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POST OFFICE BOX P
OAK RIDGE, TENNESSEE

CF-56-6-159

DATE: June 29, 1956

SUBJECT: Recovery of Uranium from Recycle Wastes
by Fluoride Volatility - Industrial
Technical Information MeetingFROM: G. I. Cathers
D. E. Ferguson**LEGAL NOTICE**

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For The Atomic Energy Commission

H. R. Canale
Chief, Declassification Branch**UNCLASSIFIED**

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POST OFFICE BOX P
OAK RIDGE, TENNESSEE

CHEMISTRY--SEPARATION PROCESSES

FOR PLUTONIUM AND URANIUM

DATE: August 3, 1956

SUBJECT: HRP-BP: HRT UO₂SO₄ Blanket Solids Dissolving
and Corrosion Tests in Phosphoric Acid Solutions

TO: E. O. Nurni

FROM: W. L. Carter

CLASSIFICATION CANCELLED

DATE MAR 19 1957

For The Atomic Energy Commission

T. R. Canale
Chief, Declassification Branch

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OAK RIDGE NATIONAL LABORATORY

August 3, 1956

TO: E. O. Nurni

FROM: W. L. Carter

SUBJECT: HRP-BP: HRT UO_2SO_4 Blanket Solids Dissolving and Corrosion Tests in Phosphoric Acid Solutions

Recent development by R. E. Leuze in dissolving simulated fission and corrosion product solids has shown that concentrated phosphoric acid--most probably pyrophosphoric acid $H_4P_2O_7$ --is quite effective in putting these solids into solution. While these experiments are continuing, it might be pertinent to perform a few larger scale dissolution tests and check the corrosion resistance of several metal coupons which might be suitable for construction materials.

In our conversation of July 23 we outlined a suitable dissolving experiment and mentioned several metals that merit testing in the dissolver solution. It is the purpose of this memorandum to reiterate the plans for this experiment as well as tabulate a few items of data that are pertinent to work on the blanket processing system.

Blanket System Solids Composition

The amounts of the various insoluble compounds to be expected in the HRT UO_2SO_4 blanket have been calculated from available corrosion data. These data indicate that the attack on stainless steel will be of the order of 5-10 mils⁽¹⁾ per year and that the attack on zirconium will be about 3 mils⁽²⁾ per year. Based on a proposed ten-day operating cycle for the hydroclone, it has been calculated that the solutions and solids for dissolving will have the following composition:

<u>Underflow Receiver Composition</u>	<u>gms/kg D_2O</u>	<u>% in Solids</u>
UO_2SO_4	355	
$CuSO_4$	0.071	
H_2SO_4		
Fe_2O_3	51.4	70
Cr_2O_3	13.3	18.1
NiO	7.1	9.7
ZrO_2	0.88	1.2
SnO_2	0.015	0.02
PuO_2	0.64	0.86
$(FP)_2(SO_3)_3$	0.0066	

(1) Personal communication with J. C. Griess

(2) Personal communication with G. H. Jenks

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This is the composition of the material drained from the underflow receiver. After evaporation to recover the associated heavy water, dissolving solution will be added.

Dissolving Conditions

R. E. Lauze and R. D. Baybarz report that simulated fission and corrosion products have been dissolved in concentrated H_3PO_4 --or $H_4P_2O_7$ --at $230^\circ C$. However, from a design and operating point of view it would be more suitable if the operation could be carried out at a lower temperature. Since 250 psia steam ($T = 205^\circ C$) is the hottest steam in the plant, attainment of $230^\circ C$ would have to be accomplished by electric heat directly on the dissolver or indirectly by superheating the steam. Whereas steam heating is convenient and more trouble-free, it would be well to dissolve at $T < 205^\circ C$ so that the available steam can be used. It should not be difficult to maintain $175^\circ C$ in the dissolver; so it is suggested that attempts be made to put the solids into solution at this temperature.

A second fact observed by Lauze and Baybarz is that solids which have been baked are easier to dissolve than those which have not been baked. This is fortuitous, and it would be well to verify this on a UNOP scale. More complete heavy water recovery could be obtained if the contents of the dissolver were evaporated to dryness; also, the need for level measurement or control would be obviated. Therefore, it is suggested that after evaporation the solids be baked by heating to the dissolution temperature before the solvent is added.

Storage Conditions

After solution of the solids in phosphoric acid and after sampling, the contents of the dissolver are drained to a storage tank to allow radioactivity to decay. Although the storage conditions have not been specified, there is a strong reason for storing at a more dilute H_3PO_4 concentration than that used to dissolve the solids. It might be necessary to keep the storage tank boiling in order to sweep out H_2 and O_2 from water decomposition. If so, it would be well to operate in the least corrosive H_3PO_4 concentration range. Hence, one aim in these experiments will be to establish a lower H_3PO_4 concentration for storage conditions that will be compatible with the more common structural materials such as the stainless steels.

Test Materials

There are three parts of the system for which materials of construction need to be specified: the dissolver, the storage tank, and supply piping. The operating conditions of the first two items have been discussed. The supply piping should see relatively mild service in cold H_3PO_4 , and probably one of the stainless steels will be satisfactory. The corrosion resistance of the more resistant metals should be checked on a UNOP scale by inserting the samples into the dissolving experiments. Data from TVA Engineering Report No. 2, p. 122, and Corrosion Handbook, p. 783, by Uhlig indicate that Hastelloy B, tantalum, gold, silver, platinum, Chlorimet 2, stainless steel CN-7, and stainless steel CN-25 are resistant to attack by concentrated ($\sim 85\%$) H_3PO_4 at elevated temperatures. Also, there are indications that stainless steel types 304, 316, 320, and 347 are resistant over a wide range of concentrations and at moderately high temperatures.

Suggested Experiments

The attached flow diagrams are suggestions for testing the dissolving procedure. Comments and improvements on these procedures are invited. In addition to this diagram it is suggested that the corrosion rate of cold (room temperature), 85% H_3PO_4 on the stainless steels be determined.

William L. Carter

W. L. Carter
Process Design Section
Chemical Technology Division

WLC:mep
Attachment

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Schematic Diagram For H₃PO₄ Dissolving & Corrosion Test

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