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

A chemically pure solution of Pu was prepared for use in a re-investigation of the present 49 titration method. The purification of this solution consisted of an oxalate precipitation from a dilute NT solution followed by dissolution in nitric acid. Two peroxide precipitations were carried out with the final precipitate being dissolved in a small volume of concentrated HNO_3.

Spectrographic analysis of this sample indicated the following; impurities which are expressed as p.p.m.: Ca 40, Cr 40, La 40, Mg 4, Na 10, Ni 20.

Three 500 µl. portions of the sample were dried and ignited to PuO_2. From the weight of the residue and the impurity analysis, the concentration, g/L, of the solution was found to be 222.2, 221.5, and 222.5; average 222.0.

This standardization was carried out April 13. On May 11 six samples of this solution were submitted to the control laboratory in 231. On May 23 six samples of this solution were submitted to the control laboratory in 234-5. The results reported were:

231 Laboratory		234-5 Laboratory	
219.3		228.2	
223.6		233.3	
221.5		220.2	
220.9		236.1	
222.3		225.2	re-run 220.6
228.2	re-run 229.3	227.9	
average 222.6		average 227.3	

The analyses made by the control laboratories were not corrected for iron since this constituent is known to be low in these samples.

Since a considerable lapse of time occurred between the gravimetric standardization and submission of the samples to the laboratories for analysis, a correction for increase in concentration due to decomposition of the water by alpha particles and evaporation was calculated. Four 10 ml. flasks of the solution had been weighed at the time of filling and again after 54 days to give a rate of loss of water from the solution. The calculated concentrations at time of submission of the samples were 222.4 g/l for 231 laboratory samples and 222.6 g/l for the 234-5 laboratory samples. A correction for iron equivalent to approximately 0.4 g/l should be applied to the laboratory results.

The results reported by the 231 laboratory are very consistant excepting for one determination and the average value agrees with the gravimetric determination. The average of results from the 234-5 laboratory are two

percent higher than the average of the gravimetric results and the method does not appear to be under as good control as it is in the 231 laboratory.

Further studies are being made on the chemical method for plutonium to solve the difficulty.

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