HW-19545

Cover Sheet for a Hanford Historical Document Released for Public Availability

Released 1995

Prepared for the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

Pacific Northwest Laboratory
Operated for the U.S. Department of Energy
by Battelle Memorial Institute

Battelle

DISCLAIMER

HW-19545

DS:
1. O. P Amacker
2. F. W. Albaugh
   L. M. Knights
3. H. W. Murray
4. R. E. Roberts
5. B. F. Butler
6. D. F. Sheperd
7. A. H. Bushey -
   H. R. Schmidt
8. W. W. Mills
9. G. B. Barton
10. 300 File
11. 700 File
12. Pink
13. Yellow

November 22, 1950

COPY 1 OF 1

This Document consists of 3 Pages

O. P. Amacker
2704 E Building - 200 E Area

ANALYSIS OF A STANDARD PU SOLUTION
BY THE CONTROL LABORATORIES IN 231 AND 234-5 BUILDINGS

In accordance with your request another set of standard plutonium samples was submitted to the Control Laboratories in Buildings 231 and 234-5. These samples were from the same lot of purified material used for the previous sample submission, reported in a letter to you dated July 14, 1950.

Before the samples were submitted, the solution was re-standardized gravimetrically on October 31. This was accomplished by pipetting 500 ul. samples into platinum crucibles, and igniting the plutonium nitrate to PuO_2. It was observed during the weighing of the samples that a considerable amount of weight was regained. This introduces a slight uncertainty in the final result, but less than that observed later in the laboratory results.

The concentration of the solution calculated from three portions corresponded to 222.47, 222.50, and 223.92 g/L. The weight regain on the third of these three samples was less than on the other two, so the result is probably high. There is a total impurity content of approximately 700 p.p.m. in the plutonium. Correcting for this gives concentrations of 222.27, 222.29, and 223.72 g/L. The true value will be assumed to be 222.28 g/L.

The samples submitted to each laboratory consisted of ten individual peanuts, each containing approximately 100 ul. of the standard solution. The peanuts were filled in consecutive order and alternate ones selected for submission to the different laboratories. The results reported by

[redacted]

the two laboratories are tabulated below:

TABLE I

RESULTS OF ANALYSIS OF STANDARD PU SOLUTION
IN CONTROL LABORATORIES IN 231 AND 234-5 BUILDINGS

Peanut No.	234-5 Original g/L	234-5 Rerun g/L	Peanut No.	231 Original g/L	231 Rerun g/L		
1	215.94	212.74	2	217.13	218.25		
3	214.02		4	219.46	217.12		
5	212.25		6	216.61	221.92	221.47	
7	221.70		8	213.47	214.74		
9	219.15		10	213.19	205.67	204.61	200.91
11	220.40		12	214.28	227.71		
13	226.98	225.82	14	218.24	214.55		
15	216.39		16	216.47	213.78		
17	229.49	230.97	18	213.53	204.87	203.97	
19	216.83		20	223.89	231.55		
Avg. (omitting 13 & 17)	216.60		Avg.	216.67			
Precision of duplicates	2.79%			2.77%			

It is to be observed that there were two samples in the 234-5 Building definitely higher than the average of the remaining samples. In the 231 Building one sample was definitely higher than the average. Whether these results indicate a definite lack of homogeneity of the original solution, it is not possible to say at the present time. It was thought that the samples might possibly have become contaminated with iron; so iron determinations were run in the 234-5 laboratory on the two samples giving high results and one in the normal range. There was no observed difference in the iron content of these three samples, all being less than 0.1 g/L.

It is interesting to observe that the results obtained in Building 231 on rerunning the samples in a period of approximately 24-48 hours after they were submitted, gives erratic results, with some answers higher and some lower than those obtained previously. These results, combined with personal observation of a large volume of gas formed in a sample stored in a peanut, would indicate that the present laboratory practice of not depending on samples after approximately 8 hours' storage in peanuts appears to be justified.

After the above results were reported by the laboratories, I undertook to titrate some of the samples on equipment in 234-5 laboratory, and obtained the following results:

Wednesday, Nov. 8 - Values of 223.68 and 221.37 g/L
Thursday, Nov. 9 - Values of 217.42 and 220.48 g/L on samples directly from the storage flask.

[redacted]

In addition, one titration was made on a sample which had been stored in a peanut for approximately 4 hours, giving a value of 217.84 g/L. When pipetting this sample, a large volume of gas was evolved from the solution. If this behavior, i.e., gas evolution, was common in the samples submitted, it would account for the low results. However, it would be expected that gas in the samples would give a wider variation in the results. As can be seen from the figures in the table, the precision of the results is only slightly higher than normal laboratory performance.

The results from the 231 Building are the work of 12 different operators while those in the 234-5 Building were obtained by 4 different operators. It is difficult to see why the control laboratory results were lower than the value obtained by gravimetric standardization. The agreement between the laboratories would indicate that their work is reproducible. It would appear that additional work should be done on preparation of a standard sample of plutonium. Variation within the standard solution may account for some of the observed discrepancies. However, in view of the known chemistry of plutonium it is difficult to see how this could be. In light of the results obtained on a previous sample submitted, the discrepancy found in this present study should not be regarded as too serious.

G. B. Barton
Analytical Section
Technical Services Division

GBB:kb