Sampling and Characterization of Sodium-Water Reaction Products

By

R.L. Eichelberger

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ACKNOWLEDGMENT

The sampling tool was designed by R. S. Kennedy. Analytical chemistry was carried out by W. A. McCollum, M. S. Maseda, R. B. Litsinger, C. H. Trippeda, and R. C. Shepard of the LMEC Chemistry and Metallurgical Laboratory, managed by D. E. Goggin. The contributions of these individuals to this report are gratefully acknowledged.

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ABSTRACT

The products of sodium-water reactions which had taken place in the Large Leak Test Rig at the Liquid Metal Engineering Center were sampled and characterized. The products consisted of the expected compounds in the Na-O-H system, and dissolved readily in ethanol. Removal of such products from a system by melting and draining does not appear feasible, but removal by dissolution in alcoholic solvents should be investigated further.

I. SUMMARY

Sodium-water reaction products (SWRP) which had accumulated in the bottom of the reaction products tank (RPT) of the Large Leak Test Rig (LLTR) were sampled and characterized. Analysis showed that the SWRP consisted of NaOH, Na₂O, NaH, and Na, as expected, in varying proportions. The unreacted sodium in the samples examined ranged from 32 to 60% by weight.

The SWRP reacts (dissolves) rapidly and completely with ethanol, and somewhat less rapidly and completely with Dowanol PM. A magnetic, metallic residue was left, originating in the Croloy - 2-1/4 of the test article in the LLTR. These solvents should be investigated further as possible agents for cleaning sodium systems that are highly contaminated with water reaction products.

The SWRP does not completely liquefy with heating to 800 °F (427 °C). Although it softens to a consistency similar to that of wet sand, it is unlikely that it would flow or could be pumped even at such elevated temperature. The presence of highly corrosive molten NaOH also makes the hot draining of SWRP unattractive.

II. INTRODUCTION

At the request of General Electric*, LMEC sampled and characterized the material which settled to the bottom of the reaction products tank (RPT) after sodium-water reactions occurred in the Large Leak Test Rig (LLTR). The sampling was performed after the fourth sodium-water reaction in the LLTR with the AI-MSG as test article. Approximately 110 lb (50 kg) of water had been injected into the sodium in the four tests. The products of the reaction in excess of the amount soluble in the sodium settled to the bottom of the RPT. An unknown, but probably large, amount of the reaction products remained in the AI-MSG and interconnecting piping.

The purpose of the sampling and characterization program was to obtain experimental data which could be used to evaluate potential techniques for removing sodium-water reaction products (SWRP) from RPT's in large sodium reactor systems. The effort fell naturally into two parts, sampling and characterization, which are discussed in the following sections.

*GE Letter XL-791-01130, T. R. Sandke to S. G. Harbison, "LLTR Reaction Products Tank Deposit Characterization, General Electric Specification N023A2244" (May 12, 1977)

III. SAMPLING

The sampling tool shown in Figure 1 was designed and fabricated. The procedure followed was designated LLTR-SP-016, and is included as Appendix A. The tool was fastened to the end of a piece of pipe about 30 ft long, in order to reach the bottom of the RPT, and rotated by means of a "T" handle at the end of the pipe. Figure 1 is a drawing of the sampling assembly. Photographs of the separated sampling tool are included in Appendix B.

Because it appeared to the operating personnel, by the "feel" of the tool as it was rotated into the deposit on the tank bottom, that the SWRP layer was only an inch or two thick, the tool was lifted and lowered in another spot three times. A sample weight of a pound or more was desired. The procedure used produced a sample that weighed about 1-1/2 lb.

During sampling, there was a layer of liquid sodium about 4 ft deep above the settled SWRP. This sodium was maintained at a temperature of 350°F (177°C) for most of the time between sodium-water reaction tests, and during the sampling of the SWRP. During the removal of the sampler from the RPT, the sodium clinging to it caught fire before the device could be cooled sufficiently to freeze the sodium to permit completion of the bagging. The burning sodium was removed from the outside of the sampler with no apparent effect on the contained sample. After removal of the 30-ft pipe, the sampler body was transported in an argon-filled plastic bag to the chemistry laboratory.

IV. CHARACTERIZATION

Characterization and analysis of the SWRP sample was carried out by the LMEC Chemical and Metallurgical Laboratory, using the specially written LMEC Procedure C&M-SP-002. The procedure and the detailed results of the characterization and analysis are included in Appendix B. The discussion which follows provides an interpretation of the results.

LMEC-38173

Figure 1. Sample Collector Assembly

LMEC-77-7

V. DISCUSSION

The results of the characterization and analysis of the sampled SWRP are described in detail in LMEC Chemical Analysis Report No. 3719, which is included in Appendix B. The X-ray diffraction patterns show that the compounds present in the SWRP are Na_2O , NaOH , and NaH , in addition to unreacted elemental sodium. These three compounds cannot all be in thermodynamic equilibrium with liquid sodium. This circumstance would be a violation of the Phase Rule. The Na-O-H system has been carefully examined thermodynamically by Jansson* and compared with experimental results from many sources. The equation,

was found to move to the left above 707K (813°F) and to the right below that temperature, theoretically. Experimentally, the temperature at which the four condensed phases can coexist was found to be somewhat lower, about 680K (764°F). This means that, at equilibrium, solid NaOH (m.p. 592K, 606°F) cannot coexist with liquid sodium. If the system had been permitted to reach equilibrium at the temperature of the sodium-water reaction in the AI-MSG, there should be no sodium oxide or hydride present. On the other hand, if the system had reached equilibrium at the temperature of the RPT, there should be no sodium hydroxide found.

Obviously, the composition of the sampled SWRP depends on its time and temperature history following the sodium-water reaction. The SWRP from some other system, or from the LLTR RPT following tests at different conditions, would contain the same compounds, but would not necessarily have the composition of any of the samples examined here.

The distribution of the various compounds found in the sample contained in the sampler is not thought to be significant, nor indicative of the distribution in the SWRP layer in the RPT. The layer in the tank seems not to be hard, but

*Sven A. Jansson, "Thermochemistry and Solution Chemistry in the Sodium-Oxygen-Hydrogen System," in Corrosion by Liquid Metals, J. E. Draley and J. R. Weeks, eds. (Plenum Press, New York N.Y. 1970)

instead to have the consistency of very wet sand. Therefore, the sample removed from the tank by the sampler did not end up as discrete layers.

The oxygen analyses by mercury amalgamation show reasonable agreement in one case and poor agreement in two cases, when compared to the composition of samples from the same sections of SWRP, found by X-ray diffraction. Because of the lack of homogeneity in the sample and the rather wide limits of uncertainty in the X-ray analysis, this circumstance is not troubling. The amalgamation reflux analyses for hydrogen agree reasonably well with the amount of hydrogen in the sodium hydride reported by X-ray diffraction.

The amount of sodium lost in the distillations is in the range found by X-ray diffraction. The analysis of the distillation residues shows that the NaH has been decomposed under heat and vacuum, as is expected, and that some of the NaOH has reacted with sodium in the process to give Na_2O and hydrogen.

The dissolution of SWRP in ethanol was rapid and complete, with the generation of substantial heat. In Dowanol PM (propylene glycol methyl ether), the solution rate was somewhat slower, and an insoluble residue of NaOH remained. Distillation residue from the SWRP dissolved more readily in both solvents than the SWRP.

A magnetic, metallic residue remained from dissolution of SWRP or distillation residue in both solvents. This material was found to have the X-ray diffraction pattern of alpha iron, which is iso-structural with the Croloy - 2-1/4 of which the AI-MSG is made. This is, therefore, probably debris arising from the sodium-water-steel interaction during the sodium-water reaction.

The heating of the SWRP in an attempt to find a "melting" point for it showed that the material does not become fluid at 550°F (288°C), the highest temperature reached in the glove box-hot plate test. The hot mass could be shaped rather easily, but it did not flow. Heating to 800°F (427°C) in a closed system did not result in a homogeneous liquid either. Although visibility in the system was not as good as it was in the glove box melting test, it was evident that a substantial quantity of unmelted material remained after the sodium and sodium hydroxide had melted.

VI. CONCLUSIONS

Sodium water reaction products which are separated in the LLTR reaction products tank are a nonequilibrium mixture of sodium hydroxide, sodium oxide, sodium hydride, and sodium. It does not seem feasible to liquefy the SWRP by heat at a temperature below 800°F (427°C). Because of the highly corrosive behavior of liquid NaOH toward steel, it is unwise to heat the SWRP above the melting point of NaOH (606°F, 319°C) in a steel vessel. The rapid and complete solubility of SWRP in ethanol leads to the conclusion that this solvent should be tested more completely as a cleaning agent for sodium systems which are contaminated with water reaction products. Dowanol PM also shows promise for this use, but the lower solubility of NaOH in it makes it less attractive.

APPENDIX A
PROCEDURE FOR SAMPLING
SODIUM-WATER REACTION PRODUCTS
IN LLTR RPT

LMEC-77-7

LLTR-SP-016
Page 01
July 21, 1977

No. TI-45-LME-011
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Date 10/13/77

SAMPLE REMOVAL OF SODIUM-WATER REACTION
PRODUCTS (SWRP) FROM THE REACTION PRODUCTS
TANK (RPT) IN THE LARGE LEAK TEST RIG (LLTR)

Approvals:

R. L. Eichellenger 7/21/77
Test Engineering

W. J. B. 7-21-77
Operations

I. PURPOSE

The purpose of this procedure is to provide instructions for the removal of an SWRP sample from the bottom of the RPT in order to perform chemical analysis on the SWRP formed as a result of SWR tests one through four.

II. METHOD

A special tool has been fabricated to be used to try and remove SWRP material from the bottom of the 26 foot deep RPT. The tool will be inserted through the manhole on the top of the RPT while the sodium is at 300F. A constant cover gas purge will be maintained in the RPT to minimize the influx of oxygen during the sample removal. The tool will be inserted into the sodium pool and rotated when contact is made with the SWRP concentration near the bottom of the RPT. When contact with the bottom of the tank has been made, the sample tool will be raised and allowed to cool prior to removal from the RPT. When the sample has cooled to room temperature, it will be removed from the RPT, and the fixture handle, and placed in a plastic bag for shipment to the Chem. Lab. at LMEC for analysis.

III. REFERENCE

1. GE Letter requesting sample of SWRP for series one tests one through three. Number XL-791-01130 P2648 (Y4), DRF-1971.

IV. TEST EQUIPMENT

1. Special tool designed for SWRP removal.
2. Plastic bag for shipment of sample.

V. SYSTEM LIMITS

None.

VI. PROCEDURE

Date/Initial

1. Shift Leader approval to start procedure.

_____ Shift Leader

2. Verify that the sample tool is clean. _____

3. Remove or verify that the decanter assembly has been removed from the RPT. _____

4. Verify that there is enough N₂ in the NS to maintain the purge on the RPT while the manhole cover is off for sample removal. _____

5. Verify that the manhole cover and any instrument leads have been removed permitting easy access for the sample tool. _____

6. Fabricate and install a plastic bag on the manhole flange. Bag should be approximately 6 to 8 feet long. _____

NOTE

The bag will be used to contain some of the RPT purge and also to make a cooling chamber for the sample as it is removed from the vessel.

7. Connect the sample tool to the overhead crane and lower it into the RPT through the plastic bag. _____

Date/Initial

NOTE

The handle of the sample tool may have to be trimmed in order to fit through the hole in the main deck.

8. When the sample tool has been lowered into the RPT, then close the bag around the shaft enough to minimize cover gas leakage. Leave the bag top loose enough so the sample tool can still be rotated to obtain a sample. _____
9. Start rotating the sample tool clockwise as soon as contact is made with the SWRP. _____
10. Stop rotating the sample tool when the bottom of the RPT is contacted. _____
11. Raise the sample tool above the sodium pool and let the sample cool to cover gas temperature. _____
12. When the sample has cooled to cover gas temperature, raise it into the plastic bag just above the RPT and let it cool to room temperature. _____
13. When the sample has cooled, remove it from the sample tool handle, place it in a plastic bag and send it to the Chem. Lab. for analysis. _____
14. Remove the plastic bag and replace RPT manhole cover. _____
15. Shift Leader sign-off procedure complete. _____

APPENDIX B

CHARACTERIZATION OF SWRP

SAMPLE, PROCEDURE, AND RESULTS

LMEC-77-7

LMEC CHEMISTRY LABORATORY WORK REQUEST					LAB USE ONLY Date: <u>10/13/77</u> LMEC LAB. NO. <u>3719</u>	
*REQUESTER <u>R.L. Eichelberger</u>	*DEPT/GRP <u>720/203</u>	*BLDG <u>T486</u>	*PHONE <u>6165</u>	*DATE SUBMITTED <u>7/22/77</u>	REQ COMP DATE	LAB ACCEPTANCE <u>[Signature]</u>
*PROJECT TITLE, TEST, OR PURPOSE OF SAMPLE <u>SWRP Characterization</u>				LEDGER	GEN ORDER	*SUBACCOUNT <u>05011</u>
*REQUESTER'S SAMPLE NO.		*TYPE OF SAMPLE (Na, Gas, Water, etc.)		*CHEMICAL ANALYSIS		REMARKS REF.
				QUANTITATIVE FOR:	QUALITATIVE FOR:	
<u>SWRP-1</u>		<u>Solid</u>		<u>See remarks</u>		<u>1)</u>
*DISPOSITION OF SAMPLE: ☒ DISCARD ☐ RETURN TO REQUESTER ☐ OTHER (Specify in Remarks)						
*SAMPLE TAKEN FROM: FACILITY <u>LLTR</u> TEST _____						
LOCATION <u>Reaction Product Tank</u>						
*SAMPLING CONDITIONS: BULK TEMP <u>DNA</u> F SAMPLE TEMP _____ F EXPOSURE TIME _____						
*SAMPLING METHOD: <u>See remarks 2)</u>						
OBSERVATIONS OR REMARKS: _____						
APPROX. WT. OR VOL. _____						
ANALYSIS TO VERIFY SAMPLE MEETS: PURCHASE ORDER NO. _____						
OR SPECIFICATIONS _____						
*ADDITIONAL DISTRIBUTION OF CHEMICAL ANALYSIS REPORT:				REQUESTER ONLY		
TO: (Name, Dept., Bldg.) _____				(2 copies)		
REQUESTER'S REMARKS:						
1) Analyze per C+M-SP-002.						
2) Sampling conducted per LLTR-SP-016.						
LMEC LABORATORY ACTION (LAB USE ONLY):						
Refer to Met Lab Work Request #743 for documentation photographs.						
*Items so marked are to be completed by Requester; other information is optional.						

FORM 735-A-35 REV 1-73

REPORT COPY

LMEC-77-7

20

No. TI-45-LME-011

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Date 10/13/77

C+M-SP-002

Page 01

7/21/77

SPECIAL PROCEDURE -

LLTR REACTION PRODUCTS TANK DEPOSIT

CHARACTERIZATION

APPROVED BY

D. E. Higgins
CHEM AND MET LAB. MANAGER

R. L. Enkelmann 7/21/77
LMEC ENGINEERING

LMEC-77-7

I. PURPOSE

The purpose of this procedure is to provide an outline of methods for 1) the physical and chemical characterization of LLTR sodium-water reaction products (SWRP), 2) testing the reactivity of SWRP with various solvents, and 3) measuring the melting point of SWRP to satisfy the requirements of General Electric Specification No. 22A2244, and agreements and commitments, 77LMEC-DRF-2058.

II. METHOD

1. Prior to delivery of a SWRP sample to the LMEC Chem Lab, LMEC Engineering shall request characterization of the sample per this test plan in accordance with LMEC Procedure V-105. A Chem Lab Work Request (CLWR), form 735-A-35, shall be submitted for approval by the Chemical and Metallurgical Laboratory Manager, or his designee.
2. LMEC Engineering shall maintain cognizance over sampling activities, and shall furnish the Chemical Laboratory with the SWRP sample.
3. Upon receipt of the SWRP sample, LMEC Chemical Laboratory shall conduct its characterization in accordance with this special procedure.
4. Chemical Laboratory shall document results per LMEC Procedure V-105 on Chemical Analysis Report form 735-A-34. Two copies of this form and form 735-A-35 will be distributed to LMEC Engineering upon their completion.

III. PREREQUISITES

LMEC Engineering shall initiate and be cognizant over sampling activities and the delivery of a suitable sample of SWRP to the Chemical Laboratory.

IV. REFERENCES

1. General Electric Specification No. 23A2244, attachment to DRF-1971.
2. LMEC Procedure V-105
3. 77LMEC-DRF-2058
4. RDT F3-40T

V. PROCEDURE: CHARACTERIZATION OF SWRP

1. Sample handling upon receipt of sample by the Chemistry Laboratory.
 - 1.1 Introduce sample into glovebox.
 - 1.2 Photograph
 - 1.3 Weigh total sample
 - 1.4 Probe sample - separate into readily identifiable like parts.
 - 1.4.1 Photograph each part
 - 1.4.2 Describe each part
 - 1.4.3 Weigh each part
2. Sample analysis
 - 2.1 XRD on each separated part
 - 2.1.1 Look for all identifiable crystalline phases with emphasis on Na, NaOH, Na_2O , and NaH.
 - 2.1.2 Perform semiquantitative analysis based on comparison of peak height intensities of strongest peaks of each component with those of a common internal standard (Linde "A" Al_2O_3).
 - 2.2 Oxygen analysis by amalgamation per RDT F3-40T on each separated part.
 - 2.3 Hydrogen analysis by amalgam reflux per RDT F3-40T on each separated part.
 - 2.4 Distillation of Na from each separated part.
 - 2.4.1 Weigh each sample.
 - 2.4.2 Weigh each recovered residue.
 - 2.4.3 Photograph each recovered residue.
 - 2.4.4 XRD analysis of each recovered residue.
3. Reactivity with solvents. (As-received SWRP).

V. PROCEDURE: CHARACTERIZATION OF SWRP (Cont.)

- 3.1 Room temperature, no agitation. 300 ml solvent,
10-20g SWRP.

3.1.1 Denatured ethanol.

3.1.2 Dowanol PM.

3.2 Elevated temperature, no agitation.

3.3 Elevated temperature with agitation.

3.4 Room temperature with water addition.

3.5 Repeat Steps 3.1 through 3.4 using residues from
distillation (2.4.2).

4. Melting characteristics.

4.1 Preliminary determination, as-received SWRP. Use hot
plate, surface thermometer, crystallizing dish, metal
dial-type thermometer in sample, sample in glovebox.

4.2 Preliminary determination, separated fractions.
(Repeat 4.1)

4.3 Repeat 4.1 and 4.2 using pyrex funnel and funnel heating
mantle, if warranted.

5. Data Recording

All observations and measurements shall be recorded in
LMEC Data Logbooks.

Results shall be reported per this Special Procedure,
Section II. 4.

CHEMICAL ANALYSIS REPORT

Date 10/13/77

REQUEST NO. 3719

ORIGINATOR R.L. Eichelberger		DEPT.-GRP. 720-203		APPROVAL SIG. <i>[Signature]</i>		DATE OF REPORT 9/16/77	
CHEMICAL ANALYSIS							
Analysis		Method		Investigator			
SWRP Char.		G+M-SP-002		WAM/ MSM/ RBL/ CHT			
RESULTS OF ANALYSIS							
Originator's Sample Number		Analytical Laboratory Number					
SWRP-1				See attached report.			
OTHER MEASUREMENT OR PREPARATION							
Documentation photographs were taken per Met Lab Work Request # 743.							
REMARKS							
LMEC data logbook references: Vol. 546, pp. 75,76,82,89,90,92,94 Vol. 388, pp. 91-93 XRD File #3719							

ATTACHMENT TO LMEC CHEMICAL ANALYSIS REPORT #3719

Subject: Characterization of Sodium-Water Reaction Products

Reference: C&M-SP-002, Part V.

INTRODUCTION

This attachment reports the data and observations obtained in the characterization of a sample of sodium-water reaction products (SWRP) taken from the LLTR reaction products tank on 7-25-77 according to special procedure LLTR-SP-006. The system of numbering used herein corresponds exactly to that which appears in the referenced procedure.

CHARACTERIZATION OF SWRP

1. Sample handling. The SWRP material in its sampling tool was received in a nitrogen-purged and sealed polyethylene bag.
 - 1.1 The SWRP sample, as-received, was opened in a glove box and removed from the sampling tool. The single solid sample of SWRP measured 2 inches in diameter at the top, 2 3/4 inches in diameter at the bottom, and was 7 inches high.
 - 1.2 Photographs:
 - 743-1 Head section of SWRP sampler (in glove box).
 - 743-3 Top view into sampler.
 - 743-5 Bottom view of sampler.
 - 743-6 SWRP sample after removal from sampler (side view).
 - 743-7 SWRP sample (bottom view).
 - 743-8 SWRP sample (top view).
 - 1.3 The SWRP sample weighed 673 grams.
 - 1.4 The solid SWRP sample appeared uniformly grey on its surface. The cylindrical-shaped sample was cut lengthwise into halves using the edge of a spatula. One half was cut into upper and lower sections. The other half was cut into upper, center, and lower sections.
 - 1.4.1 Photographs:
 - 743-9 SWRP sample sectioned into five parts.
 - 743-10 Cross-section of SWRP near bottom showing inclusions.
 - 1.4.2 The SWRP sample had the "feel" of sodium in cutting, except that it was noticeably "gritty" and somewhat harder toward the bottom. Many white and dark inclusions appear within the sodium matrix. (As an example, see photograph 743-10).

1.4.3 The total weight is given in section 1.3. The weights of the various samples taken for analysis are given in the appropriate sections which follow.

2. Sample analysis.

2.1 XRD studies.

2.1.1 Qualitative. Photograph 743-12 shows typical mode of mounting a slice of SWRP sample in x-ray sample holder for XRD analysis. All sample handling was done in the glove box. The x-ray chamber was sealed in the box atmosphere, and quickly transported to the x-ray generator where the sample was scanned under dynamic vacuum. The following crystalline compounds (listed in order of descending peak intensity) were detected:

Sample Location* and Crystalline Phases Detected

Section #1	Section #2	Section #5 (bottom)
Na	Na	Na
Na ₂ O	Na ₂ O	Na ₂ O
NaOH	NaH	NaH
NaH	NaOH	NaOH

*Location refers to numbering system shown in photograph 743-9

2.1.2 Semiquantitative. Several SWRP samples were prepared for semiquantitative XRD analysis by warming to the melting point of sodium and grinding with an equal weight of Linde "A" alpha aluminum oxide having a one micrometer particle size. Similar standards were prepared using reagent grade sodium, sodium hydroxide, sodium oxide, and sodium hydride. Photograph 743-14 shows a typical preparation. The samples and standards were prepared in the glove box and sealed in the x-ray chamber in the box atmosphere as before. Semiquantitative results were obtained by comparing peak intensities of the components in question with those of the aluminum oxide common internal standard. The following results were obtained:

2.1.2 (Continued)

Constituent	Wt % Found in SWRP Sample*			
	Section #1 Center	Section #3	Section #5 Top	Section #5 Bottom**
Na	47	32	42	60
Na ₂ O	23	42	23	27
NaOH	27	18	31	10
NaH	3	7	3	3

* Refer to numbering system shown in photograph 743-9.

** Outer layer discarded.

2.2 Oxygen analysis by amalgamation.

Samples were taken from SWRP Sections #1, #3, and #5 for determination of oxygen by the standard amalgamation technique. (See photograph 743-15).

Results are as follows:

Sample Wt.	SWRP Sample	Wt % Oxygen by Amalgamation
5.500 g	Section #1	6.3
4.015	Section #3	6.3
3.020	Section #5	8.3

A large amount of oxide residue was observed to float on the mercury during the amalgamation. (See photographs 743-37 and 743-38).

2.3 Hydrogen analysis by amalgam reflux. Samples were taken from SWRP sections #2 and #4 for determination of hydrogen by the standard amalgam reflux techniques. (See photograph 743-16).

Results are as follows:

Sample Wt.	SWRP Sample	Wt % Hydrogen by Amalgam Reflux
2.782 g	Section #2 - top	0.1
2.090	Section #4 - center	0.09

-4-

- 2.4 Distillation of sodium. Two SWRP samples taken from sections #1 and #4 were trimmed of their exterior "skin" and were subjected to dynamic vacuum distillation in tantalum crucibles at pressures of 10^{-3} torr.

Photograph 743-23 shows SWRP sample from section #1 in tantalum crucible before distillation.

Photograph 743-17 shows SWRP sample from section #4 in tantalum crucible before distillation.

- 2.4.1 Sample weights:

SWRP Sample	Wt. (grams)
Section #1 - bottom	26.80
Section #4 - bottom	31.46

- 2.4.2 Residue weights after distillation:

SWRP Sample	Residue Wt. (grams)	Residue Wt. % of Sample
Section #1	17.50	65.3
Section #4	12.20	38.8

- 2.4.3 Photographs of residues:

743-24 shows residue from section #1.

743-20 shows residue from section #4.

Both residues appeared to be sponge-like and porous in nature. Several discreet chunks were removed from each residue. The residues could easily be reduced to a fine powder by gentle grinding in the tantalum crucibles.

Photograph 743-25 shows the chunks removed from the SWRP section #1 residue, and the residue after grinding to a fine powder.

Photograph 743-22 shows the chunks removed from SWRP section #4 residue.

Photograph 743-21 shows the SWRP section #4 residue after grinding to a fine powder.

- 2.4.4 XRD analysis of SWRP distillation residues. Semiquantitative XRD analysis of the distillation residues was accomplished using the aluminum oxide internal standard technique previously described.

SWRP Residue	Constituent	Wt. % Constituent
Section #1	Na ₂ O	89
	NaOH	11
Section #4	Na ₂ O	78
	NaOH	18
	Na	4

In addition, qualitative XRD analysis was done on the large chunks separated from section #4 distillation residue (photograph 743-22).

SWRP Section #4 Distillation Residue - Chunks

Constituent Found	Qualitative XRD
Na ₂ O	Major
NaOH	Minor
Na	Minor

3. Reactivity of SWRP with Solvents.

SWRP samples and distillation residues were reacted with denatured ethanol and Dowanol PM at room temperature in an argon atmosphere. Their reactions were compared with those of reagent grade sodium under the same conditions. Because of the uncertainty of reaction rates, the experiments were scaled down to approximately single gram samples and 30 mL of solvent.

3.1 Room temperature, no agitation.

The reactions of SWRP, distillation residues, and sodium with denatured alcohol and with Dowanol PM are summarized in the following tables:

3.1.1 Reaction with denatured ethanol.

The alcohol was J. T. Baker anhydrous reagent consisting of 95 per cent SDA 3-A alcohol to which has been added 5 volume per cent isopropyl alcohol. Water content was 0.4 weight per cent, determined by the Karl Fischer method. A volume of 30 mL was used in each case. SWRP and sodium samples were single, solid pieces. Distillation residue was ground to fine powder.

3.1.1 (Continued)

Material	Wt. taken (g)	Time for Dissolution (min)	Max. Temp. of Solvent (°C)	Remarks
SWRP	1.2	5	46	Dark colored magnetic residue remained which was 0.4 wt % of SWRP sample. Identified as alpha iron by XRD.
Distillation residue from SWRP Section #4	1.5	Vigorous reaction*	60	Dark colored magnetic residue, 2.6 wt % of initial sample. Identified as alpha iron by XRD.
Sodium, reagent grade	0.8	62	27	Clear solution resulted.

* Powdered distillation residue was added in small increments.

3.1.2 Reaction with Dowanol PM.

The Dowanol was Dow Chemical propylene glycol methyl ether. Water content was 0.3 weight per cent, determined by the Karl Fischer method. A volume of 30 mL was used in each case. SWRP and sodium samples were single, solid pieces. Distillation residue was ground to a fine powder.

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3.1.2 (Continued)

Material	Wt. taken (g)	Time for Dissolution (min)	Max. Temp. of Solvent (°C)	Remarks
SWRP	1.0	6.8	94	Dark colored magnetic residue remained which was 0.9 wt % of SWRP sample. Identified as alpha iron by XRD.
Distillation residue from SWRP Section #4	1.5	Fast reaction* (slightly slower than with alcohol	40	Dark colored magnetic residue remained which was 2.5 wt % of initial sample. Identified as alpha iron by XRD. A white precipitate was observed to form upon reaction. Precipitate identified as NaOH by XRD.
Sodium, reagent grade	1.1	>60	72	Clear solution resulted

* Powdered distillation residue was added in small increments.

Photographs 743-35 and 743-36 (scanning electron micrographs) show the microscopic nature of the dark colored magnetic alpha iron particles recovered from the SWRP. This material probably represents normal system debris.

- 3.2 Elevated temperature, no agitation.
- 3.3 Elevated temperature with agitation.
- 3.4 Water addition.

Since the SWRP and residue reactions proceeded faster than those of sodium in the solvents used, it was decided to limit the experimentation to conditions of room temperature and no water addition.

- 3.5 Solvent reactions with distillation residues.
- Data have been incorporated in section 3.1.1 and 3.1.2.
- 4. Melting characteristics of SWRP.

The behavior of SWRP was observed 1) in pyrex at temperatures at 550°F, and 2) in a tantalum cup at temperatures to 800°F. In both cases solid

4. (cont'd)

and liquid phases were observed to be present.

4.1 As-received SWRP.

Photograph 743-26 shows a 15.3 gram sample of SWRP in a pyrex beaker at a temperature of 550°F, the limitation of the hot plate. At this temperature the SWRP demonstrated a consistency like that of wet sand. It could be molded with a spatula and retain its shape. (See photograph 743-27, a closer look at the SWRP in beaker). Solid particles, sand-like in appearance, were observed throughout the SWRP. This heating was done in the helium atmosphere of a glove box.

Photograph 743-29 shows a 28.4 gram sample of SWRP which had been heated to 800°F in a tantalum cup in a static helium atmosphere at a pressure of about 700 torr. Again, two phases remained. Heating was carried out in temperature increments of 100°F, and the contents of the tantalum cup were examined visually, to the extent possible, through the glass walls of the containment vessel. The top crust was visible, and it appeared to undergo no change. There was no apparent outgassing of the SWRP during heating as indicated by the lack of any significant increase in system pressure.

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Figure B1. Head Section of SWRP Sampler (in glove box) (743-1)

LMEC-P38175

Figure B2. Top View Into Sampler (743-3)

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LMEC-P38176

Figure B3. Bottom View of Sampler (743-5)

LMEC-P38177

Figure B4. SWRP Sample After Removal From
Sampler (side view) (743-6)

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LMEC-P38178

Figure B5. SWRP Sample (bottom view) (743-7)

LMEC-P38179

Figure B6. SWRP Sample (top view) (743-8)

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Figure B7. SWRP Sample Sectioned Into Five Parts (743-9)

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Figure B8. Cross-Section of SWRP Near Bottom, Showing Inclusions (743-10)

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LMEC- P38182

Figure B9. SWRP Sample Mounted in X-ray
Diffraction Sample Holder (743-12)

LMEC- P38183

Figure B10. X-ray Diffraction Standard Preparation
(743-14)

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(11) 743-15

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Figure B11. Sections of SWRP, Showing Where Amalgamation Samples Were Removed (743-15)

(12) 743-37

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Figure B12. Amalgamation Analysis, Showing Oxide Residue Floating on Mercury (743-37)

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13) 743-38

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Figure B13. Close-Up View of Oxide Residue
on Mercury (743-38)

14) 743-16

LMEC-P38187

Figure 14. Samples (center) Taken From SWRP Sections
for Hydrogen Analysis (743-16)

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(15) 743-23

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Figure B15. SWRP Sample From Section No. 1 in Tantalum Crucible Before Distillation (743-23)

(16) 743-17

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Figure B16. SWRP Sample From Section No. 4 in Tantalum Crucible Before Distillation (743-17)

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Figure B17. Distillation Residue From Section No. 1 (743-24)

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Figure B18. Distillation Residue From Section No. 4 (743-20)

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LMEC-P38192

Figure B19. Distillation Residue From SWRP Section No. 1:
Separated Chunks in Mortar, Residue After
Grinding in Crucible (743-25)

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Figure B20. Chunks Removed From SWRP Section No. 4
Distillation Residue (743-22)

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LMEC-P38194

Figure B21. SWRP Section No. 4 Distillation Residue
After Grinding to Powder (743-21)

LMEC-P38195

Figure B22. Scanning Electron Micrograph of Magnetic
Particles Recovered From SWRP Solution
in Ethanol, 200X (743-35)

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LMEC-P38196

Figure B23. Scanning Electron Micrograph of Magnetic Particles Recovered From SWRP Solution in Dowanol PM, 200X (743-36)

LMEC-P38197

Figure B24. SWRP Sample (15.3 g) in Glove Box at 550°F (743-26)

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LMEC-P38198

Figure B25. Close-Up View of SWRP Sample
at 500°F (743-27)

LMEC-P38199

Figure B26. SWRP Sample (28.4 g) in Tantalum Cup
After Heating to 800°F (743-29)

LMEC-77-7