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EVALUATION OF SAMPLING VARIABLES—
VESSEL C-102

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Photostat Price \$ 3.30

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SUMMARY

It will not be advantageous to collect more than two samples from the coarse-feed tank. The most precision for the money will be obtained by compositing aliquots from each of the two samples, and performing one analysis on the composite. Details are given in the tabulation below.

	<u>One Analysis on One Sample</u>	<u>Composited Sample One Analysis</u>	<u>One Analysis on each of two Samples</u>
	<u>CASE I</u>	<u>CASE II</u>	<u>CASE III</u>
Cost per Tank	\$13.50	\$15.00	\$27.00
Precision of Concentration Measurement	$\pm 3.32\%$	$\pm 2.71\%$	$\pm 2.36\%$
Precision of Tank Total Contents (1)	$\pm 5.50\%$	± 4.33	± 3.71
Precision of Tank Total Contents (2)	$\pm 6.0\%$	± 5.68	± 5.52

(1) Limit of uncertainty of liquid-level measurement assumed negligible.

(2) Limit of uncertainty of liquid-level measurement assumed 5 per cent.

Case III will provide control data for surveillance of "range limits" between samples. Case II provides good precision for the money. Case I yields adequate precision if liquid level uncertainty is high, and the second sample will always be in reserve in the event control charts indicate an outlier.

It is recommended that only one analysis be performed per tank. During the early stages of a run, two analyses could be performed (one each on two samples --Case III).

When adequate data have been collected for construction of control charts, either Case I or Case II - type operation can be adopted, depending upon the limit of uncertainty in liquid-level measurements.

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PURPOSE

In order to determine the optimum procedure for sampling the coarse-feed tank (C-102) of the NP and MTR process, a systematic series of tests has been conducted to measure the extent of solution stratification and effect of air-sparging as a means of vessel homogenization.

PROCEDURE

A batch size of approximately 400 liters of uranyl-nitrate solution made to simulate an actual MTR batch was prepared in vessel C-102 under controlled and regulated conditions.

After all the material had been added to the vessel, samples were pulled from two different nozzels A and B spaced at approximately 180°, from each of three different levels in the vessel. The levels, designated by 1, 2, 3, were near the bottom, near the middle section and near the top of the solution. Thus, each series comprised six samples. Series 1 was taken before sparging.

After this set of six samples was pulled, the air sparger was turned on and during a period of one hour, a set of six samples was taken at intervals of 15 minutes, from the same location in the vessel as in Series 1, constituting Series 2, 3, 4 and 5.

Fifty liters of water were then added to the vessel and the resulting solution air-sparged 15 minutes before Series 6 was taken.

After 15 minutes of additional sparging, Series 7 was pulled. The solution was then boiled down to approximately the original volume, cooled to 25°C and sample Series 8 was drawn.

Finally after allowing the solution to stand for 24 hours without agitation, Series 9 was drawn.

In addition to the 54 samples drawn in the nine series as described above, three series of duplicate samples were drawn by the regular sampler to check for sample bias. The first set was drawn after the first 5 sets of samples were pulled from C-102 in the homogeneity test and before 50 liters of water were added. These two samples are designated Bias 1A and Bias 1B, the sampler circulating for 5 minutes between samples.

The second set of samples from the regular sampler was drawn after the 50 liters of water had been added to C-102, and the resulting solution air sparged for 15 minutes. These samples are designated by Bias 2A and Bias 2B. The sampler was allowed to circulate for 10 minutes between samples.

After the solution was boiled down and cooled the final 2 samples were drawn, the circulating time between samples being 15 minutes.

Duplicate analytical determinations were made on each sample through Series 3 and a portion of Series 4 as shown by the data.

All of the data are given in Table I at the end of the Report.

RESULTS

A. URANIUM CONCENTRATION

Precision

As would be expected, the solution was not homogeneous before sparging. A considerable difference existed between the levels from which the samples were taken as well as a difference between samples taken from the same level. These differences were measured in terms of the estimated analytical and sampling error associated with the experiment.

Furthermore, the solution was not homogeneous after 15 minutes and 30 minutes of sparging. An examination of the average concentration at each sample level in the vessel indicates that the gradient was from bottom to top after 15 minutes sparging and from top to bottom after 30 minutes sparging thus indicating no consistent preferential gradient but merely a heterogeneity of solution.

For all the other conditions imposed upon the system, the difference between samples taken from the three different levels was completely explainable in terms of the estimated analytical and sampling errors. Accordingly the sparging for 45 minutes or longer will with reasonable certainty bring the solution to homogeneity. The addition of 50 liters of water accompanied with sparging for 15 minutes to 30 minutes, and boiling the solution to original volume and cooling had little if any effect on destroying this homogeneity.

Estimates for the analytical and sampling errors to be associated with concentrations of this magnitude were found to be .9% analytical and 1.3% sampling yielding 95% limits of uncertainty to associate with a single sample and a single determination on this sample of $\pm 3.3\%$.

Bias

A comparison of samples designated as 1A, 1B, 2A, 2B and 3A, 3B with the nominal concentration associated with the series from which they were drawn indicates that no bias was present in any of these pairs of values; that is, the difference between the average of the pairs and nominal value was not significant in view of the analytical and sampling errors associated with the determinations. The effect if any, of circulating 5, 10 or 15 minutes between samples could not be detected.

B. SPECIFIC GRAVITY

Precision

The analytical error associated with the specific gravity determinations for practical purposes was found to be negligible. In view of the estimated sampling error it was found that a significant difference existed in specific gravity of samples taken from different levels when no sparging was done, as would be expected. Similarly as in the uranium concentration it was also found that 15 minutes sparging was not sufficient to produce homogeneity. An anomaly occurred with respect to the Series 3 and 4 of specific gravity determinations. Series 3 representing data after 30 minutes sparging indicated that no significant difference between levels from which the samples were drawn existed; however, in Series 4 representing 45 minutes of sparging a significant difference between levels was indicated. In view of the fact that it is difficult to explain physically how the material could evidently be homogeneous and then by further sparging stratify itself, the reasonable inference to draw here seems to be to discount the specific gravity data of Series 4.

With regard to all the other conditions to which the solution was subjected, no real difference between sample levels was found to exist.

As stated before, the analytical error was found to be nil. The sampling error associated with the specific gravity determinations in this experiment was found to be .007; converted to 95% limits of uncertainty and expressed as a per cent of the mean this yields approximately .2%.

Bias

With regard to the calculated specific gravity based on the known contents of each solution the experimentally determined values were biased on the average - .0094 specific gravity units. This calculation was based upon the sample bias series 1A, 1B, 2A, 2B; and 3A, 3B.

It was interesting to note that without exception the average specific gravity of each series of observations was lower than the calculated value.

C. TOTAL SOLIDS

Precision

With regard to total solids only series 4, i.e., sparging for 45 minutes yielded anything of interest. Here as was found for specific gravity a significant difference between levels was found even though the series before and after indicated no real difference. Analysis of the data for the purpose of estimating 95% limits of uncertainty yielded $\pm 3\%$ to be associated with a single total solids determination.

Bias

No bias was evident in the total solids measurements.

ANALYSIS OF DATA

Five cases are illustrated in Table II, which represents various combinations of analyses and samples. Data on cost of laboratory effort were taken from a previous study. *

It is apparent that errors in measurement of concentration in a tank can easily become eclipsed by other sources of uncertainty, e.g., liquid-level measurement. The procedure of performing analysis on duplicate samples (case 3 in Table I) will serve as an outlier detection. This will be sufficiently important during the early stages of any processing run in order to establish the statistical behaviour of measurement and operating variables. As soon as this can be done with confidence, the procedure described under case 1 or case 5 in Table I will be adequate for surveillance. A reserve sample will always be available for checking when outliers are detected from control charts. As shown by the tabulation, case 5 provides median precision at near minimum cost.

After 45 minutes of sparging the tank contents are homogeneous as nearly as can be determined by the tank concentration measurement method (both sampling and analytical errors have been evaluated).

CONCLUSIONS AND RECOMMENDATIONS

No advantage is apparent in taking more than two samples from the coarse-feed tank. Collection of two samples is essentially no more effort than one sample, but collecting four samples would be twice as much effort as for two, as an extra sample "pig" must be manipulated.

* Vance, F. P., and Kramer, B. R., "Economic Evaluation of Input Measurement Modes," letter Va-30-54A-N, May 6, 1954.

CONCLUSIONS AND RECOMMENDATIONS (continued)

The most precision for the money is afforded by compositing aliquots from each of the two samples, and performing one analysis on the composite. This attenuates sampling error by the factor $\sqrt{2}$, and represents a greater gain than performance of duplicate analyses. This is due to the fact that failure of the sample to represent the average concentration of the tank is greater than failure of the analysis to yield the true concentration in the sample.

It is recommended that two samples be collected from each coarse-feed tank. Circulation of solution through the sample-collection line need not exceed five (5) minutes between samples. Start-up of any new or different processing run (e.g., commencement of MTR dissolution in September) should be step-wise as follows:

1. Perform duplicate analyses on each of two samples until sufficient data have been collected to establish the appropriate values for analytic and sampling errors.
2. Adopt either case 1 or case 5 (Table I). Case 5 appears to yield the most precision for the money, assuming the double-pipetting operation introduces negligible error. This can be evaluated from cumulative data.

TABLE I

<u>Sample</u>			<u>Specific Gravity</u> (Temp. at 25° unless specified)	<u>Uranium,</u> <u>g/l</u>	<u>Total Solids,</u> <u>g/l</u>
<u>Series</u>	<u>Level</u>	<u>Nozzle</u>			
1	1	A	1.3291 1.3290	3.006 2.986	95.8
1	1	B	1.3242 1.3244	2.939 2.940	96.5
1	2	A	1.2623 1.2625	2.362 2.401	77.5
1	2	B	1.2528 1.2529	2.296 2.273	75.6
1	3	A	1.1431 1.1428	1.328 1.312	41.8
1	3	B	1.1450 1.1451	1.344 1.323	42.5
2	1	A	1.2184 1.2183	1.940 1.963	65.7
2	1	B	1.2176 1.2172	1.935 2.000	65.2
2	2	A	1.2197 1.2199	1.969 1.972	65.3
2	2	B	1.2194 1.2190	1.970 1.990	65.6
2	3	A	1.2197 1.2197	1.980 2.000	65.9
2	3	B	1.2192 1.2193	2.004 1.975	65.8
3	1	A	1.2197 1.2195	2.032 1.967	66.0

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TABLE I
(continued)

<u>Sample</u>			<u>Specific Gravity</u> (Temp. at 25° unless specified)	<u>Uranium</u>	<u>Total Solids,</u>
<u>Series</u>	<u>Level</u>	<u>Nozzle</u>		<u>g/l</u>	<u>g/l</u>
3	2	A	1.2194 1.2194	1.985 1.976	65.9
3	1	B	1.2188 1.2186	1.974 1.984	66.1
3	2	B	1.2193 1.2192	1.981 1.969	66.1
3	3	A	1.2192 1.2184	1.980 1.991	65.9
3	3	B	1.2193 1.2190	1.993 1.943	65.6
4	1	A	1.2190 1.2190	2.001 2.022	65.9
4	1	B	1.2196 1.2196	1.973 2.013	66.0
4	2	A	1.2236	2.001	65.8
4	2	B	1.2218	2.004	65.8
4	3	A	1.2237	1.982	66.1
4	3	B	1.2225	1.968	66.2
5	1	A	1.2214	1.974	65.6
5	1	B	1.2214	1.975	65.7
5	2	A	1.2211	1.965	65.8
5	2	B	1.2215	1.976	65.6
5	3	A	1.7203	2.006	65.9
5	3	B	1.2227	2.115	65.2
6	1	A	1.1974	1.804	59.4

TABLE I
(continued)

<u>Sample</u>			Specific Gravity (Temp. at 25° unless specified)	Uranium, <u>g/l</u>	Total Solids, <u>g/l</u>
<u>Series</u>	<u>Level</u>	<u>Nozzle</u>			
6	1	B	1.1962	1.747	58.6
6	2	A	1.1969	1.776	58.9
6	2	B	1.1961	1.735	58.5
6	3	A	1.1957	1.763	58.5
6	3	B	1.1961	1.754	58.4
7	1	A	1.1958	1.779	58.4
7	1	B	1.1979	1.742	58.3
7	2	A	1.1957	1.729	58.6
7	2	B	1.1970	1.762	58.8
7	3	A	1.1952	1.743	58.6
7	3	B	1.1999	1.781	58.9
8	1	A	1.2217	2.011	66.8
8	1	B	1.2243	2.028	67.2
8	2	A	1.2228	2.066	66.9
8	2	B	1.2240	2.013	67.5
8	3	A	1.2223	2.046	66.7
8	3	B	1.2232	2.033	66.8
9	1	A	1.2229 022.5	2.030	66.8
			1.2229 022.4		
9	1	B	1.2234 022.6	1.993	66.6
			1.2230 022.2		
9	2	A	1.2228 022.1	1.958	66.6
			1.2229 022.0		
9	2	B	1.2223 022.8	2.030	66.6
			1.2225 022.7		
9	3	B	1.2228 022.3	2.032	66.6
			1.2223 022.2		
9	3	A	1.2228 022.5	2.021	66.8
			1.2229 022.5		

TABLE I
(continued)

Sample			Specific Gravity (Temp. at 25° unless specified)	Uranium	Total Solids
Series	Level	Nozzle		g/l	g/l
Bias	1A		1.2210	2.023	65.9
Bias	1B		1.2216	1.994	65.9
Bias	2A		1.1963	1.807	58.5
Bias	2B		1.1953	1.794	58.5
Bias	3A		1.2231	2.047	66.5
Bias	3B		1.2164	2.042	66.4

TABLE II

Case	No. Samples	No. Analyses per Sample	Total Analyses	Laboratory Cost (a)	Precision, Per Cent		
					Concentration	Tank (d)	Content(c) (e)
1	1	1	1	\$13.50	± 3.32	± 5.50	± 6.0
2	1	2	2	27.00	± 3.04	± 4.93	± 5.86
3	2	1	2	27.00	± 2.36	± 3.71	± 5.52
4	2	2	4	54.00	± 2.15	± 3.46	± 5.45
5	1 (b)	1	1	15.00	± 2.71	± 4.33	± 5.68

(a) Based upon \$7,000/MY and 4 MH/ Spike analysis.

(b) Aliquot drawn from each of two samples and composited:

(c) Limits of uncertainty in Specific Gravity Measurements taken as ± 2.5 per cent, based on analysis of data collected during recent processing of the EBR core.

(d) Limit of uncertainty in liquid level taken as zero.

(e) Limit of uncertainty in liquid level taken as ± 5 per cent.