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This document consists
of 2 Pages

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RE: ANALYTICAL RESULTS ON LEACHINGS FROM NUTSCHE FILTERS

This is to report our findings on the three leach samples submitted to us for examination. These samples were the first, third, and eleventh from a series of 60% nitric acid leaches on the filter aid and filter block of the Nutsche Filters in Cell 2 of the 231 Building. The analyses were performed by various groups of the Analytical Service Section, as well as by George Alkire (Pu-240) and Harold Miller (Am) of the Analytical Research Section. The results may be summarized:

	$\frac{La}{g./l.}$	$\frac{Pu}{g./l.}$	$\frac{PO_4}{g./l.}$	$\frac{F^-}{g./l.}$	$\frac{Pu^{240}}{\%}$	$\frac{Am}{\%}$	$\frac{Est. M.W.D.}{\%}$
Filter Aid Leach	5.3	1.89	0.50	0.25	3.46	0.11	480
1st Block Leach	<0.1	0.27	0.13	0.13	3.15	0.20	430
8th Block Leach	0.12	0.46	0.21	0.04	3.00	0.18	410

The values given for Pu-240 are weight percentage of the total plutonium and for americium, percentage of the total alpha count. The M.W.D. was estimated from a curve relating Pu-240 content to exposure level as determined on several runs over an extended range of power levels.

Examination of the data gives a Pu/PO₄ ratio in the three samples of 3.78, 2.07, and 2.19. The simple plutonium phosphates have composition ratios of Pu(H₂PO₄)₄ 0.616, Pu(HPO₄)₂ 1.24, Pu₃(PO₄)₄ 1.88. The amount of Pu which could combine with the indicated amount of PO₄ to form Pu₃(PO₄)₄ would be 0.94, 0.244, and 0.395 g./l., respectively, for the three solutions. The indicated amounts of fluoride are

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more than sufficient to combine (as any of the simple fluorides) with the remaining plutonium in the two block leach solutions and somewhat more than the stoichiometric amount for formation of Pu F_3 in the filter aid leach. Considering that entrainment of plutonium nitrate and formation of similar insoluble lanthanum compounds was not allowed for in the calculations, it appears that the plutonium was held up as an insoluble compound which was more readily dissolved in 60% HNO_3 than in 25% HNO_3 .

When we first began investigating this problem, it was thought there might be some polymeric or colloidal plutonium in the solution which was adsorbed by the filter media. Comparison of samples of the F10-P solution against samples of the derived P-1 solution with a Beckman spectrophotometer indicated there might be some polymer which was removed by the filtration step.

The data indicate that the material leached from the filter aid was definitely different from that leached from the filter block -- differences in Pu-240 and Am contents. The increase in americium content of the block leach material would appear to indicate material of an older age if the leaching rate of Am remained constant for the series of leaches.

From the data we would conclude that the holdup on the Nutsche Filter is due to insoluble plutonium compounds either filtered from solution, or more likely, formed on the filter. These compounds would have a relatively low solubility in 25% HNO_3 and would dissolve slowly, thus permitting a gradual buildup of material on the filter which was not detected until leaching with 60% HNO_3 was instituted.

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