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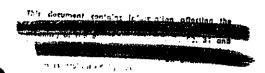
MAY 10, 1945

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MELIORANDULI REFORT SE-PC-#28

Ins Use of Ammonium Pluosilicate Analogs er Improving Decontemination in the Product Precipitation Steps

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Route List

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Introduction

Ammonium fluosilicate is now added to the process solutions in both bismuth phosphate decontamination cycles just prior to the reduction. The advantage of using fluosilicate is that it increases the decontamination of stronium and columbium by the bismuth phosphate product precipitation step. This is presumably accomplished in part at least by dissolving the "fines" of radioactive columbic oxide and sirconium phosphate that are not separated completely by the by-product centrifugation and tend to carry partially with the product precipitate.

If an agent even more effective than fluosilicate in improving decontamination in the product precipitation step were found, its use would aid in more rapid decontamination and perhaps contribute to a shortening of the separations process. This report describes the nearch for new agents for this purpose and compares their offectiveness with that of fluosilicic acid.

Summary and Conclusions

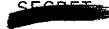
- 1. A large number of inorganic and organic compounds were evaluated as solubilizing agents for colloidal fission, and their effectiveness was compared with that of fluosilizic acid. Of those tested, only the metal fluo-complexes showed promise. A comparison of those compounds is recorded in Table I, and a discussion as to their applicability follows:
 - a) The best agent uncomered from the standpoint of effectiveness and availability is fluoboric acid (HBF4). As can be seen from the data in Table I, its use improves decontamination to a somewhat greater extent than does fluosilicic acid. Fluoboric acid is equivalent to fluosilicic acid in reducing product losses in the product precipitation step and the two are identical in their corrosion rates of 25-12 Cb steel. Furthermore HBF4 does not form an insoluble sodium or potassium salt as does fluosilicic acid. This allows a little more flexibility in process changes. Evaluation in the Hot Semi-Works may be warranted.
 - b) Ammonium fluotitanate was investigated in some detail because of its potential availability. This compound, though effective in improving decontamination, had the unfortunate and unpredictable property of complexing biamuth, completely preventing the precipitation of the phosphate under some conditions and inhibiting the precipitation under all conditions.
 - c) The use of fluomolybdic said resulted in vory good decontamination, but probably would not be practical for plant use because of its relative unavailability.
 - d) Fluorensiic acid offered some improvement over fluorilicia soid, but the difficulty of producing large amounts of vanadium compounds makes its use prohibitive.
 - e) Fluouranic acid offered some improvement in decontamination, while the use of fluozirousic acid resulted in decontamination inferior to the control.
- 2. The use of free EF in conjunction with fluosilicic acid resulted in decontamination better than that obtained by the employment of fluosilicate alone. (Table I).

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- The properties of certain metal fluocomplexes made their evaluation impossible. These are recorded in Table II.
- 4. A list of a number of agents evaluated that had little or no effect on decontemination improvement is recorded in Table III.
- 5. Several agents were evaluated for the improvement of decontamination at the extraction step by adding the compound to the UNH solution. Only oxalic acid (Table IV) has a beneficial effect. The use of oxalic acid as a pre-reduction agent in the plant might be warranted.

Experimental Details

Extractions were made from 20% UNH (Semi-Works dissolved metal) fortified with plant UNH (60 to 85 days old metal) and with " concentrations of fission elements supplied by spiking with inactive isotopes. Bith the exception of prereduction, the ex ractions were carried out as outlined in the H.E.T. October 5th flowsheet. By-product precipitations comprised bismuth phosphate only. Reductions and product precipitations were made as outlined in the plant flowsheet, and the solubilizing agent was added at the beginning of the reduction. Generally fairly large extractions (2 to 3 liters) were made and the cycle carried through the by-product on the same scale. Aliquots of the oxidized effluent were then employed for comparative tosts.

The sample of fluoboric acid was purchased from Pennsylvania Salt Co., had a density of 1.30, and assayed approximately 30% HBF4.

The sample of amuonium fluotitanate was prepared from TiO2 by G. ". Sears in the E.E. .. laboratories.

Other fluo-derivatives were prepared from suitable compounds and HF.

Fluotitanic acid inhibits the precipitation of the bismuth phosphate product precipitate from process solutions. This precipitation is actually prevented at low concentrations of phosphoric acid. This is illustrated by the following data.

0.05% H2TiF6 Present

0.03. Fe;	2.5 g/1 Bi; 1N HNO3
H3PO4 Concentration	Temperature Above Which No Crystallization Occurs
O-64	22°C
C-87	50 ⁰ €
1.0%	65-70 ⁰ C

Even though crystallization is initiated at the temperatures indicated, several hours aging at rocm temperature are required for complete precipitation to occur.

> L. SQUIRES, CHIEF SUPERVISOR SEP.ENG. -200 ARIA TECH.DEPT.

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f Other Complex Flug-Compounds with Ammonium Flugsilicate with Respect To Decontamination, at the Product Precipitation Step

			•
And Asset	Decontamination	Decontamination	Decontamination Factor
	For Step	For Cyule	Cycle Plus Extraction
None (Control)	A cs	17.1	נונ
Parsa(Very) Ego-	9	34°2	222
. 105M 1105M	27.3	104	682
THE PARTY	8.7	32.9	220
(1) PLOY OLDS MONTH MONTH	-11-6	8	291
	i ca	28.8	182
運	**	59.6	387
- 100 July 2011 6	42.1	202	1520
Taona (Control)	2.6		
~ 24	6.8		
9 at 12 (1914) 115.	8.8		
133	000		
O TO CALL OF THE PAGE	99°C		
	70.6		
Mone (Control)	4.0	19.2	129
OSW (MR4) 2SIF6	8.4	&Oc Ø	272
-OSH Fluorolybdio soid	84.4	€07	29 1
-05M Filmoranseid soid	• 57	176	2 4
Ob's Fluorismonto acid	3.2	15.4	
.Obli Finosi wanie acid	71.6	344	2300
. Obli tumping acid	20.4	98	656
AH MI . PATEOTEPHEN MOO.	. 21.8	105	704
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Table II BEST AVAILABLE COPY

Tested Fluo Compounds Whose Properties
Prohibit Their Use

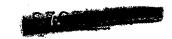
Agent Tested	Reason Thy Cannot Be Used
.ObM Fluotungstic Acid	

Table III

Agents Tested with Little or No Effect on Deschtamination in the Product Precipitation Step

Added Agant .	Decontamination Factor For Stepe	Decontamination Factor For Cycle *
None (Control)	5 ₄ 1	●40.8●
ZOT mg/kitter las.	5.6	44.8
O. M. (ME) Sir 6 (Control)	11	68
Maple (Control)	2-1	6.7
0.05M Chiloracetic soid	2.55	8.2
0.05% (NB_) 551F6 (Control)	7.5	22.6
Alone (Control)	2.5	15.7
o.one.ip	2.6	16.3
C-01N fic1	3%2	20.4
O.OIM BEF	3.5	22.0
O.OIN HI	4.8	50.2
0-05H Trichloracetic scid	3.0	18.9
O.OlM Trichloracetic acid	3 . Q	18.9
0.05% Sodium 5-nitro 6-chloro toluene sulfe	anate 3.2	20-4
0.05M (NB ₄) ₂ SiF ₆ (Control)	12.9	87.6
None (Control)	5.1	34.2
0.01% HIO3	. 3. 8	25.5
0.08 Picric acid (not completely acluble)	2+2	14.7
0.05% Ammonium Vanadate	4.5	30-2
0.05% Sulfamic anid	8.2	56
0.051 (NH4)251F6 + .011 HI	10	67
0.05E (NH4)2SiF6 (Control)	14.8	99

* Overall gamma decontamination for by-product without scavengers and product precipitation stans.



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Table IV

Complexing Agents is UNE Product Extraction

Added Agent	Decontamination Factor
None (Control)	5.2
0.03% Oxalic soid	12
0.03% Tartaric acid	4.2
0.03% Citric soid	5.1
0.03H HF	5.4
None (Control)	7.1
O.ll: Furfural	8.9

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