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TRANSFER MEASUREMENT STATISTICS DERIVED FROM
PUREX PROCESSING MEASUREMENTS' QUALITY CONTROL PROGRAM

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TRANSFER MEASUREMENT STATISTICS DERIVED FROM PUREX PROCESSING MEASUREMENTS' QUALITY CONTROL PROGRAM

INTRODUCTION

The Purex Processing Measurements Quality Control Program was designed to provide for the orderly accumulation of measurements data which are suitable for statistical evaluation and to identify those elements of a measurement which, if improved, would result in significant reduction in overall measurement variation. This report gives an analysis of the audit data obtained at four transfer stations: L9 plutonium product loadout, K6 uranium product to storage, F15 salt waste to F16 and D5 dissolved feed to process. Estimates of variation caused by analytical, sampling and volume or weight instrumentation were computed and combined to give variation associated with a single batch transfer. Procedures for collection of future data that are more suitable for statistical evaluation are suggested.

SUMMARY AND RECOMMENDATIONS

The methods used to compute random and fixed variation estimates are discussed in the text. The estimates so determined are summarized in Table 2, 5, 6, and 7 for the L9 plutonium, K6 uranium, F15 waste and D5 feed measurements, respectively. In all cases there were no data available to estimate fixed variation due to sampling. In fact, it is doubtful that there are any convenient or economical methods for collecting this kind of data except at the time the plant is built and before operation begins. All other sources of variation were estimated and combined to obtain the overall variation associated

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with a batch transfer. It was determined that:

1) L9 Plutonium Product Loadout

- a) Analytical random variation is controlling on a batch basis followed by analytical fixed variation. Other sources of variation appear to be small relative to analytical random and fixed variations. However, random variation due to sampling may be understated because the duplicate samples are taken from L9 instead of from each of the five PR cans.
- b) For longer time periods (one month or greater) analytical fixed variation is controlling.

2) K6 Uranium Product to Storage

- a) Sampling and analytical (X-ray) random variations are controlling on a batch basis. The other sources of variation are relatively small.
- b) For longer periods of time (one month or greater) fixed variation due to tank calibration slope estimate is controlling.

3) F15 Salt Waste to F16

- a) Random variation due to sampling is controlling on a batch basis. All other sources of variation are small compared to sampling and analytical.
- b) For longer time periods, random variation due to sampling is also controlling, however, we suspect that if it were possible to estimate fixed variation due to sampling, then fixed variation would be more controlling than random variation.
- c) Because product throughput is so small at this transfer station,

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the random and fixed variations due to sampling probably have a negligible effect on plant product balance.

4) D5 Feed to Process

- a) Estimates of variation due to volume measurement (original 1955 water calibrations with meter pump) were obtained. An estimate of combined sampling and analytical variation for the U analysis was obtained, but the estimate is based on a very small amount of data. Overall variation due to batch content could not be determined because of insufficient data.

The following items are suggestions for improvement of the data collection program:

1. The "PR Scale Calibration Check" and the "Gross Weight Check" can be deleted. The "Tare Weight Check" should be continued at least once per week. Once each month the data can be averaged and a bias correction factor computed. If the correction factor is significant, the month's loadouts can be adjusted. Also, a PR "Scale Calibration Check" should be made and the scale adjusted to eliminate the source of the bias. The data can also be used to estimate fixed variation. The principle here is basically the same as that used in the Analytical Control Standards Program.
2. Random variation due to sampling is computed from analytical results obtained on two independent samples of the same batch. It is important that one of the duplicate samples be analyzed a second time (Referee). All three results should be recorded on the data form.
3. It is not necessary to log Analytical Control Referee and Standards Program data on the form. However, the monthly IBM reports should be

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available for the data period in question.

4. All of the instrument calibration data were of no value from a statistical estimating viewpoint. The operator must enter the actual measurement to the nearest readable scale division - before adjustments are made to the instrument. In the case of Weight Factor instruments, readings should be made at the average transfer levels in addition to 0, 40, 80, 120, and 160 inches. Unless gross errors are indicated the instrument should not be adjusted. Assuming these checks are made once per week, the data collected during the month can be used to compute an average bias factor for a typical transfer. If significant, a correction can be made and/or the instrument adjusted to eliminate the bias. Again, the basic principle here is the same as the Analytical Control Standards Program. If the service instrument is a manometer, the temperature of the manometer fluid should be recorded on the form. Also, if the standard instrument is a manometer, the temperature of the standard manometer fluid should be recorded on the form. When a standard manometer is used to check the calibration of a Weight Factor or Specific Gravity Recorder, precautions should be taken to be sure that the recorder is not adjusted to the standard manometer if the present temperature of the manometer fluid is significantly different from the fluid temperature of the previous calibration check. Appropriate temperature corrections should be made before adjusting the recorder.

5. Precision and accuracy of Specific Gravity instruments can be determined by requesting a laboratory Specific Gravity analysis each time a duplicate sample is taken and comparing the laboratory result with the

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recorder result. Both samples should be analyzed by the laboratory. Two recorder results should also be obtained, one for each sampling time. All four specific gravity results should be recorded on the form.

The following comments pertain to tank calibration procedures:

1. It is doubtful that repeated calibrations of a vessel (more than two or three times) is worth the effort for the amount of improvement attained. More significant improvements can be attained by changing the liquid level instrument. For example, changing from a transmitter-recorder to a No. 3 oil manometer can improve accuracy by a factor of 2-3 times. Changing from a No. 3 oil manometer to a precision No. 1 oil manometer can improve accuracy by an additional factor of 2-3 times.^(4,5) Very nearly the same accuracy can be attained with the Wallace and Tiernan Model FA 145 precision pressure gauge which is much more mobile and has less maintenance problems (compared to manometer fluids). Once an instrument has been selected, future accuracy depends almost completely on periodic maintenance and calibration checks.

2. All calibration tables should be identified with some particular manometer fluid temperature. Temperature corrections should be applied when the daily temperature of the manometer fluid deviates significantly from the base temperature.

PR LOADOUT MEASUREMENT

Four checks of the PR loadout measurement are made: PR Scales Calibration Check, Weighing Procedure Audit, Sampling Procedure Audit, and Analytical Control Audit. The PR Scales Calibration Check is of little value from a

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statistical estimating viewpoint. The main reason for this is because the operator usually indicates "OK" instead of entering the actual check measurement to the nearest readable scale division before adjustments are made to the scale. No variation estimates can be made without an actual measurement that can be compared to the standard value of the checkweight. The remaining three audits are satisfactory and provide necessary information for obtaining variation estimates. These are discussed below.

Weighing Procedure Audit - Periodic checks (Tare Weight Check) of the Purex PR scales are made by weighing empty PR cans, adding a calibrated check weight that approximates the weight of L9 solution loaded out, and weighing the PR can plus check weight. The difference between the gross and tare weights should average out to the calibrated value of the check weight. Since this procedure is performed twice under independent conditions, an estimate of scale bias, random variation ("within independents" and "between independents") and fixed variation can be determined. In addition, PR cans that have been loaded out with product solution (Gross Weight Check) are weighed in duplicate. While these data do provide variation estimates, it is considered unnecessary as long as "tare weight check" data are available.

Tare weight check data were available for a total of 18 PR cans that were weighed on the Purex scales during the period October 1964 - June 1965. The differences between the "tare plus check weight" and "tare weight" are summarized in Table 1. The data were analyzed by analysis of variance techniques described by Grubbs⁽¹⁾.

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The scale bias for the first weighing (accounting weight) was + 1.1 grams and for the second weighing (independent) was + 3.7 grams. The average bias of both first and second weighings (36 measurements) was +2.4 grams. The "within independents" random variation (standard deviation) estimate was ± 20 grams for a single "by difference" (net weight) measurement. The "between independents" random variation (standard deviation) estimate was ± 28 grams for a single by difference (net weight) measurement. The "between independents" estimate is considered to be most representative of random variation introduced by the scale and scale operators. Based on an average can loadout of 10.4 kilograms, the ± 28 gram standard deviation is equivalent to ± 0.27 in percent units, or approximately 0.54 percent at the 0.95 confidence level.

The 0.95 confidence interval for the scale bias in grams is

$$2.4 - (2.11)(28)/\sqrt{18} < \beta < 2.4 + (2.11)(28)/\sqrt{18}$$
$$-11.5 < \beta < 16.3$$

where 2.11 = Students t for 0.95 confidence level and $n - 1 = 17$ degrees of freedom. This means we are 0.95 confident that the true bias β lies within the stated interval. Since the interval includes zero, we conclude the observed bias (2.4 grams) is not statistically different from zero. However, there may be technical reasons for concluding the 2.4 gram bias is significant and worthy of correction.

Fixed variation (standard deviation) of scale measurement is obtained by

$$FE = \pm 28/\sqrt{4} = \pm 14 \text{ grams or } 0.13 \text{ percent}$$

for an average can loadout of 10.4 kilograms (0.26 percent at 0.95 level).

This estimate assumes that the scale is tested by the tare weight check method four times each month and a bias correction (or scale adjustment) is made each month based on the average of the four tests.

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The "PR Scales Calibration Check" and the "Gross Weight Check" can be deleted from the measurements quality control program. The "Tare Weight Check" should be continued, and the data analyzed routinely. When the "Tare Weight Check" indicates a loss of control, then a bias correction should be made or a "PR Scales Calibration Check" should be made and the scale adjusted to eliminate the source of the bias.

Sampling Audit - In order to obtain good estimates of sampling variation, analytical variation must be determined from the same data used to estimate sampling variation. For example, the first sample is taken and the original analysis is made in the laboratory. Then a second sample is taken and analyzed in the laboratory by a different analyst. Thirdly, the first sample is analyzed a second time by a different analyst. The analyst that made the second determination of the first sample and the analyst who made the only determination of the second sample can be the same analyst if necessary. All three results should be recorded on the data sheet. Then analytical variation and sampling variation can be estimated from the following formulas:

$$S_{s+a} = (0.8862) \bar{R}_{s+a}$$

$$S_a = (0.8862) \bar{R}_a$$

$$S_s = [(S_{s+a})^2 - (S_a)^2]^{1/2}$$

where

S_s = standard deviation due to sampling

S_a = standard deviation due to analytical

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S_{s+a} = standard deviation due to sampling
and analytical

$\bar{R}_a$ = average range between first and second
determinations of the first sample

$\bar{R}_{s+a}$ = average range between original determina-
tions of first and second samples

Random variation due to sampling was estimated (Table 2) for all three analytical methods - SpG., AT and Pu X-ray. But it was necessary to use the long-term average range from the Referee program instead of the actual data described above. Therefore, the estimates are approximate only.

It is not possible to estimate fixed variation due to sampling from these data. Special thief samples from PR cans would have to be obtained in order to evaluate fixed variation. A good example of this type of program is given in reference 2, page 35, 42-44.

Analytical Control Audit - It is not necessary to list Standard Program and Referee Program results on the data form. Although the monthly average Referee and Standard results and confidence limits should be available so that random variation and fixed variation estimates and bias corrections can be determined. Random variation due to analytical is obtained from the Referee Program. Fixed variation is obtained from the Standards Program and, in particular, from the stated confidence limits associated with the bias correction. For example, if the recovery for the PuX-3a method averaged 100.3 percent based upon nine determinations in one month, and the standard deviation was one percent, the bias correction and 0.95 confidence limit would be

$$+0.3 \pm (2.31)(1.0)/\sqrt{9}$$

$$+0.3 \pm (2.31)(0.33)$$

$$+0.3 \pm 0.76$$

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where 2.31 is Students t for the 0.95 level and $n-1 = 8$ degrees of freedom. The fixed variation (0.95 level) is 0.76 percent. The methods described in reference 3 should be used when computing the effect of analytical fixed variation for time periods greater than one month.

The Standards Program data were not available for this study. Thus, the fixed variation estimates summarized in Table 2 are taken directly from Reference 3. These estimates should be very reasonable provided the same analytical method is still in service, and approximately nine standard determinations are made per month.

Propagation of Errors - Procedures for combining all random and fixed error (variation) estimates into one overall statement for a batch transfer have been given in references 2, 3 and 4. As an example, the following general formula was used for the L9.

$$\frac{V(TU)}{(TU)^2} = \left[\frac{Vr(C)}{mn\bar{C}^2} + \frac{Vr(D)}{pn\bar{D}^2} + \frac{Vr(W)}{tn\bar{W}^2} \right] + \left[\frac{Vs(C)}{k\bar{C}^2} + \frac{Vs(D)}{k\bar{D}^2} + \frac{Vs(W)}{k\bar{W}^2} \right]$$

where

V = Variance = S^2 = standard deviation squared

TU = Total units in can, batch, month, etc.

$\bar{C}$ = Average concentration of plutonium solution

$\bar{D}$ = Average density of plutonium solution

$\bar{W}$ = Average weight of plutonium solution

k = Number of months

m = Number of concentration analyses

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n = Number of batches

p = Number of density analyses

r = Denotes random error

s = Denotes fixed error

t = Number of net weight measurements per batch

V(TU) = Variance of total units in can, batch, etc.

V_r(C) = Random error variance due to a reported concentration analysis

V_s(C) = Fixed error variance due to monthly bias correction estimate for concentration analyses.

V_r(D) = Random error variance due to a reported density analysis

V_s(D) = Fixed error variance due to monthly bias correction estimate for density analyses

V_r(W) = Random error variance due to one net weight measurement

V_s(W) = Fixed error variance due to monthly adjustment of bias in PR scale

The variance terms are obtained from Table 2 by taking the 0.95 CL statement in units, dividing by two and substituting in the above equation:

$$\begin{aligned} \frac{V(TU)}{(TU)^2} &= \left[\frac{(0.013)^2}{(1)(1)(2.04)^2} + \frac{(0.004)^2}{(1)(1)(1.50)^2} + \frac{(0.028)^2}{(5)(1)(10.4)^2} \right] \\ &\quad + \left[\frac{(0.007)^2}{(1)(2.04)^2} + \frac{(0.0005)^2}{(1)(1.50)^2} + \frac{(0.014)^2}{(1)(10.4)^2} \right] \\ &= 0.00004060 + 0.00000711 + 0.00000145 \\ &\quad + 0.00001177 + 0.00000011 + 0.00000181 \\ &= 0.00004916 + 0.00001369 \\ &= 0.00006285 \end{aligned}$$

Taking the square root, we obtain standard deviation:

$$\frac{S(TU)}{TU} = 0.0079$$

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which is 0.79 in percent units. The approximate 0.95 confidence limit* is twice this result, or about 1.6%.

The same result is obtained by working directly with the percent units given in Table 2 (after dividing by two):

$$\begin{aligned}
 \% \text{ TOTAL ERROR (0.95CL)} &= 2 \left\{ \frac{[\%S_r(C)]^2}{mn} + \frac{[\%S_r(D)]^2}{pn} + \frac{[\%S_r(W)]^2}{tn} \right. \\
 &\quad \left. \frac{[\%S_s(C)]^2}{k} + \frac{[\%S_s(D)]^2}{k} + \frac{[\%S_s(W)]^2}{k} \right\}^{1/2} \\
 &= 2 \left[\frac{(0.64)^2}{(1)(1)} + \frac{(0.26)^2}{(1)(1)} + \frac{(0.27)^2}{(5)(1)} \right. \\
 &\quad \left. + \frac{(0.35)^2}{(1)} + \frac{(0.05)^2}{(1)} + \frac{(0.13)^2}{(1)} \right]^{1/2} \\
 &= 2 (0.4918 + 0.1419)^{1/2} \\
 &= 2(0.796) \\
 &= 1.6
 \end{aligned}$$

*The confidence limit is actually a "two sigma" limit which is "approximately" equivalent to the 0.95 confidence level. All limits in this report are two sigma limits except where Students t factors are specifically used as multipliers.

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K6 UNH LOADOUT MEASUREMENT

Three checks of the K6 UNH loadout measurement are made: Instrument Calibration Check, Sampling Procedure Audit, and Analytical Control Audit. As with the PR Scale Check, the instrument check is of little value from a statistical estimating viewpoint. Actual observed measurements must be recorded so that they can be compared to the standard manometer. Brief discussions of the three audits are given below, followed by a section on tank calibration.

Instrument Calibration Check - The service manometer check should be continued on a weekly basis because performance of the service manometer is critical to nuclear materials accounting. In addition to checking the manometer at 0, 40, 80, 120, and 160 inches, checks should be made at 23 and 155 inches, the average UNH transfer readings. The service manometer should be read and recorded to the nearest readable scale division before any corrective adjustments are made. Also, fluid temperature and fluid type should be recorded on the form for both manometers.

The weight factor and the Sp. Gr. recorder instruments are not used in nuclear materials measurement. Continuation of these checks should depend on operational needs.

Sampling Procedure Audit - Sampling error was estimated as described in the L9 section above. All three analytical results should be recorded. The sampling error estimates given in Table 5 are only approximate because one of the three analyses was not available.

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Analytical Control Audit - The remarks given for the L9 also apply here.

Tank Calibration - Considerable time was spent processing tank calibration data obtained during July 1963, February 1964, and January 1965. The Fortran computer code that fits a straight line model to data having cumulative errors was revised and improved considerably. Copies of the code and instruction in its use are available from the author by request. The results are summarized in Table 3.

There are three sections in the K6 tank corresponding approximately to the intervals 0-6, 6-27, and 27-104 inches (water at 4°C). Separate lines were fit in each interval. The variance estimates are about the same for both the standard and service manometers, and the slope estimates are in good agreement. The slope and intercept estimates are based on a manometer containing 2.94 red oil at 68°F. Temperature corrections would have to be made when the manometer is operated at temperatures other than 68°F. The existing calibration table (Revised January 1965) is believed to be based upon a standard manometer that is operated at 86°F although this could not be determined with certainty. Table 4 is a comparison of three curves for a typical UNH transfer, assuming a UNH specific gravity of 1.65 and a manometer fluid temperature of 86°F.

The differences are small. The service manometer line obtained by fitting of straight lines to three sets of calibration data having cumulative errors is probably the most valid because the service manometer is the manometer actually in use.

The random error, fixed error and total error associated with a typical transfer volume are determined by use of the following formula:

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$$\text{Random error (0.95 CL)} = 2[(X_1 - X_0) V(e)]^{1/2}$$

$$\text{Fixed error (0.95CL)} = 2[(X_1 - X_0)^2 V(b)]^{1/2}$$

$$\text{Total error (0.95CL)} = 2[(X_1 - X_0)^2 V(b) + (X_1 - X_0) V(e)]^{1/2}$$

To convert from gallons to percent, multiply the above equations by 100 and divide by the number of gallons transferred. Using the service manometer and the data from Tables 3 and 4 for an example, we obtain

$$\% \text{ Random error (0.95CL)} = 2 [(79.36)(7.4843)]^{1/2} (100)/3764 = 1.3$$

$$\% \text{ Fixed error (0.95CL)} = 2 [(79.36)^2(0.07266)]^{1/2} (100)/3764 = 1.14$$

$$\% \text{ Total error (0.95CL)} = 2 [(79.36)^2(0.07266) + (79.36)(7.4843)]^{1/2} (100)/3764 = 1.73$$

The fixed error is uncertainty due to the calibration line (slope estimate). This uncertainty can be reduced by repeating the tank calibration more than once. The random error is uncertainty due to difference between the full and empty manometer readings. As such, it includes uncertainty due to the mechanical system (manometer, air supply, etc.) and the uncertainty associated with the ability of the operator to read the manometer scale.

Reference 3 attributes a fixed error to the manometer difference in addition to the random error. The reason for this is because past experience has shown that the manometer fluid density becomes diluted from time to time, thereby introducing a bias until such time as the trouble is detected and corrected. If the measurements quality control program is continued and the service manometer check is made once a week as suggested in a previous section, bias would essentially be eliminated by application of appropriate corrections to the affected manometer differences. Estimates of fixed error

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associated with such bias corrections are not given in Table 5 because no bias correction data were available.

Propagation of Errors - The total error associated with a single UNH batch(es) is computed by the same equation given for the L9 except that the random and fixed variations associated with volume (Q) are substituted for net weight (W). Using the data of Table 5 (divided by two), we obtain the following for uranium:

$$\begin{aligned} \% \text{ TOTAL ERROR (0.95CL)} &= 2 \left\{ \frac{[\%S_r(C)]^2}{mn} + \frac{[\%S_r(D)]^2}{pn} + \frac{[\%S_r(Q)]^2}{n} \right. \\ &\quad \left. + \frac{[\%S_s(C)]^2}{k} + \frac{[\%S_s(D)]^2}{k} + \frac{[\%S_s(Q)]^2}{j} \right\}^{1/2} \\ &= 2 \left[\frac{(1.05)^2}{(1)(1)} + \frac{(0.27)^2}{(1)(1)} + \frac{(0.65)^2}{(1)} \right. \\ &\quad \left. + \frac{(0.21)^2}{1} + \frac{(0.015)^2}{1} + \frac{(0.57)^2}{3} \right]^{1/2} \\ &= 2 \quad (1.5979 + 0.1526)^{1/2} \\ &= 2 \quad (1.32) \\ &= 2.6 \end{aligned}$$

where j = the number of times the tank was calibrated.

F15 Salt Waste Measurement

Three audits of the F15 measurement are made: Instrument Calibration, Sampling Procedure, and Analytical Control. Again the instrument calibration audit is of little value from a statistical estimating viewpoint. Brief discussions of the three audits are given below followed by a section on tank

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calibration. Variance estimates are summarized in Table 6.

Instrument Calibration Check - The Weight Factor Recorder and the Specific Gravity Recorder should be checked once a week. The instruments should be read and recorded to the nearest readable scale division before any corrective adjustments are made. Calibration of the Specific Gravity Recorder should be reviewed to be sure Specific Gravity is comparable to laboratory Specific Gravity ($25^{\circ}\text{C}/4^{\circ}\text{C}$). If a standard manometer (2.94 oil) is used to check calibration of these instruments, the temperature of the manometer fluid should be recorded. Since no data were available to estimate instrument variations, the variations given in Reference 3 were used in Table 6 for the Specific Gravity Recorder. Variations associated with the Weight Factor Recorder were estimated from tank calibration data and are discussed below.

Sampling and Analytical Procedure Audit - As mentioned before, three analyses of the duplicate samples are needed to obtain estimates of sampling variation. Data from fourteen duplicate samples were used to obtain approximate estimates of sampling variation (TABLE 6). Analytical variation error (long term referee results) was assumed to remain constant. Actually, there were no uranium referee data available so the analytical variation estimates given in Reference 3 were used for the uranium analysis. Both Referee and Standards data were available to estimate random variation and fixed variation for the Pu analysis.

The duplicate sample data were not used to estimate fixed variation due to sampling, mainly because it is not clear that the average difference between the first and second samples is truly a measure of sampling bias. The Pu analysis data indicates an average difference of 31 percent between the first and second samples (second sample high). However, the U analysis data indicates

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no significant difference between the first and second samples. Although inconclusive, these data may indicate longer agitation and/or circulation times are needed.

Tank Calibration - The F-15 tank was calibrated during February 1965 by the weighed-water increment method. A standard manometer (2.94 red oil) was connected in parallel during the calibration, but normally, the Weight Factor-Recorder (0 to 150 scale) is the only liquid level instrument actually in service. The existing calibration table (revised March 1965) is based on the standard manometer data and a manometer fluid calibration temperature of 78°F. adjusted to a transmitter range of 150 inches.

A straight line model having cumulative errors was fit to the standard manometer data and the WF-Recorder data. The standard manometer indicates three slope regions in the vessel; however, the WF-Recorder data were not sensitive enough to detect the differences in slope. The data are summarized in the following table:

F15 TANK CALIBRATION SUMMARY				
<u>Manometer Interval</u>	<u>Intercept</u>	<u>Slope</u>	<u>Manometer Variance (Random)</u>	<u>Slope Variance (Fixed)</u>
<u>Standard Manometer at 68°F (February 1965)</u>				
0.75-2.84	45.253	50.586		
2.84-26.10	66.140	43.227		
26.10-104.98	-53.347	47.806	39.895	0.3827
<u>WF-Recorder at 78°F (February 1965)</u>				
0-69	83.042	71.527	435.173	6.3068

The three curves are compared below for a WF-Recorder transfer from 45 to 0 inches and a recorder specific gravity of 1.3.

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COMPARISON OF F-15 CALIBRATION CURVES

	Existing Calibration Table	Cumulative Error Model Standard Manometer	0-150" WF-Recorder
Initial Man. Rdg.	45.0	67.5	45.0
Final Man. Rdg.	0	0	0
Temperature Corr.	45.0	67.2	45.0
+ by Sp. Gr.	34.6	51.7	34.6
Gallons Transferred	2359	2373	2475
% Difference	0	+0.6	+4.9
% Fixed Error (0.95CR)	?	2.7	7.0
% Random Error (0.95CR)	?	3.8	9.9
% Total Error (0.95CL)	?	4.7	12.1

The fixed variation, random variation, and total variation were computed as follows for the standard manometer:

$$\% \text{ FE}(0.95\text{CL}) = 2[(51.7)^2(0.3827)]^{1/2} (100)/2373 = 2.7$$

$$\% \text{ RE}(0.95\text{CL}) = 2[(51.7)(39.895)]^{1/2} (100)/2373 = 3.8$$

$$\% \text{ TE}(0.95 \text{ CL}) = 2[(51.7)^2(0.3827) + (51.7)(39.895)]^{1/2}(100)/2373 = 4.7$$

and for the WF-Recorder:

$$\% \text{ FE}(0.95\text{CL}) = 2[(34.6)^2(6.3068)]^{1/2} (100)/2475 = 7.0$$

$$\% \text{ RE}(0.95\text{CL}) = 2[(34.6)(435.173)]^{1/2} (100)/2475 = 9.9$$

$$\% \text{ TE}(0.95\text{CL}) = 2[(34.6)^2(6.3068)+(34.6)(435.173)]^{1/2}(100)/2475 = 12.1$$

Since the WF-Recorder is actually in service and because there is no reason to believe the WF-Recorder is any better today than it was at the time of calibration, the WF-Recorder cumulative error model is considered

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most representative for purposes of estimating fixed and random errors. In fact, it may be that actual volume estimates should be based on the WF-Recorder calibration curve because of the apparent five percent bias that existed at the time of calibration (February 1965).

Analysis of K-6 and F-15 calibration data indicates there may be some misunderstanding associated with the intended use of a standard manometer during calibration. The purpose of a standard manometer is to make sure that the service manometer (or recorder) is operating properly during the long calibration operation. The service manometer (or recorder) is the one we are interested in "calibrating." In the case of the K-6, it makes no difference whether the calibration curve was based on the standard manometer or the service manometer because the two manometers were essentially identical. However, it is apparent that the F15 WF-Recorder is approximately a factor of 2.6 times less accurate than a standard manometer (2.94 oil), and it would be presumptive to assume that we can improve the accuracy by fitting a line to the standard manometer data when the standard manometer is to be removed from service.

Propagation of Errors - The total error associated with a single F15 batch is computed in the same manner as before. Using the data from Table 6 (divided by two) and assuming an average batch, we obtain the following for uranium:

$$\begin{aligned} \% \text{ Total Error (0.95CL)} &= 2 \left\{ \frac{(46.5)^2}{(1)(1)} + \frac{(0.75)^2}{(1)(1)} + \frac{(4.95)^2}{1} \right. \\ &\quad \left. + \frac{(6)^2}{1} + \frac{(0.75)^2}{1} + \frac{(3.5)^2}{1} \right\}^{1/2} \\ &= 2 (2187.3 + 48.8)^{1/2} \\ &= 2 (47.3) \\ &= 94.6 \end{aligned}$$

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and for plutonium:

$$\begin{aligned} \% \text{ Total Error (0.95CL)} &= 2 \left\{ \frac{(44)^2}{(1)(1)} + \frac{(0.75)^2}{(1)(1)} + \frac{(4.95)^2}{1} + \frac{(0.4)^2}{1} \right. \\ &\quad \left. + \frac{(4.5)^2}{1} + \frac{(0.75)^2}{1} + \frac{(3.5)^2}{1} + \frac{(0.8)^2}{1} \right\}^{1/2} \\ &= 2 (1961.2 + 33.7)^{1/2} \\ &= 2 (44.7) \\ &= 89.4 \end{aligned}$$

D5 Dissolved Feed to Process

The only usable data obtained from the form was sampling audit data from the U analysis. This is discussed below. Although the vessel is currently being calibrated, the original meter pump calibration data were processed to provide a basis for comparison of the calibration methods. The estimates obtained are summarized in Table 7, but variation associated with batch content was not determined because of insufficient data.

Sampling Procedure Audit - Twelve batches were sampled in duplicate. No Referee data were available to allow separation of sampling and analytical variation. Based on this small sample of twelve data, the random error standard deviation associated with analytical and sampling combined was 5.7 percent or approximately ± 11.4 percent at the 0.95 confidence level. This pertains to U content only.

Tank Calibration - The D5 tank was calibrated four times during April 1955 using the meter pump method. A No. 3 oil manometer and a Weight-Factor Recorder (1.95 scale) were in service and were calibrated at that time. No temperature data were available. Thus, it was assumed that the temperature

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of the water passing through the meter pump was 15°C. The gallons of water measured by the meter pump were converted to gallons of water at 4°C. The WF-Recorder was assumed to be calibrated in terms of inches of No. 3 oil at 25° adjusted to a 1.95 scale. The resulting comparison of calibration curves is shown in the following table:

SUMMARY OF D5 TANK CALIBRATION DATA (April 1955)

<u>Instrument</u> <u>Range (in.)</u>	<u>Intercept</u>	<u>Slope</u>	<u>Manometer</u> <u>Variance</u> <u>(Random)</u>	<u>Slope</u> <u>Variance</u> <u>(Fixed)</u>
<u>WF-Recorder</u>				
1. 0.1-58.4	47.075	88.758	499.851	8.574
2. 1.0-58.0	68.645	89.017	1138.950	19.981
3. 1.2-56.0	46.147	92.889	80.553	1.471
4. 0.3-56.0	14.770	93.310	286.113	5.137
Average	44.16	90.993	501.366	8.791
<u>Standard Manometer</u>				
1. 0.1-58.4	51.173	47.780	44.148	0.4076
2. 2.2-108.1	52.254	47.913	52.457	0.4953
3. 0.1-109.1	58.088	47.569	161.444	1.4811
4. 0.2-109.0	33.2088	47.770	424.381	3.9005
Average	48.68	47.758	170.607	1.5711

Although there is indication of a slope change at about 27 inches (std. man), only one line was fitted to the complete range because there were insufficient data to estimate the slope below 27 inches. The existing calibration table (Revised July 1964) does include a slope

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change. The three curves are compared below:

COMPARISON OF D5 CALIBRATION CURVES

	<u>Existing Table</u>	<u>Cumulative Error Model</u>	
		<u>Standard Manometer</u>	<u>WF-Recorder</u>
Initial Reading	150	150	76.9
Final Reading	6	6	3.1
+ by Sp. Gr. (1.6)	93.75	93.75	48.1
+ by Sp. Gr. (1.6)	3.75	3.75	1.9
Difference	90.0	90.0	46.2
Gallons Transferred	4327	4298	4204
Percent Difference	-0-	0.67	2.84
% Fixed Error (0.95CL)	?	5.2 (5.6)	6.5 (6.8)
% Random Error (0.95CL)	?	5.8	7.2
% Total Error (0.95CL)	?	7.8	9.7

When the WF-Recorder average slope estimate is divided by 1.95 (90.993/1.95 = 46.663) and compared to the standard manometer average slope estimate (47.758), a 2.3 percent bias between the two instruments is evident. This indicates one of the instruments was not properly adjusted.

The fixed error estimates do not allow for the fact that the meter pumps could be biased. Based on vendor statements, it is assumed the accuracy of a meter pump is 2 percent (0.95CL). Thus, the fixed error statements in above table should be increased to 5.6 and 6.8 percent, respectively.

The fixed error, random error, and total error shown in the above table were computed as follows for the standard manometer:

$$\% \text{ FE}(0.95\text{CL}) = 2[(90)^2(1.5711)]^{1/2} (100)/4298 = 5.2$$

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$$\% \text{ RE}(0.95\text{CL}) = 2[(90)(170.607)]^{1/2} (100)/4298 = 5.8$$

$$\% \text{ TE}(0.95\text{CL}) = 2[(90)^2(1.5711) + (90)(170.607)]^{1/2} (100)/4298 = 7.8$$

and for the WF-Recorder:

$$\% \text{ RE}(0.95\text{CL}) = 2[(46.2)^2(8.791)]^{1/2} (100)/4204 = 6.5$$

$$\% \text{ RE}(0.95\text{CL}) = 2[(46.2)(501.366)]^{1/2} (100)/4204 = 7.2$$

$$\% \text{ TE}(0.95\text{CL}) = 2[(46.2)^2(8.791) + (46.2)(501.366)]^{1/2} (100)/4204 = 9.7$$

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TABLE 1

PR SCALE TARE WEIGHT CHECK DATA

<u>Date</u>	<u>Accounting</u>	<u>Independent</u>	<u>Difference</u>	<u>Range</u>
10/8	10.055	10.000	0.055	.055
10/16	-	9.995	-	
10/29	10.000	10.020	-0.020	.020
11/2	-	10.000	-	
2/26	10.000	10.030	-0.030	.030
2/26	10.030	9.970	0.060	.060
3/4	9.975	10.010	-0.035	.035
3/9	10.005	10.000	0.005	.005
3/10	10.010	10.030	-0.020	.020
3/18	10.000	9.990	0.010	.010
3/26	9.980	10.005	-0.025	.025
4/7	10.000	10.005	-0.005	.005
4/8	9.980	9.990	-0.010	.010
4/15	9.975	10.021	-0.046	.046
4/23	10.010	10.010	-0-	-0-
4/29	10.000	9.970	0.030	0.030
5/12	10.000	10.000	-0-	-0-
5/12	10.000	10.000	-0-	-0-
5/19	10.000	10.005	-0.005	0.005
6/16	10.000	10.010	-0.010	0.010

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TABLE 2
L9 Pu LOADOUT

Measurement	Nominal Value	0.95 Confidence Level			Notes
		Random	Fixed	Total	
<u>Pu, X-Ray</u>					
Analytical		0.024 1.2%	0.014 0.7%	0.028 1.4%	1
Sampling		0.008 0.44%	? ?	0.008 0.44%	2
Total		0.026 1.28%	0.014 0.7%	0.030 1.46%	
<u>AT, PuA-6b</u>					
Analytical		0.202 2.7%	? ?	? ?	1
Sampling		0.210 2.8%	? ?	? ?	2
Total		0.292 3.9%	? ?	? ?	
<u>Specific Gravity</u>					
Analytical		0.0044 0.3%	0.0010 0.1%	0.0047 0.32%	1
Sampling		0.0066 0.44%	? ?	0.0066 0.44%	2
Total		0.0080 0.52%	0.0010 0.1%	0.0081 0.54%	
<u>Net Weight</u>		0.056 0.54%	0.028 0.26%	0.062 0.6%	3
<hr/>					
Pu, batch		123 1.4%	66 0.75%	140 1.6%	

Calculation:

$$\text{Pu, grams} = \frac{(\text{Pu, lb/gal})(\text{Net Weight, kg})(119.83 \text{ g-gal/lb-liter})}{(\text{Sp.Gr., kg/liter})}$$

- (1) Fixed error taken from HW-76361. Up-to-date estimates could be obtained from Standards Program data.
- (2) No data available to estimate sampling fixed error.
- (3) Estimates based on Tare Weight Check data.

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TABLE 3

K6 TANK CALIBRATION SUMMARY

Manometer Interval, Inches	Intercept	Slope	Manometer Variance (Random)	Slope Variance Fixed)
<u>Standard Manometer - July 1963</u>				
1.04 - 5.40	41.7593	47.7646	17.1804	0.1679
5.40 - 26.94	64.0206	43.6408		
26.94 - 103.36	-71.9445	48.6875		
<u>Standard Manometer - February 1964</u>				
1.49 - 5.56	42.4955	47.0607	1.9651	0.01916
5.56 - 26.63	62.3789	43.4875		
26.63 - 104.04	-65.7570	48.2991		
<u>Standard Manometer - January 1965</u>				
1.39 - 5.75	41.5562	47.1887	7.7626	0.07530
5.75 - 27.43	64.0095	43.2866		
27.43 - 104.47	-67.8984	48.0953		
<u>Standard Manometer - Best Average of Three Calibrations</u>				
0 - 6	41.937	47.338	8.9693	0.0871
6 - 27	63.469	43.472		
27 - 104	-68.533	48.361		
<u>Service Manometer - July 1963</u>				
0.99 - 5.35	44.1248	47.7646	3.1550	0.03083
5.35 - 25.75	66.6282	43.5573		
25.75 - 103.31	-64.1440	48.6353		
<u>Service Manometer - February 1964</u>				
1.59 - 8.94	40.0941	45.6298	12.5483	0.1224
8.94 - 26.73	59.8543	43.4202		
26.73 - 104.14	-70.5564	48.2991		
<u>Service Manometer - January 1965</u>				
1.19 - 6.86	52.9408	45.4181	6.7497	0.0650
6.86 - 27.43	68.6521	43.1266		
27.43 - 105.10	-68.0616	47.8067		
<u>Service Manometer - Best Average of Three Calibrations</u>				
0 - 7	45.720	46.271	7.4843	0.07266
7 - 27	65.045	43.368		
27 - 104	-67.587	48.247		

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TABLE 4

COMPARISON OF K6 CALIBRATION CURVES

	<u>Existing Calibration Table</u>	<u>Cumulative Error Model</u>	<u>Standard Manometer</u>	<u>Service Manometer</u>
Initial mano rdg.	155.0 (86.0°F)	155.0 (86.0°F)	155.0 (86.0°F)	155.0 (86.0°F)
Final mano rdg.	23.0 "	23.0 "	23.0 "	23.0 "
Temperature corr.	155.0 "	153.77 (68°F)	153.77 (68°F)	153.77 (68°F)
Temperature corr.	23.0 "	22.82 "	22.82 "	22.82 "
+ by Sp. Gr.	93.94	93.19	93.19	93.19
+ by Sp. Gr.	13.94	13.83	13.83	13.83
Mano. difference	80.0	79.36	79.36	79.36
Gallons transferred	3767	3773	3764	3764
% Difference		+0.16	-0.08	
% Fixed error (0.95 CL)	?	1.24	1.14	
% Random error (0.95 CL)	?	1.41	1.30	
% Total error (0.95CL)	?	1.88	1.73	

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TABLE 5

K6 UNH LOADOUT

<u>Measurement</u>	<u>Average Value</u>	<u>0.95 confidence level</u>			<u>Notes</u>
		<u>Random</u>	<u>Fixed</u>	<u>Total</u>	
Tank slope estimate	3764 gals (48 gal/in)	- -	43 gals 1.14 %	65 gals 1.73%	3
Manometer difference	3764 gals (155 to 23 in)	49 gals 1.30%	- -		
<u>Specific Gravity</u>					
Analytical	1.66	0.0060 0.36%	0.0005 0.03%	0.0061 0.37%	1
Sampling		0.0068 0.42%	? ?	0.0068 0.42%	2
Total		0.0090 0.54%	0.0005 0.03%	0.0090 0.54%	
<u>Uranium</u>					
Analytical	4.05 lb/gal	0.048 1.2%	0.017 0.42%	0.051 1.25%	1
Sampling		0.068 1.7%	? ?	0.068 1.7	2
Total		0.084 2.1%	0.017 0.42%	0.086 2.1%	
<hr/>					
Uranium, per batch	15,244 lb.	378 2.5%	117 0.8%	396 2.6%	5
<hr/>					
Calculation					
Uranium, lb = ($\frac{\text{Mano. diff., in.}}{\text{Sp.Gr.}}$) (Tank slope, gal/in)(U, lb/gal)					(4)

Notes:

- 1) Fixed error taken from HW-76361. Up-to-date estimates could be obtained from Standards Program data.
- 2) No data available to estimate sampling fixed error. Reference 3 indicates none detectable.
- 3) Estimates based on cumulative error model (see text).
- 4) This equation is not exact if the initial manometer reading occurs in a different slope region than the final manometer reading.
- 5) Fixed error due to tank slope estimate (1.14%) is reduced because tank was calibrated three times.

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TABLE 6

F15 SALT WASTE

Measurement	Nominal Value	Random	0.95 Confidence Level		Notes
			Fixed	Total	
<u>U Analysis</u>					
Analytical	6.6x10 ⁻³ lb/gal	4.0x10 ⁻³ 60%	0.8x10 ⁻³ 12%	4.08x10 ⁻³ 62%	1
Sampling		4.7x10 ⁻³ 71%	? ?	4.7x10 ⁻³ 71%	2
Total		6.1x10 ⁻³ 93%	0.8x10 ⁻³ 12%	6.3x10 ⁻³ 95%	
<u>Pu Analysis</u>					
Analytical	3.1x10 ⁸ c/m/gal	1.0x10 ⁸ 33%	0.2x10 ⁸ 9%	1.02x10 ⁸ 34%	
Sampling		2.5x10 ⁻⁸ 81%	? ?	2.5x10 ⁸ 81%	2
Total		2.7x10 ⁻⁸ 88%	0.2x10 ⁸ 9%	2.7x10 ⁸ 88%	
Sp.Gr. Recorder	1.3	0.02 1.5%	0.02 1.5%	0.027 2.1%	4
Weight Factor Recorder	2475 gals (45 to 0 in)	245 gals 9.9%	- -	245 gals 9.9%	3
Tank Slope Estimate	2475 gals (71.5 gal/in)	- -	173 gals 7.0%	173 gals 7.0%	3
ICF	1.24x10 ⁻¹¹ g/cpm	0.1x10 ⁻¹² 0.8%	0.2x10 ⁻¹² 1.6%	0.22x10 ⁻¹² 1.8%	5
<hr/>					
U, batch	16 lb	15.0 94.6%	2.2 14%	15.0 95.6%	
Pu, batch	9 gram	8.0 89%	1.2 12.9%	8.1 90%	

Calculations:

$$U, \text{ lb.} = (U, \text{ lb/gal}) \left(\frac{\text{WF diff., in.}}{\text{Sp.Gr.}} \right) (\text{Tank slope, gal/in})$$

$$Pu, g = (Pu, \text{ cpm/gal}) (\text{ICF, g/cpm}) \left(\frac{\text{WF diff., in.}}{\text{Sp.Gr.}} \right) (\text{Tank slope, gal/in.})$$

Notes:

1. No Referee or Standards data available - Estimates from HW-76361
2. No data available to estimate sampling fixed error.
3. Estimates based on cumulative error model.
4. No data available to estimate Sp.Gr. Recorder error. Estimates from HW-76361.
5. No data available. Estimates from HW-76361

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TABLE 7

D5 DISSOLVED FEED TO PROCESS

Measurement	Nominal Value	0.95 Confidence level		Notes
		Random	Fixed	
<u>U Analysis</u>				
Analytical	3.75 lb/gal	?	?	
Sampling		?	?	
Total		0.43 11.4%	?	1
Manometer difference	4298 gal (144 in)	248 gal 5.8%	- -	2
Tank slope estimate	4298 gal (47.7 gal/in)	- -	226 gal 5.2%	2
Meter pump bias		- -	86 gal 2.0%	3
Pu Analysis	?	?	?	
Specific Gravity	1.6	?	?	

- (1) Estimate based on small amount of data.
(2) Estimates based on cumulative error model.
(3) Estimate based on vendor statements.

DATE

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