OAK RIDGE NATIONAL LABORATORY

OPERATED BY

UNION CARBIDE NUCLEAR COMPANY

POST OFFICE BOX X
OAK RIDGE, TENNESSEE

February 11, 1958

U. S. Atomic Energy Commission
Technical Information Service Extension
Post Office Box 62
Oak Ridge, Tennessee

Attention: Miss Margaret Pflueger

Gentlemen:

Subject: CF 57-4-139 (Unclassified)
CF 57-12-1 (Unclassified) 4

Enclosed are seven copies each of the subject documents which we feel are of interest to the Pennsylvania Advanced Reactor Project. We will appreciate you making these documents available to them. They may also be made available to the Civilian Application Program if considered applicable.

Very truly yours,

OAK RIDGE NATIONAL LABORATORY

*1 cc. Carbide
5 cc. Westinghouse*

Carl H. Brown
N. T. Bray, Supervisor
Laboratory Records Department
Technical Information Division

/mh

Enclosures (14)

ORNL:RC

CC; J. W. Norris
Class. Dist. Br., AEC, OR
w/o encls.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

UNCLASSIFIED **OAK RIDGE NATIONAL LABORATORY**

Operated By

UNION CARBIDE NUCLEAR COMPANY POST OFFICE BOX X
OAK RIDGE, TENNESSEEEXTERNAL TRANSMITTAL
AUTHORIZED**ORNL**
CENTRAL FILES NUMBER

57-4-139

DATE: April 30, 1957

COPY NO. 274

SUBJECT: HRP Dynamic Slurry Corrosion Studies: Quarter Ending April 30, 1957

TO: E. G. Bohlmann

FROM: E. L. Compere, H. C. Savage, S. A. Reed, R. M. Warner, W. C. Ulrich,
H. D. Cole, S. R. Buxton, and J. A. Russell, Jr.DISTRIBUTION

1. HRP Director's Office	26. C. A. Burchsted	51. J. R. Engel
2. G. M. Adamson	27. S. R. Buxton	52. J. L. English
3. W. L. Albrecht	28. D. W. Cardwell	53. L. L. Fairchild
4. T. A. Arehart	29. W. L. Carter	54. H. L. Falkenberry
5. A. L. Bacarella	30. G. H. Cartledge	55. D. E. Ferguson
6. J. E. Baker	31. R. H. Chapman	56. J. D. Flynn, Jr.
7. H. F. Bauman	32. R. D. Cheverton	57. D. F. Frech
8. S. E. Beall	33. C. J. Claffey	58. W. J. Fretague
9. R. G. Berggren	34. H. C. Claiborne	59. C. H. Gabbard
10. A. M. Billings	35. W. E. Clark	60. W. R. Gall
11. E. P. Blizzard	36. H. D. Cole	61. R. B. Gallaher
12. E. G. Bohlmann	37. T. E. Cole	62. E. H. Gift
13. J. C. Bolger	38. C. J. Colling	63. T. H. Gladney
14. S. E. Bolt	39. E. L. Compere	64. H. E. Goeller
15. E. S. Bomar	40. D. D. Cowen	65. J. L. Gory
16. C. M. Boyd	41. R. S. Crouse	66. R. S. Greeley
17. N. C. Bradley	42. H. N. Culver	67. A. T. Gresky
18. J. C. Bresee	43. J. S. Culver	68. J. C. Griess
19. R. E. Brooksbank	44. C. E. Curtis	69. J. D. Grimes
20. J. W. Brown	45. N. W. Curtis	70. R. H. Guymon
21. N. A. Brown	46. R. I. Davis	71. P. A. Haas
22. F. R. Bruce	47. V. A. DeCarlo	72. J. Halperin
23. D. F. Bruley	48. J. S. Drury	73. B. H. Hamling
24. J. R. Buchanan	49. W. K. Eister	74. J. P. Hammond
25. W. D. Burch	50. L. B. Emlet (K-25)	75. P. H. Harley

NOTICE

This document contains information of a preliminary nature and was prepared primarily for internal use at the Oak Ridge National Laboratory. It is subject to revision or correction and therefore does not represent a final report.

UNCLASSIFIED

—LEGAL NOTICE—

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

- A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or
- B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission to the extent that such employee or contractor prepares, handles or distributes, or provides access to, any information pursuant to his employment or contract with the Commission.

76. R. J. Harvey	128. O. Menis	181. J. A. Swartout
77. P. N. Haubenreich	129. C. Michelson	182. F. H. Sweeton
78. J. K. Hayes	130. E. C. Miller	183. C. K. Talbott
79. J. Hernandez-Fragoso	131. I. M. Miller	184. E. H. Taylor
80. D. N. Hess	132. G. E. Moore	185. D. G. Thomas
81. J. W. Hill	133. R. L. Moore	186. P. F. Thomason
82. C. J. Hochanadel	134. R. C. Moren	187. D. O. Thompson
83. A. Hollaender	135. C. S. Morgan	188. M. Tobias
84. H. P. House	136. K. Z. Morgan	189. D. S. Toomb
85. A. S. Householder	137. L. E. Morse	190. W. C. Ulrich
86. R. F. Hughes	138. E. J. Murphy	191. W. E. Unger
87. J. P. Jarvis	139. J. P. Murray (Y-12)	192. C. F. Vanden Bulck
88. S. Jaye	140. C. W. Nestor	193. R. Van Winkle
89. G. H. Jenks	141. P. D. Neumann	194. E. Volk
90. D. M. Johnson	142. R. H. Nimmo	195. F. C. VonderLage
91. K. O. Johnsson	143. A. R. Olsen	196. D. W. Vroom
92. D. T. Jones	144. L. F. Parsly	197. R. E. Wacker
93. E. V. Jones	145. R. L. Pearson	198. F. J. Walter
94. R. W. Jurgensen	146. F. N. Peebles	199. R. M. Warner
95. S. I. Kaplan	147. M. L. Picklesimer	200. K. S. Warren
96. P. R. Kasten	148. R. M. Pierce	201. B. S. Weaver
97. T. M. Kegley, Jr.	149. R. G. Pitkin	202. B. H. Webster
98. C. P. Keim	150. B. E. Prince	203. H. O. Weeren
99. J. O. Kolb	151. J. J. Prislinger	204. A. M. Weinberg
100. R. B. Korsmeyer	152. L. R. Quarles	205. K. W. West
101. U. Koskela	153. S. A. Reed	206. S. H. Wheeler
102. K. A. Kraus	154. W. D. Reel	207. R. P. Wichner
103. N. A. Krohn	155. P. M. Reyling	208. R. R. Wiethaup
104. J. E. Kuster	156. D. M. Richardson	209. E. P. Wigner (Consultant)
105. J. A. Lane	157. M. Richardson	210. G. C. Williams
106. M. C. Lawrence	158. A. E. Richt	211. H. E. Williamson
107. C. G. Lawson	159. P. L. Rittenhouse	212. T. E. Willmarth
108. G. W. Leddicotte	160. G. W. Rivenbark	213. J. C. Wilson
109. W. J. Leonard	161. R. C. Robertson	214. J. F. Winesette
110. R. E. Leuze	162. J. N. Robinson	215. R. H. Winget
111. R. B. Lindauer	163. A. M. Rom	216. C. E. Winters
112. R. S. Livingston	164. M. W. Rosenthal	217. L. F. Woo
113. R. A. Lorenz	165. J. A. Russell, Jr.	218. W. M. Woods
114. M. I. Lundin	166. H. C. Savage	219. W. L. Wright
115. R. N. Lyon	167. W. F. Schaffer	220. W. C. Yee
116. R. O. Maak	168. R. E. Schappel	221. F. C. Zapp
117. W. L. Marshall	169. C. E. Schilling	222-223. ORNL Document Reference Library (Y-12)
118. E. A. Mason	170. H. K. Search	224. Biology Library
119. J. L. Matherne	171. C. H. Secoy	225-226. Central Research Library (2)
120. T. H. Mauney	172. C. L. Segaser	227. Health Physics Library
121. J. P. McBride	173. E. M. Shank	228. Metallurgy Library
122. H. F. McDuffie	174. M. D. Silverman	229-230. REED Library (2)
123. H. A. McLain	175. A. N. Smith	231-233. Laboratory Records (3)
124. H. M. McLeod	176. I. Spiewak	234. ORNL RC
125. R. A. McNeas	177. R. L. Stephenson	
126. J. R. McWherter	178. H. H. Stone	
127. T. N. McVey (Consultant)	179. R. W. Stoughton	
	180. D. D. Susano	

NON-PROJECT

235. D. S. Billington	244. E. P. Epler	252. F. C. Moesel, AEC
236. C. J. Borkowski	245. J. H. Frye, Jr.	253. H. W. Savage
237. G. E. Boyd	246. W. H. Jordan	254. E. D. Shipley
238. A. D. Callihan	247. L. C. Johnson	255. M. J. Skinner
239. C. E. Center	248. M. T. Kelley	256. A. H. Snell
240. R. A. Charpie	249. S. C. Lind	257. R. P. Ward
241. S. Cromer	250. J. L. Marsh, C and CC	258. T. A. Welton
242. F. L. Culler	South Charleston	259-265. Westinghouse PAR
243. J. M. Dallavalle,	251. W. O. Milligan,	Project (7)
Georgia Institute	Rice Institute	266-280. TISE-AEC (15)
of Technology	of Technology	
(Consultant)	(Consultant)	

TABLE OF CONTENTS

SLURRY CORROSION

	Page
1.0 PUMP LOOPS	9
1.1 Loop Engineering.	9
1.2 Pump Loop Tests	16
1.3 Tests with Sulfate Additives.	17
1.4 Circulation of 650°C-Calced Thoria Slurry	29
1.5 Wear of Pump Components	30
1.6 Circulation Test in Revised Loop CS	32
2.0 TOROIDS.	45
2.1 Effect of Calcination Temperature	45
3.0 IN-PILE SLURRY TOROID DEVELOPMENT.	49

LIST OF FIGURESSLURRY CORROSION

<u>Figure No.</u>	<u>Title</u>	<u>Location</u>
1.1.1	Loop CS, Schematic	11
1.1.2	Photograph of Loop CS Showing Venturi and Sample Barrel Arrangement	12
1.1.3	Comparison of Pump Amperes and Venturi d/P with Thorium Oxide Concentration for Run CS-34	14
1.1.4	Break in 100A Pump Rotor Can Found after Run CS-34	15
1.1.5 (a)	LO-14 ThO ₂ Before Circulation (400X)	26
1.1.5 (b)	After 95 hr Circulation (400X)	26
1.1.5 (c)	After 165 hr Circulation (400X)	27
1.1.5 (d)	After 189 hr Circulation (400X)	27
1.1.5 (e)	After 260 hr Circulation (400X)	28
1.1.5 (f)	After 200 hr Circulation (400X)	28
1.1.6	Localized Attack on Type 75A Titanium Pump Impeller after Run CS-34	34
3.1	Development Model of Rotator for In-Pile Slurry Toroid	50

LIST OF TABLES

SLURRY CORROSION

<u>Table No.</u>	<u>Title</u>	<u>Location</u>
1.1	Summary of Sample Data, Loop Inventory and Corrosion Rate, Run BS-9	20
1.2	Summary of Sample Data, Loop Inventory and Corrosion Rate, Run BS-10	21
1.3	Summary of Sample Data, Loop Inventory and Corrosion Rate, Run BS-11	22
1.4	Summary of Sample Data, Loop Inventory and Corrosion Rate, Run BS-12	23
1.5	Comparative Circulation and Corrosion Effects of Batch LO-14 and Batch LO-2 Slurries at 300°C	25
1.6	Summary of Sample Data, Loop Inventory and Corrosion Rate, Run BS-13	31
1.7	Summary of Sample Data, Loop Inventory and Corrosion Rate, Run CS-34	35
1.8	Corrosion Rates of Pin and Coupon Specimens, Runs BS-9 through BS-13 and CS-34	
	(a) Run BS-9	36
	(b) Run BS-10	37
	(c) Run BS-11	38
	(d) Run BS-12	39
	(e) Run BS-13	40
	(f) Run CS-34	41-42
2.1	Results of High Temperature Circulation Tests of Various Aqueous Thoria Slurries in Toroids	46

0.0 SUMMARY

Slurry Corrosion. The assembly of a second thorium oxide slurry corrosion test facility, loop BS, has been completed and 2010 hr of operation on slurry have been logged. This second test loop has proved satisfactory from an operational standpoint.

Dynamic slurry corrosion loop CS has been modified by replacing the three annular-flow corrosion sample barrels with standard type sample barrels as used in the solution corrosion test loops (this type sample barrel is also used in loop BS). This revision eliminated the small annular-flow channel in which plugging had occurred. A calibrated venturi was also installed in loop CS as an aid in following the changes in density and/or flow rate of the circulating slurry.

A new letdown type sample device to facilitate removal of samples of the circulating thorium oxide slurry has been installed on both loops BS and CS.

Corrosion data and operational observations are given for six thorium oxide slurry runs made at 300°C in 100A pump loops BS and CS.

Four of the tests in loop BS were made to complete a study of the effect of sulfate on the corrosivity of slurries at approximate concentrations of 500 g of thorium per kilogram of water. Neither the sulfate additives (at 3000 ppm $\text{SO}_4^{2-}/\text{ThO}_2$) nor the presence of spheroids, which formed during circulation, appeared to effect the rate of attack.

The fifth test in loop BS circulated slurry prepared from 650°C-calcined thoria at an average concentration of 478 g of thorium per kilogram of water for 500 hr at 300°C. Generally, corrosion rates paralleled those obtained in previous tests with slurries prepared from thoria calcined at 800°C.

In a 500-hr run in loop CS, slurry prepared from a different batch of

800°C-calcined thoria was circulated at 300°C at an average concentration of 516 g of thorium per kilogram of water. Specimen corrosion rates were moderate, but severe erosive attack occurred in the circulating pump.

Low attack rates were generally observed in toroid tests with slurries prepared from either high- or low-calcined thoria. Slurries of thoria of 1.2 μ average size fired at 1200, 1400, and 1600°C underwent little degradation during circulation, whereas those prepared from 550 and 650°C-calcined thoria of 2.6 to 4.6 μ average size were degraded to approximately 0.7 μ during 300 hr of circulation at 250°C.

A new development model of the rotator for an in-pile slurry toroid is described.

1.0 PUMP LOOPS.

1.1 Loop Engineering. (by W. C. Ulrich)

100A Loop BS: The assembly of a second thorium oxide slurry corrosion test facility, 100A pump loop BS, has been completed and 10 runs (BS-5 through BS-14), totaling 2010 hr of operation on slurry, have been completed since a report was last made on the system. (1.1.a, 1.1.b)

This second test facility has proved satisfactory from an operational standpoint. The use of the standard corrosion sample barrels (1.1.c) in loop BS eliminated plugging by thorium oxide slurry which had occurred in the annular-flow sample barrels (1.1.d, 1.1.e) previously used in loop CS.

Since two of the corrosion specimen holders in loop BS are directly in series, having loop flow, and the third is in the return line which combines loop and pressurizer flow, the velocity range to which the corrosion specimens are exposed is adjusted by the use of specimen holders with different cross-sectional area flow channels. The bulk-flow rate of the system can be adjusted by orifice-type restrictor plates and/or varying the speed of the circulating pump by means of a variable frequency motor-generator set. Operation of the pump at reduced speeds has also helped reduce pump bearing wear, particularly when thorium oxide caking on the pump impeller caused dynamic unbalance.

After run BS-11, a capillary tube letdown sampling unit was installed in the loop to facilitate sampling of the slurry. The sample unit consists of about 30 ft of 0.10 in OD stainless steel tubing coiled in a water-cooled jacket and arranged so that the tubing can be flushed with condensate before and after sampling to minimize the possibility of plugging and to insure more representative loop slurry samples. Appropriate valving and the

pressure drop through the capillary tubing allow slurry samples to be taken from the loop at operating temperature and pressure. This device has proved satisfactory in removing slurry samples from the loop. This type sampling unit has been previously used on the 200A pump loop by the Engineering Research Section.

100A Loop CS: Several changes were made to 100A pump loop CS during the period covered by this report. Fig. 1.1.1 is a schematic drawing of loop CS, and Fig. 1.1.2 is a photograph of the loop.

The three parallel, vertical, annular-flow sample barrels previously used (1.1.d, 1.1.e) have been replaced with three parallel, horizontal, standard 30-in sample barrels as used in solution corrosion loops. (1.1.c) The former arrangement was found to be unsatisfactory because of slurry buildup in the narrow (1/4-in.) flow annulus between the corrosion specimen holder and barrel wall. This flow annulus is eliminated in the standard solution corrosion sample barrels. The new barrel arrangement in loop CS allows corrosion specimen holders to be used in parallel and series simultaneously, since each 30-in. sample barrel can accommodate two 15-in. specimen holders.

Three different bulk-flow rates can be maintained through the three parallel sample barrels, and relative velocity through each specimen holder can be adjusted by changes in the cross-sectional areas of the flow channels of the specimen holders. However, bulk-flow rates and velocities in the three parallel units are interdependent and require adjusting by means of appropriate restrictors to obtain the desired combinations of velocities. Flow rates in loop CS can also be controlled by changing the pump speed with a variable frequency motor generator set, as with loop BS, but CS loop pump has not yet been operated at lower speeds.

Fig. 1.1.1 Schematic Drawing of Loop CS

Fig. 1.1.2 Photograph of Loop CS Showing Venturi and Sample Barrel Arrangement.

Another component added to loop CS was a calibrated venturi installed in the pump suction line as an aid in monitoring the density and/or changes in flow rate of the circulating thorium oxide slurry. A comparison of pump amperes and venturi differential pressure with thorium oxide slurry concentration for run CS-34 is shown in Fig. 1.1.3. Load concentration was calculated from the amount of thorium oxide added to the loop, while sample concentration was obtained by analysis of samples taken from the loop at the operating times indicated. Where two or more samples were taken at the same time, the individual concentrations obtained are shown. However, a sample average value was used in constructing the graph. With the initial addition of thorium oxide to the loop at hour 64, pump amperes and venturi differential pressure both increased, but not proportionally, either to each other or to the calculated slurry density increase. At hour 280, water was added to the system, but the expected decrease in pump amperes and venturi differential pressure was not observed. At hour 304 (slurry circulation hour 240), more thorium oxide slurry was added. Pump amperes and differential pressure readings both increased as expected with increased thorium oxide slurry concentration. Although pump amperes remained fairly constant from hour 304 until the end of the run, venturi differential pressure generally followed the decrease in thorium oxide slurry concentration in the loop as obtained from samples removed.

When the pump was disassembled at the end of the run, a break about 1-in. long was found on the cylindrical surface of the stainless steel pump rotor can (Fig. 1.1.4). This break could account for the relatively large increase in pump amperes which occurred at hour 290. Another indication of the break in the pump rotor can is that after hour 290, the ratio of $\text{Fe}/(\text{Fe} + \text{Cr} + \text{Ni})$ corrosion products in the thorium oxide slurry increased more than could be expected from the corrosion of the 347 stainless steel loop

UNCLASSIFIED
ORNL-LR-DWG 21458

Fig. 1.1.3 Comparison of Pump Amperes and Venturi Differential Pressure with Thorium Oxide Concentration for Run CS-34.

Fig. 1.1.4 Break in 100A Pump Rotor Can Found After Run CS-34.

components and corrosion specimens exposed to the slurry. (The ratio of Fe/(Fe + Cr + Ni) for type 347 stainless steel is 0.7). The increased Fe concentration in the corrosion products probably was caused by the corrosion of the iron rotor core through the break in the rotor can, although there is no direct means of verifying this assumption. The rotor can failure appeared to be mechanical, and not caused by chemical or corrosive action.

The venturi pressure taps are connected directly to an 800-in. d/P cell which, in turn, transmits the pressure differential to a gauge on the panel board. The venturi system was operated continuously, but readings were taken only every 4 hours.

In the event the venturi pressure taps should become plugged with slurry, condensate can be routed through the taps by means of the pulsafeder to remove the obstruction. Intermittent flushing with condensate, however, kept the lines free of slurry during run CS-34. The system was operated for 500 hours on slurry without plugging the 1/4 in. OD x 0.35 in. wall pressure lines.

A letdown capillary sampling device, identical to the one installed in loop BS, was also incorporated in loop CS.

1.2 100A Pump Loops Tests. (by S. A. Reed)

Five slurry corrosion test runs were made in loop BS, and one in loop CS, during the quarter. Four of the tests in loop BS were made to complete a study of the effects of sulfate on the corrosivity of oxygenated aqueous thorium slurries at 300°C. The series of tests employed slurries at a charge concentration of approximately 500 g of thorium per kilogram of water to which were added ~ 3000 ppm sulfate (based on thorium) as sulfuric acid and thorium sulfate. An 800°C-calcined thorium, Batch LO-14, prepared at ORNL was used. The corrosivity of the slurries did not appear to be much changed

by the addition of 3000 ppm sulfate (based on thoria); however, the additives were observed to markedly affect the formation of slurry spheroids during circulation.

In the fifth test in loop BS, which circulated an ORNL-produced, 650°C-calcined thoria at an average concentration of 478 g of thorium per kilogram of water at 300°C with oxygen, the thorium oxide was degraded to an average size of 0.5 μ with no spheroid formation. Corrosion rates closely paralleled those obtained with slurries prepared from 800°C-calcined thoria.

The run in loop CS circulated slurry prepared from Batch LO-2A, 800°C-calcined thoria, at 300°C for 501 hr at an average concentration of 516 g of thorium per kilogram of water. Corrosion rates were not excessive. However, pronounced erosive attack occurred in the circulating pump.

Corrosion data of pin and coupon type specimens exposed in these runs are presented in Table 1.8.

1.3 Tests with Sulfate Additives. (by S. A. Reed)

In the four loop tests made to investigate the influence of sulfate ion on the corrosivity of thoria slurries, Batch LO-14 thorium oxide was used. The unique spheroid-forming character of this thoria preparation and a summary of the loop test, Run BS-8, which circulated it with sodium sulfate as an additive, have been described previously. (1.3.a, 1.3.b)

For each test of the series, loop BS was operated isothermally at 300°C with oxygen overpressure. A by-pass mixing line leading from the pump discharge line to the loop pressurizer was used in an attempt to maintain a uniform slurry concentration throughout the loop system. Flow through the mixing line was arbitrarily maintained at 12 to 13 gpm. As a result of favorable experience

in previous tests with LO-14 slurry, the 100A centrifugal pump was operated at half of normal rotational speed, i.e., at 1800 rpm, on 30-cycle current provided by a motor generator set, to minimize bearing wear if caking occurred. The pump was equipped with Graphitar bearings and Stellite journals.

In each run, the loop was loaded with three sets of pin-type corrosion specimens to flow velocities, measured with water at room temperature, of approximately 10, 20, and 30 fps. When satisfactory operation on oxygenated water at 300°C was attained, slurry was charged to the system as a single batch addition. Each test was scheduled to run 300 hr.

Runs BS-9 and BS-10: In these two tests, the required quantity of sulfuric acid, to provide ~ 3000 ppm $\text{SO}_4^{=}$ (based on thorium) was to be injected into the loop immediately before adding the thorium. Run BS-9 was so charged to a concentration of 928 g Th/kg H_2O using Batch LO-14 thorium. The average circulating concentration for the run was 745 g Th/kg H_2O by chemical analysis. Heavy caking on the pump impeller and in the pump suction line necessitated stopping the run after 27 hours circulation. During this period the cast type 431 stainless steel pump impeller lost 4 grams.

Run BS-10 was loaded with Batch LO-14 thorium to a concentration of 562 g Th/kg H_2O , including a small thorium carry-over from the previous run. The average circulating concentration was 586 g Th/kg H_2O . On the basis of initial chemical analyses, later indicated to have been in error, additional sulfuric acid appeared to have been required to maintain the desired sulfate concentration, and 21 ml H_2SO_4 (total 9000 ppm SO_4/ThO_2) were added to the loop at hour 94, above the original 25 ml (4900 ppm SO_4/ThO_2) added at startup. The pH fell from 7.2 to 3.1. Because of erratic pump purge water flow, the run was shut down at hour 123.1. The cast

431 stainless steel pump impeller lost 128 g. Differential corrosion rates, which increased rapidly after the second acid addition, indicate that the majority of the attack occurred after this time.

Slurry spheroids 15 to 40 μ were formed during both runs BS-9 and BS-10.

Sample, loop inventory, and corrosion data for runs BS-9 and BS-10 are given in Tables 1.1 and 1.2, respectively, and specimen corrosion data in Table 1.8.

Run BS-11: In this run, a 300-hr test, wide variations in circulating concentrations occurred, spheroid growth was pronounced, and hard, tenacious deposits of thorium were formed on the pump impeller and in the pump suction line. In this test, the sulfuric acid was mixed with the slurry before charging the system. While charging the slurry, samples withdrawn from the loop main stream after 1.4 hr of circulation contained numerous 7-to-20- μ spheroids, as observed using the microscope. After 100 hr of circulation, 95% of the slurry was composed of 40 to > 120 μ spheroids. The run was shut down on schedule after 300 hours. Sample, loop inventory, and corrosion data for run BS-11 are given in Table 1.3, and specimen corrosion data in Table 1.8.

Run BS-12: Similar phenomena occurred in run BS-12, a 113-hr test in which thorium sulfate was added to the slurry charged to the system. The average slurry concentration, by chemical analysis, for the 113-hr period was 988 g of thorium per kilogram of water. Slurry spheroids > 120 μ formed during circulation. Sample, loop inventory and corrosion data for run BS-12 are given in Table 1.4, and specimen corrosion data in Table 1.8.

As in tests reported previously, ^(1.3.b) the marked increases in circulating concentration in run BS-11 and BS-12 that occurred as the spheroids became larger, resulted from a stripping action in the vertical loop pressurizer. A direct correlation was observed

TABLE 1.1

Summary of Sample Data, Loop Inventory and Corrosion Rate, Run BS-9

Slurry Circulating Hours	0	2.5	4.5	22.1	22.1	26.3	Solids From Impeller After Run
							Avg.
Th, g/kg H ₂ O	(205)	604	890	691	757	783	745
Analysis							
Th, mg/g slurry	(180) ^a	347	442	387	407	415	680
SO ₄ , mg/g slurry	0.59	0.46	0.33	0.30	0.35	0.27	0.73
Fe, ppm	----	46	56	357	350	270	290
Cr, ppm	----	7	< 5	26	29	31	25
Ni, ppm	----	< 5	6	12	13	13	13
Co, ppm	----	40	< 5	15	< 5	16	19
Ti, ppm	----	< 2	< 2	< 2	< 2 ^b	< 2 ^b	< 2
pH	----	8.8	8.7	8.7	11.2 ^b	11.4 ^b	----
Aqueous Phase							
Fe, µg/ml	----	< 1	< 1	< 1	< 1	< 1	
Cr, µg/ml	----	< 1	< 1	< 1	< 1	< 1	
Ni, µg/ml	----	< 1	< 1	< 1	< 1	< 1	
Co, µg/ml	----	< 1	< 2	< 1	< 1	< 1	
Ti, µg/ml	----	< 2	< 2	< 2	< 2	< 2	
SO ₄ , µg/ml	----	< 5	< 5	< 5	< 5	< 5	
pH	----	8.2	8.4	8.1	8.5	8.9	
Gas Analysis (Separate Sample)							
O ₂ , ppm	----	871	----	----	----	534	
CO ₂ , ppm	----	40	----	----	----	37	
Sedimentation Particle Size ^c							
Avg. Diameter, µ	----	1.7	2.0 ^e	2.6	2.8	3.0	4.5
> 7 µ, wt %	----	9	27	20	29	31	25
3-7 µ	----	25	16	28	19	18	46
1-3	----	32	22	26	25	21	22
< 1	----	34	35	26	27	30	7
Inventory							
ThO ₂ , kg	4.1 ^a	12.3	15.1	15.0	14.9	14.9	
H ₂ O, kg	15.7	14.8	14.3	14.2	14.1	14.0	
Non-circulating ThO ₂ , kg		2.2	0.7	3.9	2.7	2.4	
Total Fe Cr Ni, g		1.46	1.82	11.74	11.55	9.42	
Diff'l Corros. Rate, mpy		27	7	25	----	----	Overall Rate 16 mpy

a. ThO₂ remaining in systems from previous tests.

b. Value appears unreasonable. May have been involved in sample mixup.

c. By gravitational sedimentation in 0.005 M Na₄P₂O₇ using neutron activation analysis.

d. Based on water inventory.

e. Separate sample.

TABLE 1.2

Summary of Sample Data, Loop Inventory and Corrosion Rate, Run BS-10

Slurry Circulating Hours	0	1.5	4.1	21.4	45.3	94.0	99.0	122.9	
Th, g kg H ₂ O	Load:		702	484	420	655	570 ^e	685(avg 586)	
Analysis									
Th, mg/g slurry	(28) ^{a,h}	b	390	311	284	388	358*	434	
SO ₄ , mg/g slurry	0.75	----	0.74*	0.69*	0.56*	0.94	4.60*	1.10*	
			0.40**	0.43**					
Fe, ppm	156	----	780	685	650*	1070	1365	10,650	
Cr, ppm	< 5	----	38	38	55	110	104*	750	
Ni, ppm	10	----	37	36	37	60	51	218	
Co, ppm	< 20	----	6	< 5	7	7	*	7	
Ti, ppm	----	----	< 2	< 2	25*	4	*	< 2	
pH	----	----	8.4	7.9	7.8	8.1	3.1	4.9	
Aqueous Phase									
Fe, µg/ml	----	----	< 1	< 1	< 1	< 1	1	1051	
Cr, µg/ml	----	----	< 1	< 1	< 1	< 1	< 1	1	
Ni, µg/ml	----	----	< 1	< 1	< 1	< 1	*	1	
Co, µg/ml	----	----	< 1	< 1	< 1	< 1	*	< 1	
Ti, µg/ml	----	----	< 2	< 1	< 2	< 2	*	< 2	
SO ₄ , µg/ml	----	----	8	8	8	26	485*	1350*	
pH	----	----	7.4	7.9	6.1	7.2	3.3	4.6	
Gas Analysis (Separate Sample)									
O ₂ , ppm	74	----	----	238	----	----	----	----	
CO ₂ , ppm	< 10	----	----	10	----	----	----	----	
Surface Area(N ₂ ads's) m ² /g		22.9	23.5	24.1	24.2	28.0	28.9	27.0	
Sedimentation Particle Size ^{c, f}									
Average Diameter, µ	----	1.5	1.4	1.3	1.2	8.5	13	11.5	
> 7 µ wt %	----	6	6	3	4	58	64	72	
3-7 µ	----	21	22	21	19	11	7	6	
1-3 µ	----	35	33	32	32	11	5	4	
< 1, or ^d 0.7 - 1 µ	----	17	39	44	45	29	23	18	
0.3-0.7	----	12	----	----	----	----	----	----	
0.1-0.3	----	7	----	----	----	----	----	----	
< 0.1	----	2	----	----	----	----	----	----	
Inventory									
ThO ₂ , kg	0.5	9.4	9.4	9.3	9.2	9.2	9.1	9.0	
H ₂ O, kg	15.7	14.7	14.7	14.6	14.5	14.4	14.3	14.2	
Total ^g Fe, Cr, Ni, g	2.61	----	18.20	20.18	21.66	25.96	35.00	211.32	Overall
Diff'l Corros. Rate, mpy	----	----	167	5	3	4	79	324	Rate
									74 mpy

* Value questionable: slurry samples confused with others during analysis.

** Values originally reported.

a. Analysis and inventory based on water circulated before ThO₂ addition.

b. Sample lost during analysis.

c. By gravitational sedimentation in 0.005 M Na₃P₂O₇ using neutron activation analysis.

d. By centrifugation method described in ORNL CF-57-11-6.

e. Additional H₂SO₄ charged to system before sample was taken.

f. Photographs shown in Figure 1.

g. Based on ThO₂ inventory.h. ThO₂ remaining in system from previous test.

UNCLASSIFIED

TABLE 1.3

Summary of Sample Data, Loop Inventory and Corrosion, Run BS-11

Load Conc.: 442 g Th/kg H₂O

									Solids From Pump Suction Nozzle (Density 6.12 (c))	Solids From Pump Impeller (Density 6.56 (c))	Solids From Return Line Sample Barrel (Density 5.87 (c))
Slurry Circulation Time (hr)	3.2	21.3	93.0	166.7	189.1	212.9	260.9	300.1			
Slurry Concentration g Th/kg H ₂ O	640	593	1005		727	866	627	621			
mg Th/g slurry	370	354	469	No	398	436	366	364			
Fe, μ g/g slurry	35	96	256	Analysis	230	299	154	152			
Cr, μ g/g slurry	8	37	77		67	97	57	106			
Ni, μ g/g slurry	<5	<5	15	Sample Bomb Leaked	13	22	13	<5			
Ti, μ g/g slurry	<20	<20	30		<20	<20	20	<20	140	140	140
Al, μ g/g slurry	<20	<20	35		40	40	45	40	20	20	45
SO ₄ , mg/g slurry	0.83	0.81	0.94		0.64	0.84	0.80	0.79	1.31	1.22	1.12
pH of slurry	6.6	6.6	5.7		6.3	5.2	5.9	7.6			
Analysis of Supernate											
SO ₄ , ppm	<5	<5	<5		<5	<5	8	9			
pH of supernate	6.5	6.3	6.1		6.4	6.2	6.4	5.9			
Gas Analysis (Separate Sample)											
O ₂ , ppm	386		505, 583				1124				
CO ₂ , ppm	33		240, 1202				42				
Sedimentation Partical Size (μ) (a)											
> 7 μ (Wt %)	7	28	68						15	14	22
3-7 μ (Wt %)	21	18	8						22	19	24
1-3 μ (Wt %)	33	22	7						34	24	29
< 1 μ (Wt %)	39	32	17						29	43	25
Average Diameter (μ)	1.4	2.6	9.0						1.9	1.6	2.4
> 120 μ (Wt %) (b)				2	1	1	0	0			
33-720 μ (Wt %)				8	6	4	2	1			
10-33 μ (Wt %)				86	83	83	86	84			
< 10 μ (Wt %)				4	10	12	12	15			
Average Diameter (μ)				23	20	20	20	18.5			
Surface Area, m ² /g	23.8	26.9	27.8	26.7	26.5	25.2	25.1	24.1	21.4	22.4	21.4
System Inventory											
H ₂ O, Kg	14.9	14.8	14.7	1	14.1	15.5	15.4	15.1			
Th, Kg	6.5	6.5	6.4		6.2	6.1	6.1	5.9			
Fe, Cr, Ni, grams produced (d)	0.76	2.45	4.83		5.07	6.19	4.06	4.65			
Differential System Corrosion Rate, mpy	10.3	4.0	1.4		0.1	2.0	(-1.9)	0.6			
Overall Corrosion Rate, mpy						1.3		0.7			

(a) By gravitational sedimentation in 0.005 M Na₄P₂O₇ using neutron activation analysis.(b) By gravitational sedimentation in 19.53% cp glycerine - H₂O, 0.005 M Na₄P₂O₇ using neutron activation analysis.(c) By pycnometer using CCl₄.

(d) These values apparently unduly low, as impeller lost 19 grams.

UNCLASSIFIED

Table 1.4

SUMMARY OF SAMPLE DATA, LOOP INVENTORY AND CORROSION RATE, RUN BS-12

Slurry Circ. Time (hr)	0	3.2 ^b	4.1	14.5	14.5	34.1	34.1	38.0	38.0	106.1	106.1	107.5 ^e	107.5 ^e	108.2 ^f	108.2 ^f	108.8 ^g	108.8 ^g	112.5 ^g	112.5 ^g
Slurry Concentration	Charge																		
g Th/kg H ₂ O	583 (calc. load)	551	(630)	470	(552)	889	(4100)	562	(2940)	897	(3280)	(2570)	549	496	(2980)	354	(2309)	261	(2210)
(from chem. analysis)	average of bomb samples, 558; average of let-down samples, 2397.																		
mg Th/g slurry (chemical analysis)		344 ^d	367 ^c	306 ^d	345 ^c	442 ^d	724 ^c	343 ^d	677 ^c	444 ^d	693 ^c	655 ^c	331 ^d	318 ^d	679 ^c	252 ^d	637 ^c	201 ^d	629 ^c
SO ₄ ⁼ , mg/g slurry		0.28	0.17	< 0.10	0.23	0.20	0.32	0.15	0.26	0.22	0.28	0.29	0.23	0.15	0.25	0.11	0.32	< 0.10	0.29
Fe		63	34	64	84	176	271	133	272	183	293	288	139	138	285	109	288	99	303
Cr		26	11	19	18	44	64	31	72	39	85	23	23	23	91	30	74	17	81
Ni		< 5	< 5	6	< 5	8	14	7	14	< 5	11	12	< 5	< 5	17	7	8	< 5	14
Ti		< 20	< 20	< 20	< 20	40	60	30	55	30	30	< 20	< 20	< 20	30	< 20	50	< 20	25
pH of slurry		7.4	6.8	7.4	6.8	7.7	7.8	6.4	7.4	7.4	8.2	8.5	8.2	7.8	7.4	6.5	7.7	6.3	7.8
Analysis of Supernate																			
SO ₄ , ppm		< 5	5	< 5	5	< 5	-	< 5	-	< 2	-	< 5	5	< 5	< 5	< 5	< 5	< 5	< 5
pH of supernate		6.3	6.5	6.9	6.8	6.6	-	4.9	-	7.0	-	7.1	7.4	6.6	8.2	7.3	6.8	7.0	6.7
Gas Analysis																			
O ₂ , ppm	1245																		
CO ₂ , ppm	168																		
Sedimentation Particle Size (μ) ^a																			
> 119 μ (wt. %)		1	0	0	1	1	4	3	7	7	4	3	-	3	2	4	2	4	4
33-119 μ (wt. %)		4	1	1	3	15	57	43	57	44	42	29	-	40	13	40	19	32	38
10-33 μ (wt. %)		38	13	6	14	37	33	33	29	42	43	45	-	50	47	50	46	54	52
< 10 μ (wt. %)		57	86	93	82	47	5	21	7	7	11	23	-	7	38	6	33	10	6
Average Diameter, μ		6.5	3.0	2.0	2.5	12	35.7	32.5	44	35.6	32	27	-	31	15	32	23.7	29	31
Nitrogen Surface Area, mg ² /g		18.5	24.5	23.5	24.3	24.9	28.2	28.7	27.9	28.1	26.3	26.7	26.7	-	26.0	26.5	26.1	26.5	26.5
Loop Inventory																			
H ₂ O, kg		14.7	14.6	15.2	15.1	14.9	14.7	14.6	14.5	14.4	14.2	14.1	13.8	13.8	13.7	13.5	13.4	13.2	12.9
Th, kg		8.6	8.5	8.4	8.4	8.3	8.2	7.5	7.4	6.9	6.9	6.3	5.7	5.7	5.6	5.2	5.1	4.8	4.3
Fe, Cr, Ni, (i) grams (cumulative)		-	2.29	1.06	2.21	2.52	4.31	4.19	4.21	4.43	4.23	4.67	4.81	4.26	4.36	4.76	4.76	4.79	5.02
Corrosion Rate, Differential, mpy		(j)	11.2	(k)	5.4	(k)	4.2	(k)	0.8	(k)	0.08	(k)	2.5	(k)	1.7	(k)	15.7	(k)	1.3: Overall 1.9

(a) By gravitational sedimentation in 19.53 cp glycerin-water, 0.005 M solution of Na₄P₂O₇ using neutron activation analysis.

(b) Sample taken during slurry addition.

(c) Samples taken from let-down sampler. (g Th/kg H₂O)

(d) Regular bomb samples.

(e) Pump to 40 cycles at this time.

(f) Pump to 50 cycles.

(g) Pump to 60 cycles.

(h) By gravitational sedimentation in 0.005 M Na₄P₂O₇ solution using neutron activation analysis.

(i) Based on Th inventory.

(j) Value included in calculation of following period.

(k) Average value of Fe, Cr, Ni for this and next sample used in computation for period.

in these runs between the formation and growth of spheroids, the fluctuating concentration, and the corresponding drop or rise of the slurry miniscus in the pressurizer.

Data are presented in Table 1.5 which compare the effects of circulation on LO-14 slurries with and without sulfate additives. For purposes of comparison, data from several earlier runs have also been included in the tabulation.

Similar trends occurred in all tests. A gradual increase in spheroid size occurred during about the first 50 hr of circulation after which time, little measurable growth took place. After approximately 100 hr of circulation, the average size of the spheroids, measured by sedimentation, appeared to decrease. Actually, by microscopic examination, numerous large spheroids were still present, but many of them appeared to have been fractured or malformed during the latter stages of pumping. These changes are shown in the photomicrographs in Fig. 1.1.5^(a-f).

Although the sulfate additives appeared to have little effect on spheroid growth rate in these tests, a marked difference in ultimate spheroid size was observed. Without sulfate additives, a maximum spheroid size of 20 μ was observed. In the run with sodium sulfate, spheroids of 5 to 16 μ were formed during 300 hr of circulation. Throughout the test, both sodium and sulfate ions were found to be in almost constant ratio between the slurry solids and supernatant liquid. When sulfuric acid was used, spheroids 7 to 100 μ were present after 100 hr of circulation, but no apparent growth took place during the following 200 hr. In the test with thorium sulfate as an additive, spheroids ranged from 40 to > 120 μ after only 30 hr of circulation. No further growth occurred during the remaining 83 hr of the test. In the latter two tests, the sulfate ions were found, by chemical analysis, to be associated solely with the slurry solids.

Nitrogen adsorption surface area data, included in Table 1.5 are also seen to follow a general trend with spheroid growth. While the

TABLE 1.5

Comparative Circulation and Corrosion Effects of Batch LO-14 and Batch LO-2 Slurries at 300°C

	Run BS-7 (ThO ₂ Batch LO-14)	Run BS-8 (ThO ₂ Batch LO-14)	Run BS-11 (ThO ₂ Batch LO-14)	Run BS-12 (ThO ₂ Batch LO-14)	Run CS-27 (ThO ₂ Batch LO-2)
Duration of Test, hr	194.2	334.8	300.3	113.1	300.9
Average Circulating Concentration (g Th/kg H ₂ O)	635	484	725	558	348
Atmosphere	O ₂ (1000 ppm)	O ₂ (1000 ppm)	O ₂ (1000 ppm)	O ₂ (1000 ppm)	O ₂ (1000 ppm)
Additive		Na ₂ SO ₄ , 0.02 m	H ₂ SO ₄ , 0.02 m	Th(SO ₄) ₃ 0.001 m	
pH of slurry	8	7.5	6	7	7

Defilmed Corrosion Rates, mpy, at Indicated Flow Velocity

	10 fps	20 fps	30 fps	10 fps	20 fps	30 fps	10 fps	20 fps	30 fps	10 fps	20 fps	30 fps	10 fps	20 fps	30 fps
Austenitic SS	2	5	5	2	9	10	4	4.5	4.5	3	5	5	0.9	2	7
Titanium and Alloys	1.6	0.9	4.5	5	7	9	0.3	0.7	0.8	0.1	1.5	1.2	0.2	2	6
Zircaloy-2	WG	1.0	1.5	0.2	1.1	1.4	0.3	0.8	1.4	0.0	0.0	0.6	0.0	0.1	2
Gold	1.3	1.6	2.5	0.7	1.8	2.1	0.1	1.2	2.5	0.1	2.5	3.0	0.05	0.10	0.60
Average Loop Corrosion Rate, mpy		0.6			0.9			3.6			1.9			1.5	

Nitrogen Adsorption Surface Area and Sedimentation Particle Size

Approximate Circulation Time (hr)	Surface Area (m ² /g)	Particle Size (wt %)			Surface Area (m ² /g)	Particle Size (wt %)			Surface Area (m ² /g)	Particle Size (wt %)			Surface Area (m ² /g)	Particle Size (wt %)			Surface Area (m ² /g)	Particle Size (wt %)		
		>100	10-50	<10		>100	10-50	<10		>100	10-50	<10		>100	10-50	<10		>100	10-50	<10
0	18.5	0	0	100	18.5	0	0	100	18.5	0	0	100	18.5	0	0	100	16.5	0	1	99
5	22.3	0	6	94	22.0	0	9	91	23.8	0	7	93	23.5	1	13	86	18.7	-	-	-
20	24.2	0	39	61	25.9	0	11	89	26.9	0	28	72	28.7	16	37	47	21.9	No spheroids. Degraded to average 0.7		
50	-	-	-	-	26.0	0	6	94	-	-	-	-	28.9	64	29	7	22.1			
100	26.7	0	36	64	27.5	0	85	15	26.7	0	68	32	26.7	51	42	7	22.9			
150	27.7	0	44	56	26.1	0	1	99	26.7	10	86	4	26.5	42	52	6	22.4			
200	26.5	0	43	57	26.3	0	3	97	25.2	7	83	4	-	-	-	-	22.5			
300	-	-	-	-	26.4	0	1	99	25.1	1	84	15	-	-	-	-	22.3

Fig. 1.1.5 (a) LO-14 Slurry Before Circulation. 400X

Fig. 1.1.5 (b) LO-14 Slurry After 95 hr Circulation. 400X

Fig. 1.1.5 (c) LO-14 Slurry After 165 hr Circulation. 400X

Fig. 1.1.5 (d) LO-14 Slurry After 189 hr Circulation. 400X

Fig. 1.1.5 (e) LO-14 Slurry After 260 hr Circulation. 400X

Fig. 1.1.5 (f) LO-14 Slurry After 300 hr Circulation. 400X

significance of the surface area measurements is not clear, the changes suggest that the spheroids or their surfaces are quite porous.

Comparative corrosion rates from loop runs with LO-14 slurry are shown in Table 1.5. Also included in the table are attack data obtained in a previously reported^(1.3.c) run which circulated slurry prepared from Batch LO-2 thorium oxide where no spheroid formation was observed. There are no differences in the technique of preparation or firing of these batches. Neither the formation of spheroids nor the presence of sulfate additives appeared to significantly affect the corrosivity of the slurries.

1.4 Circulation of 650°C-Calced Thoria Slurry (by S. A. Reed)

Run BS-13: The slurry circulated in Run BS-13 was made from a new thorium oxide preparation designated as Batch LO-18. The material was produced at ORNL by precipitation of thorium nitrate with oxalic acid at 70°C and final calcination at 650° for 24 hr.

The condition of operation and system flow velocities were the same as those described for runs BS-9 through BS-12. While operating at 300°C, sufficient slurry was charged to the system to provide a calculated circulating concentration of 636 g of thorium per kilogram of water. No additions, other than gaseous oxygen, were made to the loop. The loop was operated routinely for a period of 304.6 hr at an average concentration of 478 g of thorium per kilogram of water.

The run was terminated when a gasket ruptured in the high-pressure rotameter connected in the purge water feed line to the circulating pump. The system, at 300°C, blew down in approximately 2 min from 1500 psi to atmospheric pressure.

After standing at room temperature for about 24 hr, the system was recharged with water. Without heating, the circulating pump was run for 30 min to resuspend the solids. The loop was then drained and rinsed, using the pump, in the usual manner. During disassembly, no deposits or accumulation of thoria were found in the system.

The rate of attrition of the LO-18, 650°C-calcined thorium oxide was somewhat more rapid than other oxide preparations, fired at 800°C, which have been circulated in loop BS. The average particle size of the LO-18 material, measured by sedimentation analysis, before circulation in run BS-13, was 3.3 μ . After 2.9 hr of circulation at 300°C, the average size of the particles was 0.8 μ . After 22 hr of circulation, the average size had diminished to 0.6 μ , and after 70 hr of circulation, to 0.5 μ . No further degradation was detected after that time. No spheroids formed during the test. Sample, loop inventory, and corrosion data for run BS-13 are given in Table 1.6, and specimen corrosion data in Table 1.8.

Corrosion rates observed in run BS-13 with LO-18 thorium oxide slurry were moderate. Also, there was a negligible velocity effect between 20 and 30 fps. Defilmed rates at 10, 20, and 30 fps, respectively, were (in mpy): austenitic stainless steel, 4, 5, and 6; titanium and alloys, 2, 3, and 3.5; Zircaloy-2, 0.8, 2.0, and 3.2; and gold, 0.05, 0.4, and 0.4.

1.5 Wear of Pump Components (by S. A. Reed)

With the exception of the cast type 431 steel impeller used in loop BS, the attack of pump components during runs BS-9 through BS-13 was not excessive. The front Graphitar bearing, replaced after run BS-12, had worn only 0.0034 in. after 1169.7 hr service in slurry and 410.2 hr service in oxygenated water. There was no detectable wear on the rear Graphitar bearing after run BS-13. The bearing, which is still in use, had accumulated 1474.3 hr service in slurry and 441.3 hr service in oxygenated water when run BS-13 was completed. The same titanium wear rings were used in all tests through run BS-13.

The cast type 431 steel impeller lost a total of 155 g through run BS-12, 128 g of which was lost during a 123 hr period in run BS-10 with sulfuric acid as additive. During run BS-10, most of the oxygen in the system was either consumed or lost through leakage.

TABLE 116

Summary of Sample Data, Loop Inventory and Corrosion Rate, Run BS-13

Slurry Hr	0	2.9	2.9	2.9	22.2	22.2	69.7	69.7	141.8	141.8	189.5	189.5	237.4	237.4	
Sample Valve No.	Charge	1 ^a	3 ^b	5 ^c	3 ^b	5 ^c	3 ^b	4 ^d	3 ^b	4 ^d	3 ^b	4 ^d	3 ^b	4 ^d	
g Th/kg H ₂ O (anal)	-	531	616	535	524	448	536	458	523	373	555	184	485	446	Avg 478
ANAL: Th, mg/g	514	331	362	333	328	297	332	302	327	262	340	152	312	296	
Fe, /g	7	< 5	< 5	-	67	69	147	127	256	125	274	110	265	260	
Cr, /g	< 5	6	6	-	19	14	26	45	58	53	70	40	64	86	
Ni, /g	< 5	< 5	< 5	-	8	8	24	23	40	28	38	17	39	37	
Ti, /g	< 5	< 20	< 20	-	< 20	< 20	< 20	< 20	< 20	< 20	60	< 20	55	40	
pH	-	7.2	7.2	-	7.0	6.1	5.0	6.0	5.5	6.0	5.6	4.7	5.2	5.0	
SO ₄	< 5	< 100	< 100	-	< 100	< 100	< 100	< 100	< 100	< 100	< 100	< 100	< 100	< 100	
Supernate: pH	-	7.2	7.2	-	7.7	5.8	5.7	5.9	5.7	5.6	7.0	5.6	6.5	5.7	
Sp. Cond. x 10 ⁶	-	44	36	-	77	150	50	67	80	72	97	70	81	73	
Gas: O ₂ , /ml	-	562	-	-	-	-	-	-	-	-	-	-	-	-	
CO ₂ , /ml	-	< 10	-	-	-	-	-	-	-	-	-	-	-	-	
Part. Size, d, μ	3.3	0.8	-	-	0.6	-	0.5	-	-	0.5	0.5	-	0.5	-	
Wt.%, > 7 μ	31	6	-	-	2	-	1	-	-	1	1	-	2	-	
3-7	20	6	-	-	4	-	2	-	-	1	2	-	2	-	
1-3	30	27	-	-	19	-	17	-	-	17	16	-	12	-	
< 1	19	61	-	-	75	-	80	-	-	81	81	-	84	-	
Surface Area, m ² /g	31.6	39.2	-	-	35.0	-	34.3	-	-	30.9	30.8	-	29.4	-	
Crystallite Size, Å	147	142	-	-	154	-	148	-	-	157	166	-	173	-	
INV: H ₂ O, kg	14.6	14.4	14.3	14.1	14.0	13.9	13.7	13.6	13.4	13.3	13.2	13.1	12.9	12.8	
Th, kg	9.3	9.2	9.1	9.0	8.9	8.9	8.8	8.8	8.7	8.6	8.5	8.5	8.4	8.4	
Tot. Fe, Cr, Ni formed, g	-	0.041	0.026	-	2.213	2.496	4.615	4.944	8.070	6.275	8.715	8.597	8.921	9.879	
Increm., mpy (g)	-	-	0.5	-	-	5.2	-	2.2	-	1.4	-	1.3	-	0.7	Overall: 1.7

- (a) Pump Discharge: Letdown Sampler. Valve 1, Vertical line. Mechanical failure after first sample.
 (b) Pump Discharge: Letdown Sampler. Valve 3, Bottom of horizontal line.
 (c) Pressurizer above top heater: bomb sample.
 (d) Pressurizer between heaters: bomb sample.
 (e) By gravitational sedimentation in 0.005 m Na₄P₂O₇ using neutron activation analysis.
 (f) Sample bomb not filled.
 (g) Value uses average corrosion product level computed for samples taken at same time.

UNCLASSIFIED

Page 31

UNCLASSIFIED

1.6 Circulation Test in Revised Loop-CS (by S. A. Reed)

After the revisions to loop CS were completed (see Section 1.1 above), run CS-34, a 501 hr test at 300°C with oxygenated slurry at an average circulating concentration of 516 g of thorium per kilogram of water was made. The slurry charge for the test was prepared from Batch LO-2A thoria.

Bulk flows, measured on water of room temperature, through the three sample barrels were 6, 24, and 40 gpm, respectively. Flow through the by-pass mixing line was 13 gpm. Each barrel was loaded with two sets of corrosion specimens in holders with different apertures to provide flow velocities ranging from 6 to 52 fps. A total of 76 specimens was exposed in the three loop barrels.

In the loop pressurizer, six pin specimens were exposed to slurry at 0.1 fps, and four stress specimens were placed in the gas-vapor space above the mixing line.

After a 72 hr performance test at 300°C with oxygenated water, 10.576 kg of Batch LO-2A was charged to the loop to provide a calculated circulating slurry concentration of 572 g of thorium per kilogram of water. Thereafter, the loop was operated and sampled in a routine manner for 501.2 hr.

One additional charge of slurry containing 1.15 kg of LO-2A thoria and 1.89 kg of water was added to the system after approximately 230 hr of operation as make-up for the volume of slurry withdrawn as samples.

Fifteen samples were taken during the test, most of them using the new let-down sampler. The average concentration of the samples, by chemical analysis, was 516 g of thorium per kilogram of water. Each 80 ml sample was preceded by two flush samples, of equal volume, to purge the sampler and associated tubing. No difficulty was experienced with the sampler during the test.

When the run was completed, the system was cooled to room temperature, flushed with water, using the pump, and then disassembled for routine inspection.

There was only one area in the system where slurry had accumulated. A thin, hard deposit of thoria was found in the upstream header piping to the No. 3 sample barrel. Flow through this section was approximately 6 gpm (~ 1 ft/sec). Also, both specimen holders in the No. 3 barrel were blocked with a soft deposit of slurry.

Pronounced erosion occurred in the pump. Areas adjacent to each weld pad on the pump head and on the thermal barrier were eroded to depths ranging from 0.030 to 0.115 in. Similar localized attack was found in the opening to the pump suction line. The pump head is type 347 stainless steel; the thermal barrier is type 75A titanium.

The front hub of the titanium 75A impeller, shown in Fig. 1.1.6 was similarly eroded. Numerous pits 0.13 to 0.18 in. in depth were found.

All surfaces of the pump head and thermal barrier and the front surface of the impeller were highly polished. The back surface of the impeller, only slightly abraded, was covered with a thin corrosion film.

Sample, loop inventory and corrosion data for run CS-34 are given in Table 1.7, and specimen corrosion data in Table 1.8.

Differential corrosion rates for the run, calculated from chemical analysis of corrosion products in the slurry, began at 15 mils per year for the first few hours, and successively became smaller, decreasing to a level somewhat below 1 mpy. No pronounced subsequent rise was noted. Average loop corrosion rate for the run was 1.0 mpy. Excess iron, above the stainless steel ratio, rose steadily, reaching a level of about 5 grams after 250 hours and 14 grams after 500 hours. It is this iron which apparently came from within the cracked rotor can.

UNCLASSIFIED
PHOTO 28544

Fig. 1.1.6 Localized Attack on Type 75A Titanium Pump Impeller After Run CS-34.

UNCLASSIFIED

TABLE 1.7

Summary of Sample Data, Loop Inventory and Corrosion Rates, Run CS-34

Slurry Circulation Hr	0	4.7	4.7	27.4	27.4	70.4	70.4	73.7	166.3	166.3	220.4	238.5 ^a	243.5 ^b	334.4	430.5	501.0	
g Th/kg H ₂ O	(572)	560	589	467	491	458	467	488	453	602	537	543	528	598	496	456	Avg. 516
Analysis: Slurry:																	
Th, mg/g		342	354	306	316	301	305	314	299	357	333	336	330	356	317	300	
Fe %/g		80	107	273	264	405	440	475	570	680	680	740	600	890	1040	1120	
Cr %/g		27	32	62	62	83	87	90	94	112	138	120	123	114	115	134	
Ni %/g		9	11	40	36	64	81	65	93	104	104	88	86	149	155	174	
Al %/g		<20	<20	30	40	40	50	<20	<20	<20	<20	35	5-	<20	<20	<20	
pH		7.6	8.6	7.3	7.7	6.8	6.5	6.8	5.4	6.1	6.0	5.9	6.1	5.0	4.9	5.0	
Supernatant:																	
pH		6.7	7.8	6.2	7.8	6.7	6.6	6.9	6.5	6.6	6.3	6.1	6.4	6.3	6.2	5.7	
Specific Cond x 10 ⁶		53	60	53	60	50	66	53	40	50	47	45	49	51	49	51	
Gas Analysis (Separate Sample)																	
O ₂ , %/ml		925								690			1041			960	
CO ₂ , %/ml		<10								29			<10			198	
Particle Size: ^c																	
Wt % >7 μ	32	1		1		1		3		1	1	2	2	0	0	1	
3-7 μ	18	7		5		3		5		3	2	4	3	3	3	3	
1-3 μ	30	22		17		17		22		23	22	22	34	23	34	48	
0.5-1 μ	20	26		41		41		39		44	44	53	40	54	44	35	
<0.5 μ	-	44		36		38		31		29	29	22	21	20	19	13	
Avg. Dia., μ	2.3	0.63		0.69		0.67		0.72		0.68	0.68	0.65	0.67	0.67	0.75	1.0	
Surface Area (N ₂), m ² /g	16.5	20.0		20.8		20.1		20.2		19.2	19.2	18.5	18.8	19.8	18.5	17.5	
Inventory:																	
H ₂ O, kg	16.2	16.1	16.0	15.8	15.7	15.6	15.4	15.3	15.1	15.0	14.8	15.6	15.6	15.4	15.3	15.1	
Th, kg	9.3	9.2	9.1	9.1	9.0	9.0	8.9	8.8	8.7	8.7	8.5	8.4	9.4	9.2	9.1	9.0	
Non Circ Th, kg	-	0.2	-	1.7	1.3	1.9	1.7	1.3	1.9	-	0.6	-	1.2	0	1.5	2.1	
Cr, Ni formed, g	-	0.94		1.13	2.84	2.70	3.92	4.40	4.03	4.76	4.88	5.67	4.85	4.90	5.84	6.79	8.00
Excess Fe, g	-	-	0.30	1.26	1.25	2.07	1.79	3.29	4.01	4.66	3.44	6.80	3.88	8.78	13.23	13.69	
Diff. Corrosion Rate, ^d mpy	-	-	15 ^e	-	5 ^e	-	2.2 ^e	-	-	0.5 ^e	1.0	f	f	f	0.7	1.1	Overall Rate 1.0 mpy

(a) H₂O added to system at hour 226.6.

(b) Addition of oxygen and slurry at hour 239.9.

(c) By gravitational and centrifugal sedimentation in 0.005 M Na₄P₂O₇ using neutron activation analysis.

(d) Based on Cr, Ni found in samples. Iron not included as some was indicated to have come from rotor. This excess iron is estimated above.

(e) Value based on average Cr, Ni for this and preceding sample.

(f) Rate for next period includes this result.

(g) Excess iron calculated from (excess Fe)/Th ratio of each sample; stainless steel assumed as 67% Fe, 19% Cr, 11% Ni, 3% other.

UNCLASSIFIED

TABLE 1.8

Corrosion Rates of Pin and Coupon Specimens, Runs BS-9 through BS-13 and CS-34

and Run BS-9

Time: 27.4 hr
 Temperature: 300°C
 Avg. Conc.: 745 g Th/kg H₂O
 Additives: 120 psig oxygen, H₂SO₄, 0.03 m; 2600 ppm SO₄⁼/ThO₂

	Alloy	No. of Specimens	Corrosion Rate, mpy Defilmed	Range, mpy
Flow (+ 10%)				
10 fps	316 SS	1	11	-----
Insulated	321 SS	1	11	-----
Pins	347 SS	2	10	8-11
347 SS	Armco Iron	3	75	63-81
Holder	Gold	1	None Detected	-----
	Monel	1	32	-----
	Titanium RC-55	1	2.2	-----
	Titanium 6Al, 4V	2	2.8	2.2-3.3
	Titanium 4Al, 4Mn	1	1.1	-----
	Zr (crystal bar)	1	None Detected	-----
Flow (+ 10%)				
20 fps	316 SS	2	28	27-28
Insulated	321 SS	1	34	-----
Pins	347 SS	3	26	15-35
321 SS	446 SS	1	58	-----
Holder	Armco Iron	1	86	-----
	Gold	1	3.8	-----
	Monel	1	52	-----
	Palladium	1	6.2	-----
	Titanium 6Al, 4V	1	5.5	-----
	Titanium 4Al, 4Mn	1	5.5	-----
	Zr (crystal bar)	1	8.7	-----
Flow (+ 10%)				
29 fps	316 SS	1	30	-----
Insulated	321 SS	1	34	-----
Pins	347 SS	2	40	39-40
316 SS	Armco Iron	2	84	83-85
Holder	Gold	1	5.7	-----
	Platinum	1	3.4	-----
	Titanium RC-55	1	6.6	-----
	Titanium 6Al, 4V	3	4	1.1-5.5
	Titanium 4Al, 4Mn	1	5.5	-----
	Zr (crystal bar)	1	8.7	-----

Average loop corrosion rate based on chemical analysis = 16 mpy

TABLE 1.8 (CONT'D)

Corrosion Rates of Pin and Coupon Specimens, Runs BS-9 through BS-13 and CS-34

b. Run BS-10

Time: 123.1 hr
 Temperature: 300°C
 Avg. Conc.: 586 g Th/kg H₂O
 Additives: 120 psig oxygen; H₂SO₄: 4900, later 9000 ppm SO₄/ThO₂

	Alloy	No. of Specimens	Corrosion Rate, mpy Defilmed	Range, mpy
Flow (+ 10%)				
10 fps	316 SS	1	35	-----
Insulated	321 SS	1	27	-----
Pins	347 SS	2	24	22-26
347 SS	Armco Iron	3	1433	1300-1600
Holder	Gold	1	0.3	-----
	Monel	1	240	-----
	Titanium RC-55	1	2.7	-----
	Titanium 6Al, 4V	2	4.5	4-5
	Titanium 4Al, 4Mn	1	3.5	-----
	Zr (crystal bar)	1	0.87	-----
Flow (+ 10%)				
20 fps	316 SS	2	81	80-81
Insulated	321 SS	1	55	-----
Pins	347 SS	3	51	36-59
321 SS	446 SS	1	49	-----
Holder	Armco Iron	1	2600	-----
	Gold	1	10	-----
	Monel	1	450	-----
	Palladium	1	3.4	-----
	Titanium 6Al, 4V	1	13	-----
	Titanium 4Al, 4Mn	1	11	-----
	Zr (crystal bar)	1	5.1	-----
Flow (+ 10%)				
30 fps	316 SS	1	97	-----
Insulated	321 SS	1	67	-----
Pins	347 SS	2	68	67-68
316 SS	Armco Iron	2	3150	3100-3200
Holder	Gold	1	13	-----
	Platinum	1	3.3	-----
	Titanium RC-55	1	11	-----
	Titanium 6Al, 4V	3	13	11-14
	Titanium 4Al, 4Mn	1	13	-----
	Zr (crystal bar)	1	7.5	-----

Average loop corrosion rate based on chemical analysis = 74 mpy

TABLE 1.8 (CONT'D)

Corrosion Rates of Pin and Coupon Specimens, Runs BS-9 through BS-13 and CS-34

c. Run BS-11

Time: 300.3 hr
 Temperature: 300°C
 Avg. Conc.: 733 g Th/kg H₂O
 Additives: 120 psig oxygen; 3400 ppm SO₄/ThO₂

	Alloy	No. of Specimens	Corrosion Rate, mpy Defilmed	Range, mpy
Flow (+ 10%)				
9 fps	304 SS	1	4.3	-----
Insulated	316 SS	2	4.7	4.6-4.7
Pins	321 SS	1	4.1	-----
347 SS	347 SS	3	4.0	3.6-4.2
Holder	Gold	1	0.07	-----
	Platinum	1	0.36	-----
	Titanium RC-55	1	0.20	-----
	Titanium 6Al, 4V	1	0.49	-----
	Titanium 4Al, 4Mn	1	0.29	-----
	Zircaloy-2	2	0.29	0.14-0.43
Flow (+ 10%)				
18 fps	304 SS	1	4.8	-----
Insulated	316 SS	2	4.9	4.8-5.0
Pins	321 SS	1	4.9	-----
321 SS	347 SS	3	4.4	3.6-5.0
Holder	446 SS	1	5.2	-----
	Gold	1	1.2	-----
	Palladium	1	0.23	-----
	Platinum	1	0.65	-----
	Titanium 6Al, 4V	1	0.69	-----
	Titanium 4Al, 4Mn	1	None Detected	-----
	Zircaloy-2	1	0.79	-----
Flow (+ 10%)				
30 fps	304 SS	1	5.2	-----
Insulated	316 SS	2	5.0	5.0-5.0
Pins	321 SS	1	5.0	-----
316 SS	347 SS	3	4.3	3.1-4.9
Holder	Gold	1	2.5	-----
	Platinum	1	0.69	-----
	Titanium RC-55	1	0.29	-----
	Titanium 6Al, 4V	2	0.60	0.0-1.2
	Titanium 4Al, 4Mn	1	0.39	-----
	Zircaloy-2	1	1.4	-----

Average loop corrosion rate based on chemical analysis = 3.6 mpy

TABLE 1.8 (CONT'D)

Corrosion Rates of Pin and Coupon Specimens, Runs BS-9 through BS-13 and CS-34

d. Run BS-12

Time: 113.1 hr
 Temperature: 300°C
 Avg. Conc.: 988 g Th/kg H₂O
 Additives: 120 psig oxygen, 80 psig argon; 300 ppm SO₄⁼/ThO₂

	Alloy	No. of Specimens	Corrosion Rate, mpy Defilmed	Range, mpy
Flow (+ 10%)				
9 fps	304 SS	1	4.1	-----
Insulated	316 SS	2	3.2	3.2-3.2
Pins	321 SS	1	3.2	-----
347 SS	347 SS	3	3.1	3.0-3.2
Holder	Gold	1	0.06	-----
	Platinum	1	0.06	-----
	Titanium RC-55	1	None Detected	-----
	Titanium 6Al, 4V	1	None Detected	-----
	Titanium 4Al, 4Mn	1	0.54	-----
	Zircaloy-2	2	None Detected	-----
Flow (+ 10%)				
18 fps	304 SS	1	5.2	-----
Insulated	316 SS	2	5.8	5.6-6.0
Pins	321 SS	1	6.2	-----
321 SS	347 SS	3	6.1	3.3-6.3
Holder	446 SS	1	8.9	-----
	Gold	1	2.6	-----
	Palladium	1	5.1	-----
	Platinum	1	0.80	-----
	Titanium 6Al, 4V	1	1.4	-----
	Titanium 4Al, 4V	1	1.6	-----
	Zircaloy-2	1	None Detected	-----
Flow (+ 10%)				
29 fps	304 SS	1	6.5	-----
Insulated	316 SS	2	6.1	5.9-6.3
Pins	321 SS	1	6.5	-----
316 SS	347 SS	3	5.3	3.0-6.6
Holder	Gold	1	3.3	-----
	Platinum	1	1.3	-----
	Titanium RC-55	1	1.4	-----
	Titanium 6Al, 4V	2	1.1	1.1-1.1
	Titanium 4Al, 4Mn	1	1.4	-----
	Zircaloy-2	1	0.57	-----

Average loop corrosion rate based on chemical analysis = 9.0 mpy

TABLE 1.8 (CONT'D)

Corrosion Rates of Pin and Coupon Specimens, Runs BS-9 through BS-13 and CS-34

e. Run BS-13

Time: 304.6 hr
 Temperature: 300°C
 Avg. Conc.: 478 g Th/kg H₂O
 Additives: 120 psig oxygen, 80 psig argon

	<u>Alloy</u>	<u>No. of Specimens</u>	<u>Corrosion Rate, mpy Defilmed</u>	<u>Range, mpy</u>
Flow (+ 10%)				
10 fps	304 SS	1	4.0	-----
Insulated	316 SS	2	4.2	4.1-4.2
Pins	321 SS	1	4.8	-----
347 SS	347 SS	3	4.1	3.9-4.2
Holder	Gold	1	0.05	-----
	Platinum	1	0.21	-----
	Titanium RC-55	1	1.6	-----
	Titanium 6Al, 4V	1	1.7	-----
	Titanium 4Al, 4Mn	1	1.6	-----
	Zircaloy-2	2	0.85	0.78-0.92
Flow (+ 10%)				
21 fps	304 SS	1	6.5	-----
Insulated	316 SS	2	6.2	6.0-6.4
Pins	321 SS	1	6.5	-----
321 SS	347 SS	3	5.4	4.6-5.8
Holder	431 SS	1	8.0	-----
	Gold	1	0.37	-----
	Palladium	1	5.0	-----
	Platinum	1	0.82	-----
	Titanium 6Al, 4V	1	3.9	-----
	Titanium 4Al, 4Mn	1	2.5	-----
	Zircaloy-2	1	2.1	-----
Flow (+ 10%)				
31 fps	304 SS	1	6.7	-----
Insulated	316 SS	2	6.3	6.2-6.3
Pins	321 SS	1	6.6	-----
316 SS	347 SS	3	5.5	4.4-6.4
Holder	Gold	1	0.41	-----
	Platinum	1	1.1	-----
	Titanium RC-55	1	2.9	-----
	Titanium 6Al, 4V	2	3.6	3.1-4.1
	Titanium 4Al, 4Mn	1	3.7	-----
	Zircaloy-2	1	3.2	-----

Average loop corrosion rate based on chemical analysis = 2.1 mpy

TABLE 1.8 (CONT'D)

Corrosion Rates of Pin and Coupon Specimens, Runs BS-9 through BS-13 and CS-34

f. Run CS-34

Time: 501.2 hr
 Temperature: 300°C
 Avg. Conc.: 516 g Th/kg H₂O
 Additives: 120 psig oxygen, 80 psig argon

	Alloy	No. of Specimens	Corrosion Rate, mpy Defilmed	Range, mpy
Flow (+ 10%)				
10 fps	309 SCb SS	1	0.27	-----
Insulated	316 SS	1	0.28	-----
Pins	347 SS	2	0.33	0.33-0.33
316 SS	SA-212-B Steel	1	17	-----
Holder	Armco Iron	1	28	-----
	Inconel X	1	5.9	-----
	Monel	1	28	-----
	Stellite 1	1	15	-----
	Stellite 98M2	1	26	-----
	Gold	1	None Detected	-----
	Platinum	1	None Detected	-----
	Titanium 3Al, 5Cr	1	0.24	-----
	Zircaloy-2	1	None Detected	-----
Flow (+ 10%)				
30 fps	304 SS	1	1.4	-----
Insulated	304 L SS	2	1.9	1.6-2.2
Pins	309 SCb SS	2	1.6	1.4-1.7
316 SS	316 SS	1	1.4	-----
Holder	347 SS	2	1.1	0.84-1.4
	SA-212-B Steel	1	24	-----
	Gold	1	0.17	-----
	Platinum	1	0.46	-----
	Titanium 6Al, 4V	1	2.4	-----
	Titanium 4Al, 4Mn	1	2.3	-----
	Zircaloy-2	1	0.59	-----
Flow (+ 10%)				
40 fps	304 SS	1	11	-----
Insulated	304 L SS	2	12	11-12
Pins	309 SCb SS	2	13	11-15
321 SS	316 SS	1	12	4.9-12
Holder	347 SS	2	8.5	-----
	SA-212-B Steel	1	31	-----
	Gold	1	1.9	-----
	Platinum	1	3.9	-----
	Titanium 6Al, 4V	1	9.5	-----
	Titanium 4Al, 4Mn	1	9.3	-----
	Zircaloy-2	1	3.0	-----

TABLE 1.8 (CONT'D)

Corrosion Rates of Pin and Coupon Specimens, Run BS-9 through BS-13 and CS-34f. Run CS-34
(continued)

Time: 501.2 hr
 Temperature: 300°C
 Avg. Conc.: 516 g Th/kg H₂O
 Additives: 120 psig oxygen, 80 psig argon

	Alloy	No. of Specimens	Corrosion Rate, mpy Defilmed	Range, mpy
Flow (+ 10%)				
46 fps	304 SS	1	18	-----
Insulated	304 L SS	2	15.5	15-16
Pins	309 SCb SS	2	17	16-18
347 SS	316 SS	1	20	-----
Holder	347 SS	2	14.5	12-17
	SA-212-B Steel	1	33	-----
	Gold	1	2.5	-----
	Platinum	1	4.5	-----
	Titanium 6Al, 4V	1	12	-----
	Titanium 4Al, 4Mn	1	13	-----
	Zircaloy-2		4.2	-----
Flow (+ 10%)				
18 fps	304 L SS	1	0.37	-----
Insulated	309 SCb SS	1	0.45	-----
Coupons	347 SS	3	0.51	0.41-0.69
Ti Circ.	347 SS (welded) ^a	1	0.54	-----
Chan. Holder	Armco Iron	2	12	12-12
	Incoloy	1	0.33	-----
	Titanium 75A	1	0.18	-----

	Alloy	Velocity fps (+ 10%)	Corrosion Rate, mpy Defilmed	Weight Loss ₂ mg/cm ²
Insulated				
Coupons	347 SS	6	0.19	0.23
Ti Double		7	0.17	0.20
Tapered		8	0.15	0.18
Holder		11	0.17	0.20
		14	0.30	0.36
		22	0.48	0.58
		25	0.70	0.83
		29	2.2	2.6
		33	3.5	4.2
		41.52 ^b	17	20

a. Flow across weld bead

b. Velocity at trailing edge

Loop corrosion rate based on chemical analysis = 1.0 mpy

Corrosion rates calculated from defilmed weight losses of pin specimens exposed at 10, 30, and 40 fps were moderate. At 10 fps, austenitic stainless steel specimens corroded at an average rate of 0.3 mpy, mild steels at 23 mpy and titanium alloys at 0.2 mpy. There were no detectable weight losses of gold, platinum, or Zircaloy-2 pin specimens at 10 fps.

At 30 and 40 fps, respectively, the average rates (in mpy) were: austenitic stainless steels, 1.5 and 12; mild steels, 24 and 31; titanium alloys, 2.4 and 9.4; Zircaloy-2, 0.6 and 3; and gold and platinum, 0.3 and 2.9.

At 46 fps, corrosion rates of pin specimens of the austenitic stainless steels ranged from 12 to 20 mpy; mild steel, 33 mpy; Zircaloy-2, 4 mpy, and titanium alloys, 12-13 mpy.

Coupon specimens of the austenitic stainless steels exposed at 18 fps averaged 0.5 mpy; titanium 75A, 0.2 mpy; and mild steel, 12 mpy.

Type 347 stainless steel coupons contained in a variable velocity specimen holder displayed rates from 0.2 to 17 mpy over the velocity range 6 to 52 fps. No critical velocity was noted. The last three specimens in the holder, at respective velocities of 33, 41, and 52 fps, showed marked localized attack. Irregular areas were attacked, apparently as a result of turbulence patterns, to a depth of 250 to 340 microns (0.010 to 0.015 in.).

No cracks were found in four stress specimens exposed in the vapor space of the pressurizer. The four alloys, each stressed to approximately 80% of their yield values at temperature, were: types 347 and AM 350 stainless steels, titanium - 6Al-4V alloy, and Zircaloy-2.

REFERENCES

- 1.1.a E. L. Compere, H. C. Savage, S. A. Reed, et. al., HRP Dynamic Slurry Corrosion Studies, April 30, 1956, ORNL CF-56-4-139, p 10, Fig. 1.1.2.
- 1.1.b E. L. Compere, H. C. Savage, S. A. Reed, et. al., HRP Quar. Prog. Rep., April 30, 1956, ORNL 2096, p 88.
- 1.1.c H. C. Savage, et. al., HRP Quar. Prog. Rep., March 15, 1952, ORNL-1280, Fig. 16.
- 1.1.d J. C. Griess, H. C. Savage, et. al., HRP Quar. Prog. Rep., Oct. 31, 1954, ORNL-1813, p 63.
- 1.1.e J. C. Griess, H. C. Savage, et. al., HRP Quar. Prog. Rep., July 31, 1955, ORNL-1943, Fig. 8.3.
- 1.3.a E. L. Compere, H. C. Savage, S. A. Reed, et. al., HRP Quar. Prog. Rep., Oct. 31, 1956, ORNL-2222, p 81.
- 1.3.b E. L. Compere, H. C. Savage, S. A. Reed, et. al., HRP Quar. Prog. Rep., Jan. 31, 1957, ORNL-2272, p 90.
- 1.3.c E. L. Compere, H. C. Savage, S. A. Reed, et. al., HRP Quar. Prog. Rep., July 31, 1956, ORNL 2148, p 83.

2.0 TOROIDS. (by S. A. Reed)

Tests were continued to study the corrosion effects of slurries prepared from oxide calcined at different temperatures. Thoria prepared from thorium oxalate precipitated at 10°C was calcined at temperatures of 1000, 1200, 1400, and 1600°C. Average particle sizes, approximately 1 to 1.5 μ , were little changed in 314 hr of circulation in toroids.

In a 309-hr toroid run at 250°C, 26 fps and 50 psi oxygen with slurries at 500 and 1000 g of thorium per kilogram of water using thorium oxides calcined at 550°C and 650°C, corrosion rates were generally ordinary or slightly lower for type 347 stainless steel, titanium 75A, Zircaloy-2 or carbon steel SA-212-B. Final particle sizes were approximately 0.7 μ .

2.1 Effect of Calcination Temperature.

Results of several runs, made to investigate the effect of calcination temperatures on thorium oxide attrition and metal attack rates during circulation in toroids, have been reported previously. (2.1.a) An additional test was made during the quarter using a new batch of ORNL-produced thoria which was prepared by precipitation of thorium oxalate at 10°C and calcined at 1000°C. Portions of this preparation were subsequently re-calcined at 1200, 1400, and 1600°C, respectively, for 4 hr in order to evaluate the most suitable temperature at which to calcine the entire batch for use in a proposed 100A loop run. Slurries prepared from each firing were circulated for 314 hr at 26 fps, 250°C with 50 psi oxygen at approximately 500 g of thorium per kilogram of water. Oxygen was provided by the thermal decomposition of hydrogen peroxide. Results of this test are given in Table 2.1.

No apparent degradation occurred in any of the tests. The average sedimentation particle size of each thoria preparation before and after circulation was $1.2 \pm 0.2 \mu$.

Corrosion rates of pin specimens were (in mpy): type 347 stainless steel, 1.4 to 0.4; titanium 75A, 0.9 to 0.0 and 0.7 to 0.0; and carbon steel

UNCLASSIFIED

TABLE 2.1

Results of High Temperature Circulation Tests of Various Aqueous Thoria Slurries in Toroids

Run No.	Hours	Fps	Toroid	Temp. or °C	Th Conc. g/kg H ₂ O	Batch Identif.	Calcin. °C-hr	Atmos.	Additive	Conc. m	Avg. Dia.		pH Post	(a) Corrosion Rate, mpy (Pins Defilmed)					Remarks
											Pre	Post		Toroid	347 SS	SA-212-B	Zr-1/3A	Ti	
116	314	26	I	(250)	500	TO-10-1000	1000-4	O ₂	None	-	1.2	1.3	6.2	0.4	1.4	1.1	WG	0.9	Toroid Leaked
			J	249	500	TO-10-1000	1200-4	O ₂	None	-	1.2	1.2	5.1	0.2	1.1	1.6	WG	0.2	
			K	249	500	TO-10-1000	1400-4	O ₂	None	-	1.2	1.4	-	0.03	WG	0	WG	0.03	
			L	243	500	TO-10-1000	1600-4	O ₂	None	-	1.3	1.5	4.6	0.1	0.4	0.8	WG	0.1	
117	309	26	M	233	500	LO-17	550-24	O ₂	None	-	4.7	0.6	5.2	0.3	0.9	2.1	WG	3.3	
			N	(250)	500	LO-18	650-24	O ₂	None	-	3.3	0.6	5.4	0.3	1.0	2.2	WG	0.8	
			O	265	500	LO-19	650-4	O ₂	None	-	2.6	0.7	5.9	0.4	1.1	3.0	0.1	0.8	
			P	247	1000	LO-18	650-24	O ₂	None	-	3.3	0.7	4.9	0.4	1.5	4.7	1.0	1.0	

(a) Corrosion Rates not corrected for plug flow. Fill was approximately 45% at 250°C.

UNCLASSIFIED

SA-212-B, 1.6 to 0.8. In general, the lower rates were observed with slurries prepared from the higher calcined thorium. Differences within the above ranges, however, are not regarded as significant. Consequently, it has appeared feasible to use any of these temperatures, including 1600°C, to calcine this material for loop use.

In another run, slurries made from three lots of ORNL-produced thorium which were prepared from thorium oxalate precipitated at 70°C and calcined at 550°C for 24 hr, 650°C for 4 hr, and 650°C for 24 hr, respectively, were circulated for 309 hr at 26 fps with 50 psi of oxygen. The 550 and 650°C-calcined batch which was fired for 4 hr were circulated at a concentration of 500 g of thorium per kilogram of water. The 650°C-calcined batch, which was fired for 24 hr, was circulated at concentrations of 500 and 1000 g of thorium per kilogram of water. Results of this test are given in Table 2.1.

Before circulation, the slurries were composed of particles whose average sizes were 2.6 to 4.6 μ . After circulation, the average particles sizes ranged from 0.6 to 0.7 μ . Attack rates of the 500 g of thorium per kilogram of water slurries at 26 fps ranged from 0.9 to 1.1 mpy for type 347 stainless steel, 2 to 3 mpy for SA 212 Grade B steel, and 0.0 to 0.1 mpy for Zircaloy-2. No significant difference in corrosivity between the above slurries was indicated.

At 1000 g of thorium per kilogram of water, using the 650°C-calcined oxide fired for 24 hr, corrosion rates (in mpy) were: type 347 stainless steel, 1.2; titanium-75A, 1.0; Zircaloy-2, 1.0; and SA 212 Grade B carbon steel, 4.7.

A loop test, run BS-13, which circulated this same preparation at 500 g of thorium per kilogram of water for 500 hr at 300°C is described in section 1.4 of this report.

REFERENCES

- 2.1.a G. E. Moore and E. L. Compere, Dynamic Slurry Corrosion Studies: Quarter Ending Jan. 31, 1956, ORNL CF-56-1-168, p 33.

3.0 IN-PILE SLURRY TOROID DEVELOPMENT. (by R. M. Warner)

During the past quarter, test evaluation of the experimental model of an in-pile toroid rotator, described previously, (2.1.a, 3.a) has continued. The primary mechanical difficulty encountered was the wearing of the rotator arm bearings and rotator gears. Neither standard ball nor needle type roller bearings have been satisfactory for this application. At present, a rotator arm which will employ double-row angular contact ball bearings and case-hardened gears is being fabricated for test and evaluation.

A second experimental model of an in-pile toroid rotator which has been designed and constructed is shown in Fig. 3.1. Motion is applied to the toroids by an entirely different mechanism than the first model. The air motor used in the first model is retained. However, the gear train is replaced by two rods with two gimbals and a universal joint. The two gimbals prevent rotation of the toroids around their own axis and the universal joint permits circular motion of the ends of the rotator arms. The double instead of single rotator arms and gimbals have been used to attain dynamic balance of the rotating mechanism.

The advantages of this second model over the first are: (1) The point where the rotator arm passes through the gimbals provides a null point of motion from which thermocouple, power, coolant, and pressure connection leads can be routed with minimum flexing during rotation and (2) The toroids can be operated close to the reactor lattice for maximum neutron flux exposure since operational clearance is not required for routing service lines at the forward or reactor side of the toroids. Dynamic balance of the second model appears to be a difficult problem and has not yet been demonstrated. Good dynamic balance is desirable during in-pile operation to prevent excessive vibration of the mechanism in the beam hole liner.

Development work will be continued on both of the toroid rotator models in an effort to obtain a satisfactory mechanism for in-pile operation.

Since controlled operating temperatures to 300°C will be required to obtain the in-pile slurry corrosion data, tests have been initiated to obtain the

Photo 28578
UNCLASSIFIED

Fig. 3.1 Development Model of Rotator for In-Pile Slurry Toroid.

information necessary for the design of suitable service systems. A contained cooling system will be operated to remove the gamma heat generated in the toroids (2 watts/gram has been estimated) and portions of the rotator structure while the reactor is operating at power. However, heating facilities, probably consisting of resistance heating wire, will be required to maintain the experiment operating temperature when the reactor is down.

REFERENCES

- 3.a E. L. Compere, H. C. Savage, and R. M. Warner, HRP Quar. Prog. Rep., Jan. 31, 1957, ORNL-2272, p 100 - 101.