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This document consists of 2 Pages

G. V. Packer
Separation Technology Division

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 66% nitric acid leaches on the filter aid and filter block of the Nutche 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:

<table>
<thead>
<tr>
<th></th>
<th>g/l.</th>
<th>g/l.</th>
<th>g/l.</th>
<th>%</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Filter Aid Leach</td>
<td>5.3</td>
<td>1.89</td>
<td>0.25</td>
<td>3.46</td>
<td>0.11</td>
</tr>
<tr>
<td>1st Block Leach</td>
<td>&lt;0.1</td>
<td>0.27</td>
<td>0.13</td>
<td>3.15</td>
<td>0.20</td>
</tr>
<tr>
<td>8th Block Leach</td>
<td>0.12</td>
<td>0.46</td>
<td>0.21</td>
<td>3.00</td>
<td>0.18</td>
</tr>
</tbody>
</table>

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 Pu₄(PO₄)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 0.325 g/l., respectively, for the three solutions. The indicated amounts of fluoride are...
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 stoichio-
metric amount for formation of Pu F₃ in the filter aid leach. Considering that
entrainment of plutonium nitrate and formation of similar insoluble lanthanum com-
pounds 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% HNO₃ than
in 25% HNO₃.

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
F-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 dif-
ferent from that leached from the filter block -- differences in Pu-240 and Am
contents. The increase in samarium 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% HNO₃
and would dissolve slowly, thus permitting a gradual buildup of material on the
filter which was not detected until leaching with 60% HNO₃ was instituted.