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SUBJECT: HRT-CP: Results of Solids Dissolution Tests

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HRT-CP: RESULTS OF SOLIDS DISSOLUTION TESTS

Summary

A dissolution cycle in the chemical plant consists of refluxing with 10.8 M sulfuric acid for 4 hours followed by refluxing for a like period with 4 M acid. In each of 3 tests in which 370-gram batches of simulated corrosion product solids were subjected to two dissolution cycles using 820 per cent excess acid, more than 99.6 per cent of the solid material was dissolved (based on the amount of undissolved material collected). In another test, using one dissolution cycle and 820 per cent excess acid, 99.5 per cent of the solids material was dissolved. In the final test in which a 530-gram batch of solids was subjected to two dissolution cycles using 540 per cent excess acid, only 91.7 per cent of the solid material was dissolved.

The corrosion rate of a specimen of Carpenter 20 suspended in the vapor space of the dissolver during these tests was 0.01 mil per year.

Procedure and Results

A series of five solids-dissolution tests was carried out in accordance with a procedure similar to that to be followed in actual operation of the chemical plant. Briefly, this procedure involved (a) charging a batch of simulated solids and uranyl sulfate solution to the dissolver, (b) evaporating to dryness, (c) charging 4 M sulfuric acid to the dissolver, (d) concentrating to 10.8 M acid and refluxing for 4 hours, (e) diluting to 4 M and refluxing for 4 hours, (f) repeating steps d and e (except in one test), (g) transferring the solution from the dissolver and sampling, and (h) rinsing the dissolver.

The simulated solids used in the tests contained about 47 per cent ZrO_2, 40 per cent Fe_2O_3, and 13% Cr_2O_3. The solids were suspended in a uranyl sulfate solution.

The results of the dissolution tests are presented in Table I.

Table I. Results of Dissolution Tests

Test No.	Wt of Solids, g	Excess Acid, %	No. Dissolution Cycles	Solids Dissolved, [a] %
1	370	820	2	99.7
2	370	820	2	99.9
3	370	820	2	99.6
4	370	820	1	99.5
5	530	540	2	91.7

[a] Determined by weighing the undissolved solids in the dissolver solution and rinse solutions.

Dissolution of solids was substantially complete when the amount of excess acid was about 820 per cent, even when only one dissolution cycle was employed. In the test in which the amount of excess acid was 540 per cent, the proportion of solids dissolved was considerably lower. This result is not in agreement with those of similar experiments carried out by the development groups. No reason for the disparity is evident. It is anticipated that in actual operation, less than 400 grams of solids will be handled in a dissolution. With this amount of solids, sufficient acid can be employed to obtain complete dissolution.

Material balances for H_2SO_4, Cr, Fe, U, and Zr are shown in Table II.

Table II. Material Balances for H_2SO_4, Cr, Fe, U, and Zr

	Run Number				
	1	2	3 [b]	4	5
H_2SO_4	97%	100%	111	95%	98%
Cr [a]	115	87	85	97	97
Fe [a]	101	104	130	107	133
U [a]	110	120	99	120	99
Zr [a]	64	81	82	86	96

It is apparent that the balances are poor; no conclusions can be drawn from them.

Analyses of dissolver solutions are shown in Table III. Spectrographic analyses of undissolved solids from runs 3 and 4 are shown in Table IV.

Table III. Analyses of Dissolver Solutions

Constituent	Run Number				
	1	2	3	4	5
H_2SO_4, N	7.3	7.6	7.4	7.2	7.5
U, mg/ml	7.1	6.1	6.3	6.1	7.0
Fe, mg/ml	6.9	7.3	8.1	7.4	9.2
Cr, mg/ml	2.0	1.7	1.5	1.9	1.7
Zr, mg/ml	5.4	6.8	6.9	7.2	5.6
Cu, ppm	48	21	-	-	-
Ni, ppm	225	25	112	18	180
Cl, ppm	2	0	-	3	-

Table IV. Average Spectrographic Analyses[a] of Undissolver Solids

Constituent	%	Constituent	%
Al	0.1 - 1	Ni	10 - 100
Cr	10 - 100	Pb	0.01 - 0.1
Cu	0.01 - 0.1	Si	0.1 - 10
Fe	10 - 100	Ti	0.01 - 0.1
Mn	0.01 - 1	Zr	0.1 - 10

[a] Includes only those constituents present in amount greater than 0.01%.

The nickel content of the dissolver solutions appears to show that corrosion had taken place at a rate of 5 mils per year on the Carpenter 20 lines connected to the tantalum-lined dissolver. It is more likely, however, that the nickel resulted from corrosion of the equipment in which the solids were prepared. Although analyses of the slurries charged in these tests were not made, slurries prepared in the same manner contained enough nickel to account for that found in the

dissolver and rinse solutions. The results of a corrosion test bear out the belief that there was little corrosion of the piping adjacent to the dissolver.

Prior to the dissolution tests, a 2-inch-long piece of 1/2-inch Carpenter 20 tubing was suspended in the vapor space of the dissolver near the top flange. This specimen was exposed during 52 hours of refluxing at 4 M acid concentration, 18 hours of concentration to 10.8 M, and 66 hours of refluxing at 10.8 M, or a total of 136 hours. The weight loss of the specimen was 1.1 mg, corresponding to a penetration of 0.01 mils per year, based on 52 runs of 20 hours each per year.

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