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CONCRETE PROTECTION FROM
SODIUM SPILLS BY INTENTIONALLY
DEFECTED LINERS --
SMALL-SCALE TESTS S9
AND S10

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HANFORD ENGINEERING DEVELOPMENT LABORATORY
Operated by Westinghouse Hanford Company
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P.O. Box 1970 Richland, WA 99352

Prepared for the U.S. Energy Research and Development
Administration under Contract No. AT(45-1)-2170

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Printed in the United States of America
Available from
National Technical Information Service
U.S. Department of Commerce
5285 Port Royal Road
Springfield, Virginia 22161
Price: Printed Copy \$ 4.00 ; Microfiche \$2.25

HEDL-TME 75-75
UC-79a, i, p

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R. K. Hilliard

and

W. D. Boehmer

Hydraulics & Mechanics Programs

July, 1975

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SMALL-SCALE TESTS S9 AND S10

R. K. Hilliard and W. D. Boehmer

ABSTRACT

Two small scale tests were performed to determine the protection against sodium attack afforded to a concrete surface by a defected steel liner. An inert atmosphere was maintained over the sodium pool, which was heated electrically to 1600°F for 2-6 hrs in one test, to 1380°F for 19 hrs in the other. The 10 inch diameter vertical concrete surface was separated from the sodium by a liner plate in which small defects had been drilled. The plates provided significant protection against direct chemical attack, but most of the water was released from the concrete through the defects to react in the sodium pool region. The liners were corroded significantly in the defect areas.

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CONCRETE PROTECTION FROM SODIUM SPILLS
BY INTENTIONALLY DEFECTED LINERS --
SMALL-SCALE TESTS S9 AND S10

1.0 INTRODUCTION

The concrete floor and walls of cells in which sodium piping and equipment are installed are often lined with steel plate. The chief purpose of the liner is to minimize nitrogen loss in inerted atmosphere cells, but the steel plate is also expected to provide protection to the concrete against direct chemical attack by the sodium. A large-scale test⁽¹⁾ showed that concrete can be extensively damaged by spills directly onto a concrete floor in an air atmosphere. Small-scale tests in which sodium was spilled directly onto concrete surfaces in inerted atmosphere vessels that were heated electrically showed that the heterogeneous reaction rate is highly time dependent⁽²⁾. Rapid reaction occurred with fresh concrete surfaces, but in the absence of concrete breakup, the reaction was self-limited by the formation of adherent reaction products. The small-scale tests also showed that most of the water content of the concrete was released to the sodium space from the maximum concrete depth studied (12 inches)⁽²⁾.

The information provided by the earlier small-scale tests was supplemented by performance of two additional tests (S9 and S10) in which defected steel liners separated the concrete from the sodium pool. The results of these two tests are documented in this report.

2.0 SUMMARY AND CONCLUSIONS

Two tests (S9 and S10) were performed in which 11 lb of sodium at approximately 400° F were spilled into an inerted atmosphere reservoir. One wall of the reservoir was a vertical 10-inch diameter liner plate separating the sodium pool from a second vessel containing high density (magnetite) concrete. The liner touched the surface of the concrete in one test; there was a 0.25-inch gas-filled gap in the other. In each test the liner was intentionally defected by drilling a hole and a horizontal slit both below and above the sodium liquid level. Stainless steel type 304 liner plate was used in one test; carbon steel ASTM A-36 in the other.

The sodium pool was heated rapidly to the specified maximum temperature, held at that temperature for a specified period of time, then allowed to cool. In test S9 the temperature was held at $1380^{\circ}\text{F} \pm 20^{\circ}\text{F}$ for 18.7 hr. In test S10 the temperature was $1600^{\circ}\text{F} \pm 20^{\circ}\text{F}$ for 2.6 hr. After the test articles had cooled to ambient temperatures, they were destructively disassembled for examination and sampled for chemical analysis. The results of the tests support the following observations and conclusions.

- The defected liners provided significant protection to the concrete against direct chemical attack by the sodium. The concrete did not crack as it had in a previous test (S-4), nor was the concrete as deeply penetrated⁽²⁾. The depths of sodium penetration into concrete were 5/8 inch in test S9 and 1.25 inches in test S10, which are to be compared with 2.3 inches in test S4.
- Most of the water content of the concrete was driven out of the concrete through the liner defects, the only available points of egress.
- The water from the concrete reacted with sodium in the vapor space and on the vessel interior surfaces. Approximately 75% of the released water formed hydrogen on a mole per mole basis. The remaining 25% of the water was swept out of the sodium reservoir before having an opportunity to react.
- Carbon steel and SS/304 were both severely corroded. The most severe point of attack was in the defected areas in the liner plates. For the worst case (vapor area defects, S10), the 0.28-inch diameter hole was enlarged to a 1.75-inch by 1.55-inch ellipse, and the slit from 1/16-inch by 1-inch to an ell shape with 0.95-inch by 1.62-inch dimensions. Corrosion was more severe in the vapor space than below the sodium surface.
- The solid reaction product material which formed in the gap between the liner and the concrete remained largely in the gap. Very little of the concrete constituents were washed through the defects into the sodium reservoir. One defect below the sodium level was plugged tightly with reaction product and the defect dimensions had remained unchanged.

- Even though no significant quantity of the sodium-concrete reaction product washed into the sodium reservoir, a large quantity of reaction product had accumulated at the bottom of the sodium pool in each test. This material averaged 9 lb. total weight compared to the 11 lb. of metallic sodium initially present, and was identified as being chiefly a mixture of sodium and iron compounds. It is a hard, reddish-brown, brittle material with a melting point of approximately 600° F. It is believed to be the product of reaction by the water vapor with sodium and the interior metal surfaces of the sodium reservoir, including the liner plate. Visual examination showed that the metal surfaces had been corroded in an uneven fashion, with more corrosion in the vapor space and especially near to the sodium surface. The iron content of this material was 1.2 lb in test S9; 1.9 in test S10. If the corrosion had been uniform over all the vessel interior surface, approximately 0.020 inches of metal would have been removed to account for the quantity of reacted iron found.

3.0 EXPERIMENTAL CONTITIONS

3.1 Test Articles

3.1.1 Reaction Vessels

The test articles used in tests S9 and S10 were similar in construction. Each was made with two schedule 10 SS/304 10-inch diameter cylindrical sections; one section was filled with concrete and the other served as a reservoir for the sodium pool. The sections were joined together with a seal weld to form a complete reaction vessel. The test articles incorporated concrete liner plates into which defects purposely had been formed. Figure 1 shows details of the types and positions of liner defects tested. Type 304 stainless steel was used as the liner material in test S9 and carbon steel was used in test S10.

The test articles were instrumented with thermocouples as shown in Figures 2,3 and 4. The sodium reservoir sections of both articles were fitted with external electrical heating elements and a 4-inch thickness of ceramic fiber material* was used to insulate both the sodium and concrete sections.

* Kaowool blanket, 8 lb/ft³, k = 0.033 Btu/hr-ft-°F at 600° F. Manufactured by Babcock & Wilcox Co.

Both tests were performed with the test articles installed inside a steel cabinet to provide the necessary inert containment. Sealed penetrations were made in the cabinet for sodium, nitrogen, and vent lines and thermo-couple and heater leads. The entire cabinet enclosure was placed in a controlled ventilation cell described in Section 3.2.

3.1.2 Concrete

Both Tests were performed using high-density magnetite concrete (FFTF type M-225). The composition of the concrete at the time of mixing is shown in Table I. The concrete was poured into 10-inch diameter steel shells and vibrated on a shaker table. The top surfaces were leveled with a wooden trowel. The concrete had aged 178 and 206 days respectively for tests S9 and S10 at the time of testing.

3.2 Test Facility

The tests were performed in the Large Sodium Fire Facility (LSFF). The test articles were placed within a sheetmetal pipeway mockup used in previous large-scale proof tests⁽¹⁾ and small-scale reaction tests⁽²⁾. The mockup was equipped with a nitrogen flooding system with the capacity for delivering measured flow rates of 0 - 200 SCFM. Temperature measurements were made with thermocouples and recorded by multi-point stripchart recorders. A vacuum pump and multiple sampling tubes leading to an oxygen meter, hydrogen analyzer, and hygrometer provided gas sampling and analyzing capabilities. The O₂ and H₂ concentrations in the gas vented from the test articles were recorded on stripchart recorders.

The electrical current and voltage supplied to the test article heaters were monitored on meters and the current was recorded on a stripchart recorder. The room in which the pipeway mockup was located was provided with 5000 CFM of ventilating air flow. The exhaust air was scrubbed, filtered and discharged through a 250-ft stack.

A 55-gallon drum melt station was used to provide the required amount of liquid sodium.

TABLE I
CONCRETE COMPOSITION⁽¹⁾

	<u>lb/ft³</u>	<u>Wt. %</u>
Portland Cement	23.5	10.1
Water	11.6	5.0
Pozzolan	1.6	0.7
Aggregate (magnetite, 3/4 in.)	107.6	46.2
Sand (magnetite)	<u>88.6</u>	<u>38.0</u>
TOTAL	232.9	100.0

(1) From batch plant records dated February 15, 1974.

3.3 Test Procedures and Conditions

Tests S9 and S10 were performed according to the basic procedure outlined below:

- . Melt and preheat the sodium
- . Purge the system enclosure with nitrogen
- . Preheat the test article
- . Spill sodium into the test article reservoir
- . Heat the sodium pool to a specified temperature
- . Maintain the pool temperature for a specified period of time
- . Discontinue heating and allow cooldown to room temperature
- . Disassemble the test article
- . Perform visual examination and chemical analysis

The specific conditions under which the two tests were performed are listed in Table II. Test S9 was performed first, and the results were used to determine the parameters for test S10. Significant parameters that differed between the two tests were the type of liner material, the maximum sodium temperature, the length of time the maximum temperature was maintained, and the gas gap between liner and concrete surface.

4.0 RESULTS

4.1 Temperature History

The temperatures of the sodium pool and liner plate are shown as a function of time in Figures 5 and 6 for tests S9 and S10 respectively. The electrical heater power supplied to the sodium reservoir is also plotted on these figures. Fluctuations in the vapor space and liner plate temperatures were evident in both tests at early times and indicate that water vapor released from the concrete was reacting with sodium vapor. The liner plate attained temperatures up to 250° F higher than the sodium pool and in some cases 100° F higher than the vapor temperature. This suggests that a heterogeneous reaction was occurring on the liner surface. These fluctuations did not persist beyond 250 minutes after the spill and corresponded to the time period when hydrogen and moisture were released in the vent gas. Post-test examination of the liner plate showed extensive corrosion of the liner had occurred near defects in the vapor area.

TABLE II
SUMMARY OF TEST CONDITIONS

<u>Parameter</u>	<u>Test S9</u>	<u>Test S10</u>
Date performed	August 13, 1974	September 10, 1974
<u>Concrete</u>		
Type	Magnetite, M-225	(a)
Age, days	178	206
Mass, lb	68.3	68.5
Container	10-in. pipe, Sch. 10, 6" long	(a)
Surface Orientation	Vertical	(a)
<u>Sodium</u>		
Mass, lb	11.0 $\pm$ 0.5	(a)
Reservoir	10-in. pipe, Sch. 10, 8" long	(a)
<u>Liner</u>		
Material	304 SS	ASTM A-36 carbon steel
Thickness, in.	0.15	0.25
Defects	1"x1/16" slots & 0.28"D holes; one each above & be- low Na surface	(a)
Gap between liner and concrete, in.	0 to 0.10	~0.25
<u>Temperature History</u>		
Max Na temp., °F	1380	1620
Time at temp., hrs	19	2.8
Na spill temp., °F	400	400
Avg heating rate, °F/min	8	12
<u>Miscellaneous</u>		
N ₂ purge rate, SCFM	2.7	2.3 SCFM @ t < 95 min. 3.0 SCFM @ t > 95 min.
avg O ₂ conc., vol. %	0.9	1.1

(a) Same as in Test S9

The temperature of the concrete at various distances from the liner is shown in Figures 7 and 8 for tests S9 and S10, respectively. Thermocouple 1, located 1-inch deep opposite the sodium pool, malfunctioned in test S9, but data for this location was obtained in test S10. Note in Figure 8 that whereas the 1-inch deep TC opposite the vapor space lagged below the 1-inch deep TC opposite the liquid pool during heat-up, it exceeded the lower TC after 225 minutes. This is indicative of the exothermic reaction in the vapor space region.

4.2 Water and Hydrogen Evolution

The purge gas vented from the test enclosure was continuously monitored by on-line instruments for hydrogen, oxygen and water vapor concentrations. At selected times during the tests, grab samples of the vent gas were taken for mass spectrometric analyses. Figure 9 presents the hydrogen and moisture concentrations for test S9. Similar data are presented in Figure 10 for test S10. It is apparent in both tests that moisture and hydrogen were released at the same time in fairly constant ratios. The mass spectrometer data are presented in Table III.

TABLE III
COMPARISON OF GAS SAMPLE ANALYSES

Test No.	Sample Time, Min.	Mole Percent				
		N ₂ Mass Spec	(a) O ₂ Mass Spec	(b) O ₂ On-Line Analyzer	(a) H ₂ Mass Spec	(c) H ₂ On-Line Analyzer
S9	26	96.6	1.25	1.28	2.60	2.10
S9	1140	99.3	0.56	0.62	0.06	0.18
S10	20	97.5	0.49	0.63	1.90	1.68
S10	50	93.2	0.79	0.92	4.86	4.20
S10	92	89.2	1.13	1.26	8.72	6.92

- (a) Mass spectrometric analyses were performed on grab samples taken over a 30 sec period of time.
- (b) On-line oxygen analysis was by Beckman Model 742, read at middle of grab sample period.
- (c) On-line hydrogen analysis was by MSA thermal conductivity analyzer model T3, calibrated with dry nitrogen carrier.

In both tests there was a high level of hydrogen evolution during the initial 250-minute period. As was stated in the previous section, this period corresponds to the period when fluctuating thermocouple responses were recorded, indicating that an exothermic sodium-water reaction was occurring. In safety analyses there is a need to know the stoichiometry of the chemical reaction in order to estimate the quantity of hydrogen formed. Assuming that sodium hydride is unstable at the high sodium temperatures involved in these tests, and that solubility effects are minimal due to the sweeping action of water in the vapor space, the two possible reactions of concern are:

If the reaction proceeds according to Equation (1), one-half mole of hydrogen is produced per mole of water, while Equation (2) produces one mole of hydrogen per mole of water. In Figure 11 the total quantity of hydrogen released is plotted as a function of time for both tests. The higher temperature attained in test S10 caused both a more rapid release and a slightly higher overall release than test S9.

The concrete was analyzed post-test for water content as a function of depth and the results are summarized in Table IV. Most of the concrete had been dehydrated of both its evaporable and chemically bound water content. Table V summarizes the quantities of water released during both tests and compares these quantities with the hydrogen that evolved.

The ratio of hydrogen formed to water released was greater than 0.5, which indicates that Equation (2) applies. The fact that the ratio was not closer to unity is explained by the fact that not all of the water released from the concrete reacted. Figures 9 and 10 show that a significant quantity of water vapor accompanied the hydrogen in the vent gas. Apparently, the geometry of the test article was such that water vapor escaped from the concrete through the defects in the liner located above the sodium surface. Some of the water vapor reacted with sodium vapor above

TABLE IV
WATER CONTENT OF CONCRETE

Depth from Concrete Surface (in.)	Weight % H ₂ O in Post-Test Samples	
	Test S9	Test S10
1-2	0.18	0.19
3	0.11	0.18
5-6	0.17	0.29
Weighted average entire section	0.16	0.22
Pretest H ₂ O content	4.25	4.25
% of original H ₂ O remaining	3.80	5.20

TABLE V
COMPARISON OF WATER RELEASED AND HYDROGEN FORMED

Test Number	Initial H ₂ O Content, lbs	H ₂ O Released			H ₂ Evolved, lb-mole	Mole Ratio H ₂ /H ₂ O
		wt. %	lbs.	lb-mole		
S9	2.90	96.2	2.80	0.156	0.113	0.72
S10	2.91	94.8	2.77	0.154	0.125	0.81

the pool, some reacted with the pool surface and with sodium deposits on the liner and other surfaces of the vessel, but some water vapor was swept out of the test article unreacted. These results are in agreement with earlier tests⁽²⁾.

4.3 Post-Test Examination

When the test articles had cooled to room temperature, they were destructively disassembled and photographed and samples were taken for chemical analysis. The articles from both tests were similar in appearance, so only the test S10 photographs are shown in this report in Figures 12 through 19. An exception is the view of the liner plate in test S9 as shown in Figure 20.

Figure 12 shows the test article with heaters and vent pipe. The concrete portion was removed by grinding off the seal weld on the flange, exposing the interface shown in Figure 13. The 1/4-inch gap between the concrete and liner was filled with an oxide--hydroxide product at levels below the sodium pool surface. The gap at elevations above the pool surface was partially filled with reaction product. A closeup view of the concrete is shown in Figure 14. Figure 15 shows a section of the concrete after splitting it across the diameter. The maximum depth of penetration of the dark colored reaction product was 1.25 inches. The balance of the concrete was hard and difficult to break by chiseling. It was not cracked by the sodium as in test S4⁽²⁾, which had no liner.

The defects in the lower liner plate (below the sodium pool level) had not changed appreciably. The slot was packed with reaction product and its dimensions were the same as before the test. The hole had enlarged from 0.28 inch diameter to 0.32 by 0.38-inch oval, and some frozen sodium had penetrated to the concrete surface opposite the hole. The plate thickness had decreased from 0.25 to 0.22 inches.

The defect areas in the upper liner plate were greatly enlarged, as shown by Figure 16. The slot had increased from 1-inch long by 1/16-inch wide to an ell shape with 1.62 by 0.95 inch major dimensions. The 0.28 diameter hole had become an ellipse with 1.75 by 1.55 inch axes. The liner plate had thinned to a dull knife edge at the upper defects, with most of

the corrosion occurring on the sodium side. The liner plate had broad grooves and dimples over its entire surface on the sodium side. The concrete side had been attacked, but to a much lesser extent. The liner plate was cleaned, weighed and found to have lost 0.62 lb mass. Approximately 0.22 lb loss is explained by the enlargement of the defect areas, but the remaining 0.4 lb loss was due to a general attack on the entire liner surface. This general attack averaged 18 mils, but grooves up to ~50 mils were observed.

The top of the sodium reservoir was sawed off to expose the surface of the sodium pool. Figure 17 shows a view of the pool with the liner plate in the background. The liner was removed to expose the sodium pool material which had been touching the liner, as shown in Figure 18. The maximum pool depth was 3.75 inches deep, which included a top layer 1.16 inches deep of fairly pure metallic sodium and a bottom layer of hard, reddish brown crystalline material. The upper, metallic layer is clearly seen in Figure 18 as a white band. The lower material was brittle and fairly difficult to chisel. Its melting point was determined to be in the range of 570 - 660° F.

Figure 19 shows the liner from the sodium side after it had been cleaned. The three bands corresponding to the vapor space, the metallic sodium and the bottom reaction product are clearly evident.

Test S9 gave very similar visual results as S10 except that the liner was not corroded quite so severely, and the concrete was penetrated to a maximum depth of 5/8 in. The dimensions are listed in Table VI, along with those of Test S10. Figure 20 gives a view of the liner plate from Test S9.

4.4 Chemical Analyses

Samples of the residual sodium and the reaction products found between the liner and the concrete and beneath the sodium pool were taken for chemical analysis. The results are listed in Table VII.

TABLE VI
CHANGES IN LINER DIMENSIONS

	S9	S10
Hole in vapor space, initial	0.28" dia	0.28" dia
Hole in vapor space, final	0.56" x 0.63" oval	1.55" x 1.75" oval
Slot in vapor space, initial	0.0625" x 1"	0.0625" x 1"
Slot in vapor space, final	0.15" x 1.06" (max.)	0.95" x 1.62" (max.) (ell shape)
Hole in liquid space, initial	0.28" dia	0.28" dia
Hole in liquid space, final	(a)	0.32" x 0.38" oval
Slot in liquid space, initial	0.0625" x 1"	0.0625" x 1"
Slot in liquid space, final	(a)	0.0625" x 1"
Weight loss of liner material	(b)	0.62 lb

(a) Not available, but known to be less corrosion than in vapor space.

(b) Not available, but beleived to be less than test S10.

TABLE VII
SUMMARY OF CHEMICAL ANALYSES

	WEIGHT %					
	Material in Gap Between Liner(a) & Concrete		Material on Top of Sodium Pool		Material on Bottom of Sodium Pool(a)	
	S9	S10	S9	S10	S9	S10
Mass, lbs	0.2	0.3	5.1	3.9	8.6	9.3
Water-soluble material	37.0	41.0	100.0	(b)	79.0	69.0
Soluble in hot HCl	(b)	(b)	(b)	(b)	99.7	(b)
Metallic Na	0.05	4.6	99.9	99. ^{+(c)}	3.5	5.2
Na ₂ O	29.0	26.5	(b)	0.14	(b)	69.5
NaOH	(b)	3.6	(b)	(b)	(b)	3.9
Total Na	21.2	28.0	99.9	99. ^{+(c)}	53.5	47.5
Fe	39.8	34.0	0.003	(b)	14.0	20.5
Total Si	2.4	0.58	(b)	(b)	0.29	0.88
Water Soluble Si	0.40	0.18	0.02	(b)	(b)	0.38
Ca	4.0	4.6	<0.003	(b)	0.03	0.13
Al	0.7	0.29	<0.002	(b)	0.04	0.16
Mg	0.38	0.55	<0.001	(b)	0.006	0.005
Cr,Mn,Ni	(b)	0.11	(b)	(b)	0.25	0.73

(a) Average of two to four samples

(b) Not analyzed

(c) Estimated visually

For both tests, the material on the top of the sodium pool was nearly pure metallic sodium. The reaction product in the gap between liner and concrete contained approximately 60 percent water insoluble material which was probably unreacted concrete constituents. The rock-like material in the bottom of the pool was chiefly compounds of sodium and iron. Only small amounts of concrete constituents (Ca, Si, Al, Mg) were present in the

pool residue, showing that concrete-sodium reaction products from the gap between liner and concrete had not washed into the sodium pool.

The exact chemical form of the reaction products was not determined, but an attempt was made to estimate the approximate composition by assuming that all of the sodium formed Na_2O and Na_2SiO_3 , all iron was present as FeO , all soluble silicon formed Na_2SiO_3 , insoluble silicon was SiO_2 , and all other metallic elements formed their stable oxides. The approximate composition is shown in Table VIII.

TABLE VIII
APPROXIMATE COMPOSITION OF REACTION PRODUCT

	Calculated Weight Percent			
	Material in Gap Between Liner and Concrete		Material on Bottom of Sodium Pool	
	S9	S10	S9	S10
Na_2O	29. (a)	27. (a)	65.	70. (a)
NaOH	(b)	3.6 (a)	(b)	(b)
Na_2SiO_3	1.7	0.8		1.7
FeO	51.	44.	18.	26.
SiO_2	4.3	0.9	0.6	1.1
CaO	5.6	6.4	0.04	0.2
Al_2O_3	1.3	0.6	0.1	0.3
MgO	0.6	0.9	0.01	0.1
Other	6.5	16.	16.	0.6

(a) Measured by chemical analysis

(b) For calculation purposes, it is assumed that all reacted sodium forms Na_2O and Na_2SiO_3 .

The mass of iron in the reaction product beneath the sodium pool was 1.2 pounds and 1.9 pounds as iron for tests S9 and S10, respectively. This fairly large amount of iron must have come from the reservoir and liner surfaces, since only small amounts of concrete constituents entered the sodium pool through the defects. Not all the corrosion occurred on the liner; significant corrosion of the main reservoir walls must also have occurred because in test S10 the liner was carbon steel, yet significant quantities of Cr, Mn and Ni were in the pool reaction product. More corrosion occurred in test S10 than in S9 even though time at temperature was much less. Apparently, the higher temperature of test S10 provided more severe corrosion conditions than time. Corrosion rate data at high temperatures are scarce, but it is known that the rate of steel corrosion in steam rapidly accelerates at temperatures above 1000° F and that sodium hydroxide at high concentrations and temperatures is very corrosive to steel and stainless steels⁽³⁾.

5.0 ACKNOWLEDGEMENTS

The test articles were prepared with the assistance of J. D. McCormack, J. J. Mahoney, and W. H. Romsos. R. E. Menard performed the facility operations. Chemical analyses were by R. Keough and D. Baldwin. Valuable suggestions for planning and evaluating the tests were given by P. R. Shire and D. D. Stepnewski.

6.0 REFERENCES

- (1) R. K. Hilliard and J. M. Yatabe, "FFTF Secondary Sodium Fire Protection Systems Test F1," HEDL-TME-73-48, April, 1973.
- (2) J. A. Hassberger, et al., "Sodium-Concrete Reaction Tests," HEDL-TME-74-36, June, 1974.
- (3) Metals Handbook, 8th Ed., Vol. 1, Properties and Selection of Metals, American Society for Metals, Metals Park, Ohio, p. 265 (1961).

HEDL 7507-53.10

FIGURE 1. Details of Liner Defects.

HEDL 7507-53.8

FIGURE 2. Test Article For Test S9.

HEDL 7507-53.9

FIGURE 3. Test Article For Test S10

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Figure 4. Photograph of Sodium Pool Reservoir Showing Defects In Liner Plate.

FIGURE 5. Temperature of Sodium Pool and Liner, Test S9.

HEDL 7507-53.4

HEDL 7507-53.7

FIGURE 6. Temperature of Sodium Pool and Liner, Test S10.

HEDL 7507-53.5

FIGURE 7. Concrete Temperatures During Test S9.

HEDL 7507-53.6

FIGURE 8. Temperature of Concrete at Various Locations In Test S10.

HEDL 7507-53.3

FIGURE 9. Comparison of Moisture and Hydrogen Concentrations In Cabinet Exhaust Gas, Test S9.

HEDL 7507-53.2

FIGURE 10. Comparison of Moisture and Hydrogen Concentrations In Cabinet Exhaust Gas, Test S10.

HEDL 7507-53.1

FIGURE 11. Total Hydrogen Release As A Function of Time.

745597-1

Figure 12. Photograph of test article after cooling--elevation view--Test S10.

Figure 13. View of Concrete-Liner Interface. Concrete on Left, Liner on Right--Test S10.

745597-6

Figure 14. Closeup View of Concrete Surface Showing Reaction Product Filling Gap--Test S10.

745597-2

Figure 15. Concrete Section Showing Depth of Penetration of Reaction--Test S10.

745597-13

745597-3

Figure 16. Closeup of Defect Areas in Upper Liner Plate Looking From Concrete Side--Test S10.

745597-5

Figure 17. View of Sodium Pool Surface and Vapor Side of Liner--Test S10.

Figure 18. View of Frozen Sodium Pool Surface Adjacent to Liner-Test S10.

745597-14

745597-15

Figure 19. Sodium Side of Liner After Cleaning-Test S10.

Figure 20. View of Liner Looking From Concrete Side--Test S9.

745140-2

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