there is borderline evidence of non-normality.

L. BIAS COMPONENT ESTIMATE

The bias variance component is estimated by

$$s_b^2 = \frac{\bar{d}^2}{2} - \frac{s_m^2}{n} = \frac{(5.2 \times 10^{-4})^2}{2} - \frac{16.01 \times 10^{-8}}{14} = 12.4 \times 10^{-8}$$

$$s_b = 3.5 \times 10^{-4}$$

$$Rs_b = \frac{s_b \times 100\%}{\bar{\bar{x}}} = \frac{(3.5 \times 10^{-4})(100\%)}{0.1389} = 0.25\%$$

$$RV_b = (rs_b)^2 = 6.3 \times 10^{-2} (\%)^2$$

M. PROCESS VARIANCE ESTIMATES

X_i is defined as $(x_{1i} + x_{2i})/2$. Then one has the following table.

<u>i</u>	<u>X_i</u>
1	0.13135
2	0.12075
3	0.13540
4	0.13790
5	0.14420
6	0.14405
7	0.15525
8	0.15560
9	0.07185
10	0.14750
11	0.14265
12	0.15485
13	0.14645
14	0.15685

Then

$$\sum X_i^2 = 0.276336625$$

$$\sum X_i = 1.944650$$

$$\bar{X} = 0.13890$$

$$(\sum X_i)^2/n = 0.270118829$$

$$SXX = 0.0062178$$

$$s_X^2 = 0.0004782$$

$$s_p^2 = s_X^2 - s_m^2/2 = 0.0004780$$

$$s_p = 0.0219$$

$$R_{s_p} = \frac{s_p \times 100\%}{\bar{X}} = 15.74\%$$

$$RVp = (sr_p)^2 = 247.7(\%)^2$$

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