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SUBJECT: Preliminary Solids-Injection Experiments
 on the HRT Mockup at Y-12

TO: F. R. Bruce

FROM: J. R. Engel

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PRELIMINARY SOLIDS-INJECTION EXPERIMENTS
ON THE HRT MOCKUP AT Y-12

Summary

Three experiments were performed which involved the injection of simulated reactor fission and corrosion product solids into the HRT Mockup and their attempted removal with a hydroclone.

The first two runs, in which solids were injected as dissolved sulfates and precipitated in the high-pressure system, provided only a rough qualitative picture of the behavior of the solids. Apparently all of the solids plated out over the walls of the high-pressure system with the result that essentially none were recovered via the hydroclone underflow pot.

In the third experiment, the solids were injected as the already-precipitated oxides of the corrosion product materials. In this test a solids concentration ratio of 28:1 was achieved between the hydroclone underflow pot and the main loop. The concentration ratio for Nd, a representative of the rare earth fission products, was 60 as determined from tracer activity measurements. An 88% material balance was obtained on the Nd activity in the test.

Introduction

Solids will be formed in the HRT reactor system as a result of the fission process and by corrosion of piping and equipment. The rare earths which form a large fraction of the nongaseous fission products are only slightly soluble in the sulfate form at the reactor operating temperature and may be expected to precipitate in the fuel solution. In addition, the oxides of Fe, Zr, and Cr which result from corrosion will be at least partially suspended in the solution after an equilibrium scale has been built up on all of the equipment. The HRT processing plant is designed to remove these suspended solids from the circulating stream with a hydroclone operating on a small side stream from the main loop.

A large amount of information has been obtained concerning the behavior of simulated fission and corrosion product solids at HRT conditions of temperature and pressure in small pump loops equipped with hydroclones. The purpose of the

work described here was to study this behavior in equipment which more nearly approximates the HRT system in size. Therefore, the HRT Mockup at Y-12 was selected for the tests since it contains several components that are duplicates of those installed in the reactor.

The general plan was to inject solids, or materials that would precipitate to form solids, into the high-pressure circulating stream of the mockup and then attempt to remove them with a hydroclone. Radioactive tracers were employed to follow the behavior of the solids within the system.

Description of the Tests

A 0.4-inch hydroclone, equipped with a 1.5-liter underflow pot was connected between the suction and discharge lines of the mockup circulating pump as shown in Fig. 1. An ORNL, 5-gpm, canned motor pump was installed in the hydroclone overflow stream to increase the pressure drop available to drive the hydroclone. The heater and screen which precede the hydroclone in the HRT chemical processing plant were not included in the installation. However, the ratio of underflow pot volume to high-pressure loop volume is approximately the same as in the HRT. Two HRT valves were used to provide a means of isolating the hydroclone loop from the rest of the system.

The operating procedures for the first two solids injection tests were essentially the same. In both tests, a 10-gm U/l solution of UO_2SO_4 was continuously pumped into the mockup high-pressure system with a Lapp pulsafeeder at a rate of 1 to 2 liters per hour. This solution contained dissolved sulfates of iron, chromium, zirconium, and mixed rare earths in the same proportions as they are expected to occur in the HRT. Upon exposure to the high temperature, the rare earths precipitated as the sulfates while the other metals hydrolyzed and precipitated as the oxides. The hydroclone was operated continuously during the injection periods. Enough solution was added in each of the tests to deposit 350 grams of fission and corrosion product solids. Regular samples were taken through an HRT-type sampler and from the mockup let-down stream at the let-down valve.

The solution in the first of these runs was traced with La^{140} as a representative of the rare earths and the second was traced with Zr^{95} to follow the corrosion products. Radiation monitors were installed at several points around the high-pressure loop in an attempt to detect any localized buildup of solids.

SIMPLIFIED MOCKUP FLOWSHEET
WITH SOLIDS ADDITION AND
REMOVAL EQUIPMENT

Fig. 1

After each test, the hydroclone loop was isolated and the mockup was shut down and drained. Samples of the loop drain and the underflow pot contents were taken for analysis.

The method of solids injection was modified before the start of the third test. The hydrolysis of large quantities of metal sulfate to the oxides left the mockup solution with an abnormally high sulfate ion concentration. It appeared that this might adversely affect the corrosion data on the loop. Therefore, provision was made to permit the addition of solids in the already-precipitated form, as the oxides.

Two lines were installed across the hydroclone loop so that a bomb containing small batches of solids suspended in UO_2SO_4 solution could be attached. These solids were displaced into the mockup high pressure system with the hydroclone isolated by the HRT-type valves. Following each addition, the hydroclone was operated to remove the solids from the circulating stream. The solids bomb was isolated and removed from the loop at varying times after the hydroclone was started to provide samples for determining the rate of solids removal.

For the test in question 341 grams of simulated fission and corrosion product solids were added in four batches as shown in Table 1. A Nd^{147} tracer was used to follow the general behavior of the rare earths. In addition to the four samples taken when the solids bombs were removed, three other bomb samples were taken, one at the time the hydroclone was shut down and two several hours later. The results are shown in Table 2. Nine hours after the last solids addition the hydroclone was isolated and the underflow pot cooled and drained. Material balances were made based on total solids and on the Nd tracer activity.

Results

The results of the first two experiments provided only very general information about the behavior of the solids. In neither case was any appreciable quantity of solid material recovered, either from the underflow pot or from the solution drained from the system. In the La-traced run the activity was distributed uniformly throughout the high- and low-pressure parts of the system. However, the Zr tracer in the second test all remained in the high pressure system with slight concentration effects in the heat exchanger and the gas separator. Shortly after the first of these tests was started, the feed injection line became plugged and all subsequent additions were made through one of the taps in the orifice flange.

TABLE 1. SOLIDS ADDITIONS TO HRT MOCKUP

Bomb No.	Date	Solids Added (gm)
1	7/23	92.5
2	7/24	92.5
3	7/25	92.5
4	7/26	65

Total additions 341.5

Previous additions 700

1,041.5 gm

Solids concentration

in bombs : 185 gm/l

Tracer : Nd^{147}

Supernate : UO_2SO_4 solution

Uranium concentration: 10.37 gm/l

TABLE 2. SAMPLES FROM HRT MOCKUP

Bomb No.	Date Removed	Time After Hydroclone Start-up	Solids Concentration (gm/l)
1	7/23	15 min.	4.15
2	7/24	30 min.	1.99
3	7/25	1 hr	4.35
4*	7/26	4 hr	8.75
5	7/26	9 hr	0.56
6	7/27	-	0.18
7	7/27	-	0.20

*Mockup was thermal-cycled while bomb was in place.

All of this information seems to indicate that the highly insoluble oxides were plating out in the high pressure system a very short time after their formation. The even distribution of the La may be explained by the relatively high solubility of $\text{La}_2(\text{SO}_4)_3$ under the test conditions. Since the samples

withdrawn from the HRT sampler contained no solids, no directly applicable information was obtained from them. However, the operability of the sampler mechanism was demonstrated.

During the first two tests, the HRT valves that were used to isolate the hydroclone were observed to leak badly. It was learned that Stellite trim had inadvertently been used in both valves. During the shutdown for installation of the solids injection bomb, this trim was replaced with type 347 stainless steel. Subsequent operation of the valves was very satisfactory.

The results of the third experiment provided more specific information than either of the first two. However, several factors tend to introduce uncertainty into the interpretation of the data. The mockup was started and operated for three days before any solids were added. During this time the hydroclone was not in operation. The addition of the first bombful of solids should have resulted in a concentration of 1 gm/l. However, the sample (see Table 2) indicated a concentration of 4.15 gm/l. During the injection of the fourth batch of solids, circulation was maintained through the bomb for a period of four hours. In this time, failure of a rupture disc in the high-pressure steam boiler which maintains the mockup temperature allowed the loop to go from 300°C to 140°C and back to 290°C. This sample also showed an abnormally high concentration of solids. Both of these items seem to indicate that thermal cycling has a pronounced effect on the resuspension of solids that have plated out on pipe walls. The effects of previously added solids and of thermal cycling made it impossible to determine from the sample results any information relating to the rate of solids removal by the hydroclone.

At the completion of this test, the hydroclone underflow pot was cooled, drained, and flushed with water. A total of 236 grams of solid material was recovered, which contained an appreciable amount of Zr⁹⁵ activity. This indicates that some of the solids that were injected in the second run were removed in this test. Comparing the solids concentration in the main loop at the time the hydroclone was shut down (bomb No. 5, Table 2) with that in the underflow pot, it is evident that a concentration factor of 281 was achieved.

Following the completion of this test, the mockup was shut down and cut open to permit the removal and inspection of corrosion specimens. At this time a large deposit of solids was discovered in the horizontal line connecting the pressurizer to the main loop. A total of 325 grams was removed from this location. The solids removed from the pressurizer line had a much higher specific activity of the Zr⁹⁵

than did those in the underflow pot, indicating that they were primarily solids injected in the first two tests. A total of 628 grams of solids was recovered from the Mockup for these three experiments. This represents 60% of the amount injected. A summary of the solids recovery is given in Table 3.

In addition to the solids material balance, an activity balance based on Nd^{147} was made for the third experiment. Of the Nd injected, 88% was recovered from the underflow pot and high pressure system. The concentration factor achieved for the Nd was 61. Within the limits of analytical accuracy, all of the Nd which remained in the high pressure system was in solution so that further separation by the hydroclone could not be expected. Table 4 gives a summary of the Nd activity balance.

Conclusion

The results of these experiments have served to corroborate information obtained on smaller loops. It has been shown that solids will be efficiently removed by a hydroclone if they remain in suspension long enough to be transported to the clone. The results also indicate that solids were not transferred to the dump tanks.

Since the solids removal was much more successful in the third test than either of the others, it appears that the mechanism of formation of the solids has a definite effect on whether they will plate out on the walls or remain in suspension. Further work will be required before any definite conclusions can be reached in this matter. It also appears that thermal cycling of the equipment is beneficial in resuspending solids that have been deposited on the walls. This, however, needs to be verified.

The deposition of solids in the pressurizer connection is the result of an unfortunate design of the joint. The solids are carried into this relatively stagnant region by eddy currents which are not strong enough to resuspend the solids, once they have settled out. A small difference in elevation between the main circulating line and the pressurizer connection would probably alleviate the problem.

TABLE 3. SOLIDS BALANCE

Total solids added		1,042 gm
Solids recovered		
Underflow pot drain	236 gm	
Underflow pot rinse	14	
Pressurizer connection	358	
Loop drain	9	
Samples	<u>11</u>	
Total solids recovered		628 gm
Percent solids recovered		60%
Solids concentrations		
Underflow pot	157 gm/l	
Loop	0.56 gm/l	
Solids concentration factor		281

TABLE 4. Nd¹⁴⁷ ACTIVITY BALANCE

Total Nd tracer activity added:		7.48x10 ⁸ c/m
Activity recovery		
Underflow pot solids	0.37x10 ⁸ c/m	
Underflow pot liquid	2.56x10 ⁸	
Loop solids	0.08x10 ⁸	
Loop liquid	<u>3.57x10⁸</u>	
Total Nd tracer recovered		6.58x10 ⁸ c/m
Material balance		88%
Fraction of Nd activity in underflow pot		44%
Specific activities		
Underflow pot	1.91x10 ⁵ c/m/ml	
Loop	3.10x10 ³ c/m/ml	
Nd concentration factor		61

The concentration factors obtained on the Mockup, both for corrosion produce solids and for rare earths, as represented by Nd, are considerably greater than those required for successful operation of the HRT processing system. The Mockup operation also indicates that the perpendicular take-off lines from the main circulating stream to the solids removal equipment will function satisfactorily.

J. R. Engel

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