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UNION CARBIDE NUCLEAR COMPANY • POST OFFICE BOX P. OAK RIDGE, TENNESSEE

To (Name) Mr. E. C. Bollinger
Mr. J. A. Parsons
Company
Location

Date January 20, 1960
Originating Dept. Production Engineering
Answering letter date

Copy to Mr. A. L. Allen
Mr. J. W. Arendt
Mr. J. Dykstra
Mr. A. P. Huber
Mr. W. D. McCluen
Mr. M. F. Schwenn
Mr. R. D. Shaffer ✓
Mr. S. S. Stief
Mr. A. Varlan
Production Division File

Subject Test Run of Savannah River UNH

K-25 RC KP-1815
NOT TO BE LOANED FROM
PLANT RECORDS K-1034

REC-2-34708
FILE
X-REF.
X-REF.

Union Carbide Nuclear Company, Oak Ridge Gaseous Diffusion Plant, Operating Contract for the U.S. Atomic Energy Commission.

As outlined in the minutes of the November 6 meeting of the UCNC Salvage Panel, the processing of three tank trailers of highly enriched (~85% U-235) Savannah River reactor tails UNH to oxide has been completed at ORGDP. Particular emphasis was placed on health physics during the tests to determine the scope of this problem at K-1420 while operating with highly enriched material. In this regard, a complete program of health physics measurements was conducted during the processing of the three shipments of UNH. A secondary objective was to substantiate, through practical application, the uranium accountability procedure as related to shipper-receiver data for dilute, highly enriched uranium solutions.

The following comments present a summary of pertinent facts regarding the test

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1. Three tank trailer shipments (3800 gallons of UNH containing approximately 70 kilograms of uranium each) were received at ORGDP, as scheduled; shipment No. 75 on November 13, shipment No. 76 on November 17, and shipment No. 77 on November 19, 1959.
2. Since no information was available concerning the homogeneity of the solution on an "as received" basis, a pumping facility was set up to agitate or mix the contents of the first shipment (No. 75) by recycling at the rate of 1800 gallons per hour. One

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liter samples were taken at the bottom, middle, and top of the tank before agitation and at 30-minute intervals during agitation for a period of 2-1/2 hours. The results of this experimental sampling established that the solution was homogenous in the "as received" state.

3. A comparison of the shipping and receiving uranium accountability data is shown in table 6. It will be noted that a significant weight difference appeared for shipment No. 76. This resulted from the gross receiving weight being higher than that at which the tank trailer was shipped. A subsequent scale check with a standard weight, however, indicated the scale to be performing within its warranted accuracy.
4. During processing, the tank trailer served as a feed storage tank, that is, UNH was pumped directly from the tank to process equipment at a rate compatible with the process rate, thus eliminating the need for intermediate storage facilities.
5. Processing steps included the concentration of the dilute UNH by continuous evaporators, and the drum drying and calcining of the evaporator concentrate to produce uranium oxide. No unusual operating difficulties were experienced during the processing of this material.
6. With the exception of the first tank trailer, each shipment was emptied, inspected, weighed-out, and ready for release to the common carrier within forty-two hours after receipt of shipment at ORGDP.
7. As previously stated, a detailed program of health physics measurements was conducted during the processing of each shipment. In addition to five continuous air monitors, which were set up at appropriate working locations, numerous short-term spot samples were taken near specific pieces of operating equipment. Alpha and beta-gamma surveys of the operating area and process equipment were also completed after each shipment was processed. Significant findings of this survey are summarized:
 - A. Shift length air samples showed that the air activity in the immediate processing area was greater than the accepted ORGD plant allowable limit for operation without respiratory protection. As suspected, it was determined that the drum dryer-calciner units provided the major source of air contamination. Since this condition also exists to a lesser degree during the processing of uranium of lower enrichment, steps have been taken to eliminate this source of air activity. An estimate of \$10,000 has been developed as the cost for design and installation of an adequate dry-box and ventilation system for these units.

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Mr. J. A. Parsons

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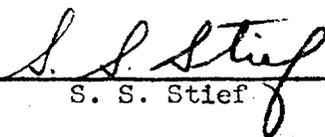
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- B. A steady increase was also noted in the surface alpha contamination of the working area. However, with the elimination of excessive air activity, the spread of alpha contamination should not remain a serious problem.
- C. Although of relatively low intensity, there was a steady build-up of gamma radiation in selected portions of the evaporators and drum dryers; the radionuclides responsible for this build-up were determined to be ruthenium-106, niobium-95, and zirconium-95.
- D. Personnel protection was adequate throughout the test run as evidenced by the fact urinalyses obtained on the employees involved showed no significant uptake of uranium.

Attached are more detailed accounts of the test run, emphasizing the sampling procedures, accountability data, and the detailed analysis of the health physics measurements.

In summary, highly enriched uranium materials can be processed in the existing K-1420 safe-geometry facilities, providing respiratory protection is provided. Since this is normal practice in similar operations at ORGDP, this represents no departure from present plant procedure. Continued operation on Savannah River material would make the enclosing of the drum dryer-calciner units and providing separate and more adequate ventilation very desirable.


A. Varlan


S. S. Stief

AV/SSS: jc

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ACCOUNTABILITY PROCEDURES USED FOR PROCESSING SAVANNAH RIVER
URANYL NITRATE SOLUTION - SHIPMENT No's. 75, 76, and 77

On Friday, November 13, 1959, the ORGDP received the first tank trailer (shipment No. 75) of uranium bearing materials from Savannah River. This material consisted of 3,800 gallons of uranyl nitrate (UNH) solution. Shipper analysis indicated the solution to contain 4.91 grams uranium/liter (4.91 gU/l), with an assay of 85.66% uranium-235. Two other tank trailer shipments (No. 76 and No. 77) were received on November 17, 1959 and November 19, 1959, with vendor analyses of 4.94 grams uranium/liter and assays of 85.84% uranium-235, respectively. Since the actual processing operations for each tank trailer were identical, the procedures contained herein outline the methods used for accountability in the processing of the material contained in shipment No. 75 at the Oak Ridge Gaseous Diffusion Plant. Table 1 presents the time-table of operations used in the processing of this tank trailer.

TABLE 1
TANK TRAILER PROCESSING OPERATIONS TIME-TABLE

<u>Date</u>	<u>Time</u>	<u>Operations</u>
11-13-59	1800	Tank trailer arrived at ORGDP.
	1810	Inspection by Materials Handling.
	1825	Weighed-in by the Receiving Department.
	1835	Arrived at K-1420.
	1840	Positioned at K-1420.
	1845	Radiation Survey by the Health Physics Department.
	1855	First group of samples taken.
	1955	Recycling operations started.
	2025	Second group of samples taken.
	2055	Third group of samples taken.
	2125	Fourth group of samples taken.
	2155	Fifth group of samples taken.
	2225	Sixth group of samples taken.
	2235	Processing through the evaporators began.
2315	First condensate sample.	
11-14-59	0830	C-Calcliner started.
11-15-59	1230	Completed processing of the tank trailer material.
11-17-59	1745	Tank trailer released to carrier.

Total time the trailer spent at ORGDP was 95.75 hours. Of this time, process preparations (weighing, sampling, etc.) took 4.5 hours, and the processing time through the evaporators, drum dryer, and calciners was 36.5 hours.

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1. Inspection of the Tank Trailer

When the tank trailer arrived at the ORGDP north portal, it was visually inspected by Materials Handling for all foreign matter such as ice, snow, and mud adhering to the trailer. This inspection revealed no excessive deposits of foreign matter, therefore, no cleaning (steam) was necessary. After the inspection was completed, the tank trailer was escorted by a security guard to K-1215 for weighing.

2. Weighing-in of the Tank Trailer

The K-1215 truck weight scales have been designated as the official scales to be used in weighing of the tank trailer. These scales are graduated in ten pound (10 lb.) increments and have a capacity of 30 tons. These scales are periodically checked by the State of Tennessee Department of Agriculture. Arrangements for weighing the tank trailer and the weighing operation were supervised by Materials Handling in conjunction with the Receiving Department. The tank trailer was positioned in the approximate center of the scales platform. Before the trailer was weighed, the tractor was uncoupled and removed from the scales platform. The weighing operator then weighed the truck, using the scales print-weigh attachment. The gross weight of the tank trailer was 45,850 pounds. This indicated that the ORGDP gross weight was twenty (20) pounds heavier than the Savannah River gross weight of 45,830 pounds. The weighing operator then completed and signed the print-weigh weigh-in form and presented it to Materials Handling. After the weighing operation was completed, the tank trailer was escorted to K-1420 by a security guard.

3. Radiation Survey of the Tank Trailer

Upon arrival at K-1420, the tank trailer was positioned in an area designated for this purpose on the north side of the building. This area had painted boundary lines and was free of uranium-bearing materials. After positioning, Materials Handling released the tank trailer to Chemical Operations for further processing. The Health Physics Department then made a radiation survey of the entire trailer. The complete results of all radiation surveys made are found in a letter issued by the Safety and Health Physics Department (attached).

4. Sampling of the Tank Trailer

A sampling test was accomplished on the material of tank trailer (shipment 75) in order to ascertain the degree of homogenization required for future tank trailer sampling. The test included sampling of the trailer, as received, and multiple sampling of the trailer after varying intervals of mixing. The mixing of the solution was accomplished by recycling the material from the dip-leg feed line from the trailer and returned to the open inspection port on the trailer through a nozzle equipped with an anti-splash cover. The solution was recycled by a pump capable of moving 1,800 gallons of solution per hour.

Before agitation by the recycling pump, the first group of samples was withdrawn. This sampling consisted of withdrawing three (3) one liter samples, and one (1) one-half liter sample. The three (3) one liter samples were taken in groups such that the liquid depth was represented. One sample was taken approximately 2 inches above the tank bottom, one sample at half depth, and one sample approximately 2 inches below the liquid surface. The one-half liter sample was withdrawn at half depth. The tank diameter is about 54 inches, and the liquid level in the tank was about 52 inches. When this phase of the sampling test was completed, the recycling pump was started. After the solution had been agitated by recycling for thirty (30) minutes, a second group of samples, identical in size, number, and sampling position of the first group, was withdrawn. This sampling operation was continued at thirty (30) minute intervals until two and one-half hours (2.5 hours) of agitation time had elapsed. The results of successive one-half liter samples (ppm U) indicated no significant differences, and the sampling was discontinued because equilibrium had been attained. A total of twenty-four (24) samples, eighteen (18) one liter samples and six (6) one-half liter samples was withdrawn. During the recirculation of the solution in the tank trailer, Health Physics Department took air samples at the tank manhole used to receive the solution from the recycle pump. All tank trailer samples were aspirator thief samples, withdrawn by Process Laboratory personnel through the open inspection port of the trailer.

A. One-Liter Samples

All one liter samples were transferred to the Works Laboratory where duplicate analyses for uranium by potentiometric titration to a precision of $\pm 0.5\%$, and duplicate analyses for uranium-235 by mass spectrometer to a precision of $\pm 0.2\%$ were obtained. One composite sample from all groups was analyzed spectrographically and for Np, Pu, and beta and gamma activity. Activity was to be reported with respect to aged normal uranium in equilibrium with its daughter products. Table 2 presents the data obtained from this sampling test.

B. Statistical Analysis of Test Samples

Statistical analysis of the sampling test data indicated the material was homogenous as received since mixing produced no significant change in the uranium analysis over the testing period. The sampling and analytical limits of error for the material was determined to be $\pm 0.25\%$ of the reported value as estimated by analysis of variance test on the data.

C. Process Sampling

All one-half liter samples were analyzed by the Process Laboratory for ppm uranium, specific gravity, and pH. Table 3 presents the results of all control samples taken from the tank trailer.

TABLE 2
ANALYTICAL RESULTS OF THE SAMPLING TEST

Group	Sample No.	Sampled Position	Analyses (gU/g)		Assay (% U-235)	
			1	2	1	2
I	394276	Bottom	.00484	.00483	85.49	85.52
	394277	Middle	.00483	.00484	85.54	*
	384278	Top	.00483	.00483	85.55	*
II	394279	Bottom	.00484	.00483	85.51	*
	394280	Middle	.00483	.00484	85.56	*
	394281	Top	.00484	.00484	85.55	*
III	394282	Bottom	.00482	.00482	85.50	85.44
	394283	Middle	.00483	.00483	85.53	85.54
	394284	Top	.00484	.00484	85.51	85.51
IV	394285	Bottom	.00483	.00482	85.50	85.46
	394286	Middle	.00483	.00484	85.55	*
	394287	Top	.00483	.00483	85.53	85.52
V	394288	Bottom	.00483	.00483	85.54	85.53
	394289	Middle	.00484	.00484	85.43	85.57
	394290	Top	.00484	.00483	85.50	85.51
VI	394291	Bottom	.00483	.00483	85.57	*
	394292	Middle	.00483	.00483	85.53	85.54
	394293	Top	.00483	.00482	85.56	*
Average			.004832		85.5272	

* Insufficient Sample

	ppm Np	ppb Pu	Total Spectrographic	β activity	Gamma Activity
Composite Sample	1.8	97	1000	9 X aged normal	129 x aged normal

TABLE 3
CONTROL SAMPLE RESULTS BY PROCESS LABORATORY

Time	Ppm U	Specific Gravity	pH
18:55	4,900	1.015	0.63
20:25	4,900	1.015	0.70
20:55	4,800	1.015	0.70
21:25	4,850	1.015	0.70
21:55	4,850	1.018	0.70
22:25	4,800	1.018	0.70
Averages	4,850	1.016	0.688

5. Process Sampling of the Tank Trailer Material

Sampling of the feed line to the process equipment was made by the continuous sample method already established at the ORGDP. Each feed line was sampled by a continuous sample integrated with the material feed rate. Each time the feed rate changed, the full or partially full sample container in use was removed and replaced with a clean, empty sample container. Logs of feed rates were maintained in order to apply appropriate sample results to quantities fed. The samples withdrawn from the feed lines were transferred to the Works Laboratory for analysis of uranium and uranium-235. Table 4 presents the data obtained from this sampling operation.

TABLE 4
ANALYTICAL RESULTS OF CONTINUOUS FEED SAMPLES

<u>Sample No.</u>	<u>Analysis, gU/g</u>	<u>Assay, % U-235*</u>
394264	.00484	85.5272
394265	.00487	85.5272
394266	.00488	85.5272
394267	.00471	85.5272

* Average of Sample Test.

The waste line from the primary evaporator and the waste lines from the three (3) post evaporators were sampled once each hour. A tolerance of one (1) ppm uranium had been established for this sample. When this tolerance was exceeded, the feed rates to the evaporators were reduced to meet this tolerance. A total of two (2) samples per hour, one from each line, was withdrawn and analyzed for ppm uranium by Process Laboratory personnel. Table 5 presents the results of this sampling operation.

TABLE 5
PROCESS LABORATORY SAMPLE RESULTS OF CONDENSATE

<u>Date</u>	<u>Time</u>	<u>Equipment</u>	<u>ppm U</u>	<u>Condensate Rate Gallons/Hr.</u>	<u>Discarded Gms. U</u>
11-13-59	23:15	B-4	5.	72	1.36
	23:15	B-27 (B,C)	2.	66	0.50
11-14-59	00:35	B-4	75.	66	18.74
		B-27	4.	66	1.00
	01:50	B-4	50.	70	13.25
		B-27	4.	70.	1.06
	02:15	B-4	10.	70.	2.65
		B-27	3.	70.	0.79
	03:15	B-4	30.	61.3	6.96
		B-27	3.	56.4	0.64
04:15		B-4	37.	55.	7.70
		B-27	1.	55.	0.21

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TABLE 5 (Continued)

<u>Date</u>	<u>Time</u>	<u>Equipment</u>	<u>ppm U</u>	<u>Condensate Rate Gallons/Hr.</u>	<u>Discarded Gms. U</u>
11-14-59	05:15	B-4	3.	52.2	0.59
		B-27	1.	39.6	0.15
	06:15	B-4	1.	43.	0.16
		B-27	2.	39.6	0.30
	07:15	B-4	2.	57.	0.43
		B-27	1.	39.6	0.15
	08:15	B-4	2.	51.	0.39
		B-27	2.	40.	0.30
	09:15	B-4	2.	80.	0.60
		B-27	2.	45.	0.34
	10:15	B-4	2.	45.	0.34
		B-27	2.	47.	0.35
	11:15	B-4	2.	43.	0.32
		B-27	1.	45.	0.17
	12:20	B-4	1.	37.	0.14
		B-27	3.	57.	0.65
	13:20	B-4	1.	37.	0.14
		B-27	3.	57.	0.65
	14:00	B-4	1.5	65.	0.38
		B-27	3.0	70.	0.80
	15:00	B-4	1.5	65.	0.88
		B-27	3.0	70.	0.80
	16:30	B-4	1.5	58.	0.33
		B-27	2.	50.	0.38
	17:10	B-4	1.5	54.	0.31
	17:15	B-27	2.	47.	0.36
	18:15	B-4	1.5	58.	0.33
		B-27	2.	41.	0.31
	19:10	B-4	2.	64.	0.48
	19:15	B-27	2.	37.	0.28
20:15	B-4	2.	80.	0.61	
	B-27	1.	38.	0.14	
21:15	B-4	2.	80.	0.61	
	B-27	1.	46.	0.17	
22:15	B-4	5.	76.	1.44	
	B-27	1.	38.	0.14	
23:15	B-4	1.	35.	0.13	
	B-27	0.5	61.	0.12	
11-15-59	00:15	B-4	3.	65.	0.74
		B-27	1.	40.	0.15
	01:15	B-4	2.	65.	0.49
		B-27	2.	40.	0.30
	02:15	B-4	3.	65.	0.74
		B-27	2.5	40.	0.38
	03:15	B-4	1.	63.	0.24
		B-27	2.	30.6	0.23
	04:15	B-4	1.5	63.	0.36
	B-27	2.5	35.	0.33	

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TABLE 5 (Continued)

<u>Date</u>	<u>Time</u>	<u>Equipment</u>	<u>ppm U</u>	<u>Condensate Rate Gallons/Hr.</u>	<u>Discarded Gms. U</u>
11-15-59	05:15	B-4	1.	65.	0.25
		B-27	2.	35.	0.26
	06:15	B-4	4.	68.2	1.03
		B-27	2.	39.6	0.30
	07:15	B-4	2.	72.	0.55
		B-27	2.	39.6	0.30
	08:30	B-4	2.	39.6	0.20
		B-27	2.	41.	0.31
	09:30	B-4	1.	45.	0.17
		B-27	1.	66.5	0.25
	10:30	B-4	1.	61.	0.23
		B-27	1.	41.	0.16
	11:30	B-4	1.	63.	0.24
		B-27	1.	41.	0.16
	12:30	B-4	1.	82.	0.31
		B-27	1.	47.	0.18
Totals			3.80*	104.1*	78.49

* Averages

<u>Summary:</u>	<u>B-4 Evaporator</u>	<u>B-27 Evaporator</u>	<u>Totals</u>
Total hours operation	36.5	36.5	36.5
Average Condensation Rate - Gallons/Hr.	58.3	46.1	104.1
Average Purity - ppm U	7.03	1.9	3.80
Total Discard - Grams U	64.42	14.07	78.49
Value of Material - \$11.00 Basis	\$648.99	\$141.75	\$790.74

The Safety and Health Physics groups took spot air samples adjacent to appropriate equipment during the processing of the UNH to oxide. Continuous air monitors were located in the processing area during the time of processing.

The Process Laboratory waste material accumulated from the analyzing of all process samples amounted to 81.67 liters for the three trailers processed. Based upon the sample analysis (1.46 grams uranium/liter and 68.09% uranium-235), this material contains 119 grams uranium and 81.19 grams uranium-235.

6. Feeding of the Tank Trailer

Feeding of the tank trailer to process equipment was dependent upon the equipment operating experience by Chemical Operations. A log was kept by Chemical Operations on the complete feeding operations of the tank trailer material.

After the feeding operations of the tank trailer had been completed, a radiation survey was made. This survey indicated that Health Physics standards had been met (less than 500 disintegrations per minute), therefore, it was not necessary to steam clean the tank trailer. After this survey by the Health Physics Department, the tank trailer was returned to K-1815 for a gross weighing-out. This transfer was made by Maintenance Division personnel. This weight was made in the same manner as prescribed for the receiving weighing operation.

After the weighing-out operations had been completed, the tank trailer was transferred back to K-1420 to await releasing to the Carrier. All tank trailer movements, except as stated above, were made by the transfer company (Rowe), pre-arranged by Materials Handling.

7. Oxide Processing

A log book was maintained by Chemical Operations for the drum-drying, calcining, and rod-milling of the oxide. Upon completion of the rod-milling phase of the process, the oxide was packaged, using individually-safe containers. All seven (7) containers of oxide packaged in the Chemical Operations building were distinctively tagged. These tags were pre-numbered and contained all pertinent information, except the weight of the container. The weight of each container was put on the tag by Materials Handling. Each container used in packaging oxide in the Chemical Operations building had previously been tare weighed and identified by Materials Handling. After packaging, the oxide was transferred to Materials Handling for weighing and sampling.

8. Oxide Weighing

Each container of oxide packaged by Chemical Operations was weighed by Materials Handling on a Toledo Bench Weight Scale, whose smallest dial graduation was one (1) gram. The maximum weight tolerance for this type container has already been established from statistical tests (1.4 grams/container) performed for this purpose.

9. Oxide Sampling and Analysis

Each container of oxide was sampled by Material Handling. Each sample consisted of approximately 100 grams of oxide, obtained by core sampling technique. These samples were transferred to the Works Laboratory for uranium and uranium-235 analyses. The Works Laboratory analytical procedure was potentiometric titration to a precision of $\pm 0.5\%$ for uranium and by optical spectrograph for uranium-235.

10. Summary

Shipper - Receiver Measurement Differences

Shipper - receiver measurement differences for the individual shipments are tabulated in table 6. It is noted that all measurements are in excellent agreement with the exception of the weights on shipment 76. The ORGDP scales were checked with standards subsequent to receiving this trailer and were found in calibration. The SRO analytical data for shipments 76 and 77 are corrected data from an original reported analysis of 5.06 gU/l.

TABLE 6

SHIPPER-RECEIVER DIFFERENCES FOR SRO-UHJ SOLUTIONS

<u>Plant</u>	<u>Net Weight (Lbs.)</u>	<u>Analysis (gU/l)</u>	<u>Assay (% U-235)</u>	<u>Uranium (Grams)</u>	<u>U-235 (Grams)</u>	<u>Specific Gravity</u>
<u>SHIPMENT 75</u>						
SRO	32,630	4.91	85.66	71,739	61,452	1.013
CCC	32,600	4.91	85.53	71,421	61,108	1.016
Difference*	30	0	0.13	318	344	-0.003
<u>SHIPMENT 76</u>						
SRO	31,960	4.94	85.84	70,625	60,624	1.014
CCC	32,580	4.94	85.76	71,821	61,594	1.017
Difference*	- 620	0	0.08	-1,196	- 970	-0.003
<u>SHIPMENT 77</u>						
SRO	32,720	4.94	85.84	72,305	62,067	1.014
CCC	32,670	4.95	85.79	72,019	61,800	1.018
Difference*	50	-0.01	0.05	286	267	-0.004
<u>SUMMARY</u>						
SRO	97,310	4.93	85.78	214,669	184,143	1.014
CCC	97,850	4.93	85.69	215,261	184,502	1.017
Difference*	- 540	.00**	0.09**	- 592	- 359	-0.003

* SRO minus CCC

** Average