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REACTION OF SODIUM AND NaK
WITH MOBILTHERM LIGHT OIL

HANFORD ENGINEERING DEVELOPMENT LABORATORY
Operated by Westinghouse Hanford Company
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Refueling and Plant Programs

January 1975

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ABSTRACT

The compatibility of "Mobiltherm Light" heat transfer oil with sodium and NaK under simulated accident conditions was demonstrated. Kilogram quantities were mixed at various temperatures (to 800°F) in air and in nitrogen atmospheres. Chemical reactions between the oil and the liquid metals were shown to have negligible effects on the accident severity.

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I. INTRODUCTION

The auxiliary heat transport systems for the Fast Flux Test Facility (FFTF) employ a proprietary heat transfer oil, Mobiltherm Light (MTL)*. Since large quantities of MTL will circulate in close proximity to sodium and NaK, assurance must be provided that unintentional direct contact of oil with the liquid metals will not have unacceptable consequences. Tests were performed in the Large Sodium Fire Facility (LSFF) at Hanford Engineering Development Laboratory (HEDL) to verify that MTL does not react exothermally with sodium or NaK under hypothetical accident conditions.

MTL is not a specific chemical compound, but rather is a complex mixture of many organic compounds. The manufacturer does not identify the specific chemical species composing MTL, but states that MTL consists of a mixture of 70% to 90% aromatic hydrocarbons, with the remainder being aliphatic petroleum naptha⁽¹⁾. Straight chain hydrocarbon oils are generally considered to be unreactive with sodium or NaK, but compatibility of aromatic compounds with the liquid metals is not assured. Estimates of chemical reactions between MTL and the liquid metals based on thermodynamics are unreliable because the exact composition of the MTL is unknown.

Since MTL is a mixture of organic hydrocarbons, it does not have a definite boiling point. The manufacturer reports a boiling range of approximately 450°F to 700°F⁽¹⁾. Exposure of MTL to temperatures above 450°F at normal pressure will result in vaporization of the more volatile fraction of MTL. Sustained exposure at temperatures above 450°F will cause thermal decomposition to lower molecular weight materials, hydrogen and carbon⁽¹⁾.

The hypothetical plant accident conditions on which the test conditions were based involve spills of MTL and the liquid metal onto a cell floor in either a typical FFTF inert atmosphere (1% O₂, 99% N₂) or an air atmosphere at ordinary atmospheric pressure.

For this report all Figures are in Appendix A. Tables are included in the text material.

* Mobiltherm is a trademark of the Mobil Oil Corporation.

II. SUMMARY AND CONCLUSIONS

Four tests were performed in which two-pound quantities each of MTL and sodium or NaK were mixed under different conditions while temperatures and effluent gas composition were monitored. Post-test chemical analyses were performed to determine the extent of reaction. The test parameters included type of atmosphere (inert or air), method of mixing (rapid spill or slow heating from ambient temperature), and type of liquid metal (sodium or sodium 22% - potassium 78% alloy).

The physical properties of the MTL used in the test were measured to ensure that the test material had the same characteristics as that to be supplied for plant use.

The results of the tests support the following observations and conclusions.

- There were no detectable exothermic reactions between MTL and sodium or NaK for the conditions tested. If any reactions did occur, the thermal effects were insignificant compared to normal heat transfer processes.
- No products of reaction were identified by chemical or mass spectrometer analyses other than those expected due to reaction of the liquid metal with the atmosphere.
- The liquid metals and MTL were immiscible and formed separate phases. Sodium was covered by a layer of MTL which prevented oxidation of the sodium. On the other hand, NaK formed the top layer over the MTL and was thus exposed to the oxygen in the atmosphere.
- A significant fraction of MTL was evaporated in all the tests with sodium. The vaporized MTL partially condensed in cooler areas of the test apparatus.
- In the test where hot sodium was spilled rapidly into cold MTL, the sodium formed spheres ranging from 0.1 cm to 1 cm diameter and remained in the dispersed state after freezing.

- The MTL was pyrolyzed to a small extent ($\sim 0.1\%$) as evidenced by the formation of carbon particles.
- Cold MTL was ignited immediately by hot sodium spilling in an air atmosphere. The sodium was quickly submerged beneath the MTL and did not react until all of the MTL had been consumed by the fire. The sodium then oxidized completely to Na_2O and Na_2O_2 .
- The MTL burning in air generated a very dense black smoke. A layer of soot approximately 1/16 inch thick covered all surfaces of the test cell after the fire was over. Orange flames one to two feet in height were observed during the MTL fire. No flames were seen during the sodium oxidation portion of the test.
- Reaction between MTL and superoxides of sodium or potassium were not studied in these tests. Formation of superoxides during long-term exposure to the accident environment is possible and caution should be exercised during cleanup after an accident where MTL has contact with oxidized sodium or NaK.
- Pretest analyses of the MTL showed that the physical properties (viscosity, specific gravity, flash point, pour point, and boiling range) were in reasonable agreement with the manufacturer's product data sheets.

III. PRETEST CHARACTERIZATION OF MOBILTHERM LIGHT OIL

A one-liter sample was taken by pipette from near the surface of a 55-gallon drum of MTL and sent to Battelle Memorial Institute, Pacific Northwest Laboratories (PNL) for analyzing physical characteristics. The purpose of this analysis was to characterize the test material to assure similarity to future shipments for plant use. The analytical results are summarized in Table I, along with typical values listed by the manufacturer⁽¹⁾.

The agreement between the PNL analyses and the manufacturer's product data sheet is considered to be reasonable. The manufacturer states⁽¹⁾ that his listed values are "typical" or maximum values. The test material

TABLE I
PHYSICAL PROPERTIES OF MTL

	Manufacturer's Typical Values ^(a)	PNL ^(b) Analysis
Specific Gravity (60/60F)	0.98	0.986
Viscosity at 100°F, SUS	41.5	45.3
Viscosity at 210°F, SUS	32.5	31.7
Viscosity at 100°F, stokes	0.047	0.060
Viscosity at 210°F, stokes	0.016	0.013
Flash Point, °F	250 ^(c)	290
Fire Point, °F	(e)	315
Pour Point, °F	-20 ^(d)	-20
Color	dark	dark
Distillation, °F		
First drop	(e)	199
10% fraction	500	518
50% fraction	575	608
90% fraction	670	(f)

- (a) Ref (1)
- (b) See Appendix B for details.
- (c) Minimum value
- (d) Maximum value
- (e) Not reported
- (f) Not analyzed

was less volatile than the manufacturer reports, as evidence by the higher flash point (290°F compared to 250°F) and the higher distillation temperatures. The distillation data are plotted in Figure 1. All of the PNL analytical results are reported in Appendix B.

The specific gravity data were obtained by hydrometer, using the ASTM D-1298-67 procedure. Specific gravities as a function of temperature over the range 40°F to 255°F are plotted in Figure 2.

The flash point (290°F) and fire point (315°F) were determined by the Cleveland open cup technique described in ASTM D-92-66. The pour point (-20°F) was obtained by ASTM D-97.

The viscosity data were obtained from a Saybolt viscometer, using the ASTM D-88 procedure. The Saybolt universal seconds (SUS) data were converted to stokes by the following equation⁽²⁾:

$$\nu = 0.0022 (\text{SUS}) - \frac{1.8}{(\text{SUS})} \quad (1)$$

Where ν = kinematic viscosity, stokes

SUS = Saybolt universal seconds

The kinematic viscosity is plotted as a function of temperature in Figure 3. The graph is reproduced from the manufacturer's technical bulletin⁽¹⁾ and the PNL analytical results are shown as a broken line.

No analysis was made for water content, but a few visible drops of a separate phase were observed in the initial fraction during distillation. This probably explains the low temperature (199°F) observed for the first drop. After the first few drops were distilled, the temperature climbed rapidly to ~400°F. It is estimated that the volume of water was <0.1 cm³, which is <0.04% of the 240-g sample being distilled. No separate phase water was visible in the original sample, which was taken from the top of the 55-gallon drum. A second sample was drawn from the bottom edge of the drum after it had been in a tipped position for several hours. No separate phase water could be seen in the bottom sample.

The MTL was examined by infrared spectroscopy. The spectrum indicated a mixture of aromatic and aliphatic hydrocarbons. This agrees with the manufacturer's report⁽¹⁾. The IR spectrum showed no significant amounts of alcohol, carbonyl, or amine groups present.

IV. EXPERIMENTAL PROCEDURE

The test conditions for the four tests are summarized in Table II. In each test, the MTL and liquid metal were contained in an insulated 4-liter stainless steel beaker. For the three tests with an inert atmosphere, the reaction vessel was located within an inert atmosphere chamber that was maintained at 1% O₂ by providing a constant bleed of nitrogen gas. In the air atmosphere test (M4), the inert atmosphere chamber was not used and the reaction vessel was located in the center of the air-filled test cell. Figure 4 is a schematic diagram of the equipment arrangement.

The liquid metal was heated to the specified initial temperature in a small tank located outside the test cell. A trace-heated dump line connected the liquid metal dump tank to the reaction vessel. A shroud was provided to help contain the reactants in the event of vigorous splashing. The shroud extended seven inches above the top of the reaction vessel.

A photograph of the reaction vessel is shown in Figure 5. The five internal thermocouples extended 1.5 inches from the wall at various distances above the bottom of the beaker. Only in Test M1 was the electrical heating cable provided. The locations and recorder points for all of the thermocouples used in the tests are listed in Appendix C. The thermocouples were type K, 1/16-inch OD stainless steel sheath, with ungrounded junction. Readout was by multipoint recorders.

Figure 6 shows the insulated reaction vessel and shroud within the inert atmosphere chamber. The liquid metal dump line penetrated the side of the chamber and terminated vertically 7 inches above the bottom of the reaction vessel.

TABLE II
SUMMARY OF TEST CONDITIONS

Test No.	Method of Mixing and Heating	Liquid Metal			MTL		Atmosphere	Purpose
		Type	Mass (lb)	Initial Temp (°F)	Mass (lb)	Initial Temp (°F)		
M1-A	Dump LM rapidly into empty beaker ^(a)	Na	2.0	800	0	85 ^(b)	Inert	Determine thermal response in 1% O ₂ atmosphere.
M1-B	Slowly heat mixture electrically from ambient to 500°F	Na	2.0	70	2.1	70	Inert	Determine threshold temperature for any exothermic reaction
M2	Dump LM rapidly into MTL	Na	2.4	800	2.1	105	Inert	Effect of fresh, hot sodium mixed suddenly into MTL
M3	Dump LM rapidly into MTL	NaK	2.0	710	2.0	98	Inert	Compare NaK and sodium reactions with MTL
M4	Dump LM rapidly into MTL	Na	2.0	810	2.1	80	Air	Determine effect of air atmosphere on reaction

(a) Weight of beaker 1.53 lb plus 0.47 lb of heating cable

(b) Temperature of beaker

The quantity of sodium dumped was measured by observing the difference in liquid level in the dump tank. An inductance type liquid level probe inserted into a well in the tank was used for this purpose. The accuracy was $\pm 1/8$ inch, or ~ 0.25 lb of liquid metal.

The depth of 2 lb each of reactant in the 6-inch ID beaker at 210°F was 2.1 inches for MTL, 2.1 inches for sodium and 2.3 inches for NaK.

V. RESULTS

A. GENERAL RESULTS

The highlight results of the four tests are summarized in Table III. The chief conclusion drawn from the results of these tests is that any chemical reaction between MTL and sodium or NaK had an insignificant effect on the thermal behavior of the system. This conclusion is based on the following facts.

- The electrical heating rate in test M1-B at thermal equilibrium equalled the calculated heat loss rate assuming no chemical heat term.
- The measured temperature of the "reactants" was the same as predicted for adiabatic mixing in the absence of a chemical reaction.
- No exothermic peaks were observed during electrical heating in test M1.
- No gaseous reaction products were found by mass spectrometric analysis.
- Post-test chemical analysis showed that the sodium and NaK were only oxidized in amounts explainable by reaction with the atmosphere.

The calculated heat loss rate during the 30-minute steady state period at 500°F in test M1-B was 115 ± 25 watt. This value was calculated by summing losses by conduction through the beaker walls and insulation, radiation and convection from the top surface and evaporation of MTL, and ignoring any chemical heat of reaction. This is identical with the measured electrical heating rate of 115 ± 5 watt, confirming that chemical reaction heat was negligible.

TABLE III
SUMMARY OF TEST RESULTS

	Test M1	Test M2	Test M3	Test M4
Predicted Equilibrium temp (°F) ^(a)	(c)	378	275	336
Measured temp two minutes after spill (°F) ^(b)	(c)	362 ± 15	283 ± 15	380 ± 15
Maximum temp (°F) ^(d)	500	395	350	1550
Top layer (after cooldown)	MTL	MTL	NaK	MTL
Bottom layer (after cooldown)	Na	Na	MTL	Na
Atmosphere ^(e)				
Oxygen (mole %)	0.5-1.0	0.5-1.0	0.6-1.2	12-21
Hydrogen, max (mole %)	<0.1	<0.1	0.02	0.09
MTL remaining in beaker (%)	61.0	93.3	~100	0
Total LM remaining in beaker (%)	100	100	100	95
Percent of LM oxidized (%)	0.3	0.6	4 ± 2	~99
Detectable exothermic reaction between LM and MTL	no	no	no	no
Splashing from beaker	nil	nil	nil	minor

(a) Adiabatic heat transfer between MTL, liquid metal and SS beaker; no chemical reaction, no vaporization.

(b) Average of 4 thermocouples in beaker contents and one thermocouple on beaker exterior surface.

(c) Not applicable.

(d) Maximum attained by any thermocouple immersed in beaker contents.

(e) Sampled from inert gas chamber vent for tests M1, M2 and M3; from 6 inches above beaker top for test M4.

For the tests where hot liquid metal was spilled rapidly into ambient MTL, the resulting theoretical equilibrium temperature of the mixture was calculated by Equation (2).

$$T_{eq} = \frac{\sum^n m_i C_{p_i} T_{oi}}{\sum^n m_i C_{p_i}} \quad (2)$$

Where,

T_{eq} = equilibrium temperature

m = mass of individual species (i)

C_p = mean specific heat

T_o = initial temperature before mixing

n = number of unique thermal species

The assumptions on which Equation (2) are based are: adiabatic heat exchange, no chemical reaction, no vaporization of reactants, and no change of state. Table III shows that the measured temperatures shortly after the liquid metal spill in the inert atmosphere tests agreed with the predictions within the limits of experimental error.

The mean bulk temperature of the "reactants" contained in the reaction vessel is plotted in Figure 7 as a function of time for each of the four tests. This figure shows that only in tests M1-B and M4 did a positive temperature ramp occur. In test M1-B, this was caused by the electrical heaters. In test M4, the combustion of first, MTL and finally, sodium, in the air atmosphere provided sufficient chemical energy to cause the temperature transient.

Results for each of the tests are discussed in detail in the following sections of this report.

B. TEST M1 - HEATED MIXTURE OF SODIUM AND MTL

Test M1 was performed in two stages. In the first stage (M1-A), 2.0 lb of sodium was spilled into an empty cold reaction vessel in a 1% O_2 atmosphere. This phase of the test served to verify the experimental arrangement and procedure and also provided the specified quantity of sodium for the second stage of the test (M1-B).

After the sodium had cooled to 70°F, 2.1 lb of MTL was added. Although sodium is less dense than MTL at temperatures <100°F, the MTL remained on top of the sodium because the sodium adhered to the vessel walls. Figure 8 is a plot of the specific gravities of sodium, NaK and MTL as a function of temperature and shows that at temperatures greater than 100°F, the sodium is more dense and should form a bottom layer.

During the second stage of the test (M1-B), 285 watts of electrical power was applied to the vessel walls at time zero. The electrical power was maintained constant until the bottom contents of the vessel approached 500°F, at which time the power was reduced to hold the temperature at 500°F for an additional 30 minutes. The temperature and heater power are plotted as a function of time in Figure 9. No effect of an exothermic chemical reaction can be seen. The sodium apparently remained on the bottom, as evidenced by the isotherms shown by the two lower thermocouples (TC-1, TC-2) during sodium melting and freezing.

A significant amount of the MTL was vaporized during the high temperature stage of the test. A portion of the vapors condensed on cold surfaces of the inert gas chamber and leaked onto the floor of the test cell. Figure 10 is a view of the chamber after the test, showing the condensed MTL on the cell floor. It is estimated that about 50 grams of MTL had leaked onto the floor, or about 5% of the original quantity. A total of 582 grams of MTL (61% of the original) remained in the vessel at the end of the test.

Figure 11 shows a view of the beaker contents after cooling. Approximately 1.5 inches of MTL formed the top layer, with 2 inches of solid pack sodium on the bottom. A very thin film of sodium oxide floated on the MTL surface, and a small amount of spongy sodium extended as a column from the sodium layer to the surface near the center of the beaker.

The residual MTL was darker than before heating, due to a small amount of suspended carbon particles. The carbon was probably produced by thermal decomposition. The stainless steel walls of the reaction vessel reached a maximum temperature of 535°F at a point two inches above the MTL surface, well above the 450°F maximum recommended by the manufacturer. Some of the

residual oil from test M1-B was distilled for comparison with the original. The curves are plotted in Figure 12. It is obvious that the lighter (more volatile) fraction of the MTL is missing from the residual oil.

Mass spectrometric analysis of grab samples of the vent gas from the inert gas chamber showed <0.01% H₂, <0.01 CH₄, <0.05% C_xH_y, and <0.1 CO. This indicates that there was no significant reaction which produced low molecular weight gaseous reaction products. The mass spectrometer results for all the tests are shown in Table IV. Apparently, the volatile MTL fraction that had vaporized during heating had condensed in the gas sample line and thus escaped detection by the mass spectrometer scan. All of the peaks in the scans occurred at mass numbers corresponding to the normal gaseous components listed in Table IV. These results substantiate that no reaction had occurred between MTL and sodium.

TABLE IV
MASS SPECTROMETER ANALYSIS OF GAS SAMPLES

Sample No.	Sample Time (min.)	Mole (%)								
		CO ₂	Ar	O ₂	N ₂	CO	He	H ₂	CH ₄	C _x H _y
M1-1	31	0.01	0.03	0.44	99.5	<0.10	<0.01	<0.01	<0.01	<0.05
M1-2	43	0.01	0.03	0.49	99.5	<0.10	<0.01	<0.01	<0.01	<0.05
M1-3	74	0.01	0.03	0.43	99.5	<0.10	<0.01	<0.01	<0.01	<0.05
M1-4	96	0.02	0.04	0.73	99.2	<0.10	<0.01	<0.01	<0.01	<0.05
M2-1	2	<0.01	0.09	0.43	99.5	<0.10	<0.01	<0.01	<0.01	<0.05
M3-1	2	0.01	0.21	0.90	98.9	<0.10	<0.01	0.02	<0.01	<0.05
M4-1	23	4.25	0.97	14.5	80.1	<0.10	<0.01	0.09	<0.01	<0.05
M4-2	55	0.20	0.95	20.5	78.3	<0.10	<0.01	0.05	<0.01	<0.05
M4-3	80	0.23	0.93	21.1	77.7	<0.10	<0.01	0.05	<0.01	<0.05

C. TEST M2 - SPILL OF HOT SODIUM

In Test M2, 2.4 lb of 800°F sodium was dumped into 2.1 lb of 105°F MTL in an inert atmosphere. The equilibrium temperature predicted by Equation (2) was 378°F. The average temperature of the sodium, MTL and beaker was 362 ± 15°F two minutes after the spill, in good agreement with

the prediction. The temperature then cooled continuously, as shown in Figure 13. There was no flame or other exothermic gas phase reaction, as shown by the low temperature measured by TC-5, which was located two inches above the MTL surface.

The sodium spill lasted seven seconds. The average flow rate was 3 gpm through the 1/2-in., 35-mil wall, stainless steel tubing. The exit velocity was 6.5 ft/sec. The sodium plunged beneath the surface of the MTL and formed spheres ranging from 0.1 to 1 cm diameter, as shown in Figure 14. The mass of spheres extended to the bottom of the vessel and adhered to each other and to the vessel walls after cooldown. Reference to Figure 8 shows that at temperatures above the sodium melting point, the sodium is more dense than MTL. It is hypothesized that the sodium formed spheres due to surface tension and that the oil kept the spheres from combining to form a continuous phase.

No reaction between MTL and sodium could be detected by thermal behavior, chemical analysis or mass spectrometer scan of gaseous effluent.

D. TEST M3 - SPILL OF HOT NaK

Test M3 was performed in an identical manner to test M2 except that 2.0 lb of 710°F NaK-78 was used instead of sodium. The equilibrium temperature predicted by Equation (2) was 275°F. The average temperature of NaK, MTL and vessel immediately after the spill was $283 \pm 15^\circ\text{F}$, in good agreement. A slight amount of heat may have been generated by reaction of NaK with the 1% oxygen atmosphere. NaK, being less dense than MTL (see Figure 8), formed the top layer and was thus exposed to the atmosphere, whereas in the sodium tests, the oil protected the metal from oxidation. Approximately 4% of the NaK was found in oxide form after cooldown.

Figure 15 gives a plot of the temperature as a function of time. Again, no flame or vapor phase reaction was indicated by either TC-5 or mass spectrometer scan of the vent gas. Figure 16 gives a view of the NaK surface. A black film of approximately 1 mm thickness covered about three fourths of the surface, with bright silvery metal under it. The total depth of liquid was 4.25 inches. A patch of white oxide approximately 2 in. diameter floated

on the surface. Approximately 50g of friable, loosely adhering oxide was found below the surface, equivalent to 4% of the original NaK inventory. Samples of NaK and MTL were taken by a vacuum thief. The NaK layer averaged 30 ppm of oxide. The MTL infrared spectrum was similar to pre-test oil. A small amount of H_2 was found in the vent gas immediately after the spill (0.02%). This probably came from NaK reacting with moisture in the cabinet atmosphere.

It is concluded that chemical reaction between NaK and MTL was insignificant for the conditions of the test. Reactions between superoxides of potassium and MTL were not investigated. It is conceivable that superoxides might be formed during long-term exposure of NaK to cell atmospheres during cleanup after accidents. However, the oxides which formed in this test (probably monoxides) did not induce a reaction.

E. TEST M4 - SODIUM SPILL IN AIR ATMOSPHERE

Test M4 was performed in a manner identical to test M2, except that the sodium spill into MTL occurred in an air atmosphere. In the FFTF, all sodium and NaK containing equipment is located either within inerted cells or within air filled rooms which can be inerted by space isolation and nitrogen flooding ⁽³⁾. To maximize the effect that an air atmosphere might have on the presumed reaction between sodium and MTL, nitrogen flooding was not activated in this test and a normal air atmosphere prevailed in the test cell throughout the test.

As soon as the hot sodium (800°F) contacted the MTL, the MTL ignited, producing orange flames and copious black smoke. The flame flickered to heights up to two feet. Visibility was reduced to less than five feet within five minutes. After 50 ± 5 minutes, the MTL was entirely consumed and a transition occurred to sodium burning. The flames and black smoke were replaced by a glowing oxide surface with white smoke and a gradually improving visibility.

Figure 17 gives a plot of the temperature of the MTL and sodium as a function of time. The temperature of the air at various elevations above the vessel is shown in Figure 18. The temperatures in the atmosphere shown

in Figure 18 are probably much lower than those which would be registered if the entire cell floor were covered with burning MTL and sodium.

The temperature of the sodium (TC-1, TC-2) was 400°F immediately after the spill. Its temperature increased slowly due to heat conducted through the burning MTL until a maximum of 950°F was reached at 60 minutes. The temperature of the MTL (TC-3, TC-4) varied greatly with time and depth in the beaker. As the MTL was consumed, the level dropped and TC-4 became exposed to the combustion zone. A maximum temperature of 1500°F was recorded by TC-4.

The concentration of oxygen in the cell atmosphere was measured by pulling a continuous sample from a point 6 inches above the top of the beaker. Figure 19 shows that the O_2 concentration dropped rapidly to about 14% at the time of the spill and fluctuated between 12 and 15% for a period of 45 minutes. It climbed rapidly after the MTL fire was over. The average oxygen concentration in the 3300 ft³ test cell remained at essentially the normal 21% level because the cell was ventilated at ~1000 ft³/min.

Two hours after the spill, the sodium fire had self extinguished. The cell was entered and the beaker contents examined. The surface was covered by a crust of oxide, mostly white (Na_2O) but with some yellow patches (Na_2O_2). The thermal insulation was removed from the beaker and the contents were stirred. The sodium re-ignited and burned to completion during the next two hours.

At the end of the test, the floor of the test cell was covered by a layer of black soot particles approximately 1/16 inch thick. The vertical walls also had a thinner coating of soot. The soot was very light weight and air currents washed the soot into piles. The cell was cleaned satisfactorily by hosing with water.

VI REFERENCES

- (1) Mobil Technical Bulletin "Heating with Mobiltherm," C.R.I., R.G.M. 1-93-006, Mobil Oil Corporation, New York, N.Y.
- (2) J. H. Perry, editor, "Chemical Engineer's Handbook," 3rd ed., p.370 McGraw-Hill, New York, N.Y., 1950.
- (3) R. K. Hilliard, P. E. McPherson and J. M. Yatabe, "Sodium Fire Protection by Space Isolation with Inert Gas Flooding," in Proceedings of the Fast Reactor Safety Meeting, p. 431, held April 2-4, 1974 in Beverly Hills, CA, Conf-740401.

APPENDIX A
FIGURES

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HEDL 7501-124.3

FIGURE 3. Viscosity of Mobiltherm Light Oil as a Function of Temperature.

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FIGURE 4. Equipment Arrangement (Elevation).

745140-10

FIGURE 5. View of Reaction Vessel Showing Internal Thermocouples.

745221-2

FIGURE 6. View of Reaction Vessel Installed in Inert Gas Chamber.

FIGURE 7. Mean Reaction Temperatures During Four Tests as a Function of Time.

FIGURE 8. Specific Gravity of Sodium, NaK and Mobiltherm Light Oil as a Function of Temperature.

FIGURE 9. Temperature and Heater Power - Test M1-B.

745283-4

FIGURE 10. Post-Test View Showing Condensed Mobiltherm Oil on Cell Floor.

745283-2

FIGURE 11. Post-Test View of Beaker Contents - Test M1-B.

FIGURE 12. Comparison of Distillation Curves for Pre-Test and Post-Test Oil - Test M1-B.

FIGURE 13. Temperature as a Function of Time - Test M2.

64600-1

FIGURE 14. Sodium Spheres Formed During Test M2.

A-15

FIGURE 15. Temperature as a Function of Time - Test M3.

74H0570-1

FIGURE 16. Post-Test View of NaK Surface - Test M3.

FIGURE 17. Temperature of Beaker Contents as a Function of Time - Test M4

FIGURE 18. Temperature of Cell Air Above Reaction Vessel - Test M4.

FIGURE 17. Temperature of Beaker Contents as a Function of Time - Test M4

FIGURE 18. Temperature of Cell Air Above Reaction Vessel - Test M4.

A-19

FIGURE 19. Oxygen Concentration in Cell Air Near Reaction Vessel - Test M4.

APPENDIX B
PRETEST ANALYSIS OF MTL

Date September 25, 1974
To R. K. Hilliard, 3702 Bldg., HEDL
From E. C. Martin *E.C. Martin*
Subject Physical Properties of Mobil Therm Light Heat Transfer Oil

Reference: W.O. #C-43113, Ltr dtd July 31, 1974, from R. K. Hilliard
to E. C. Martin

Although this information has already been relayed to you by phone,
this letter will confirm that information and indicate the procedures
used for these analyses.

Flash Point, Fire Point

Flash Point - 290°F Fire Point - 315°F

Data obtained from Cleveland Open Cup Technique as described in ASTM-D-92-66.

Pour Point

Pour Point - -20°F

Data obtained from modification of ASTM D-97. The modification involved
substitution of a smaller Dewar flask for the 4-gallon constant temperature
bath indicated in the ASTM procedure.

Viscosity

Viscosity at 100°F - 45.3 SUS
Viscosity at 210°F - 31.7 SUS

Data obtained from ASTM D-88 procedure using Saybolt Viscometer

Specific Gravity

Specific gravity 60/60°F 0.980/24°C

Data obtained by hydrometer as indicated in ASTM D-1298-67.

An additional request was received for specific gravity values over
a range of temperatures. These were obtained using the hydrometer method
and heating the Mobil Therm in a suitable vessel in an oil bath. Great
care was taken to insure a homogeneous temperature throughout the sample.

<u>Specific Gravity</u>	<u>°C</u>	<u>°F</u>
0.992	5.2	41.4
0.980	24.3	75.7
0.970	37	98.6
0.951	65	149
0.938	82	179.6
0.923	103	217.4
0.916	113	235.4
0.908	124	255.2

See accompanying graph.

Distillation Data

Original Mobil Therm-Light

<u>Distillation Range</u>		<u>Temperature</u>	
<u>Wt</u>	<u>%</u>	<u>°C</u>	<u>°F</u>
1st drop		93	199
9.3 g	3.85	249	480
14.7	9.94	270	518
17.2	17.07	275	527
29.4	29.25	284	543.2
15.8	35.79	295	563
23.1	45.36	311	592
11.1	49.96	313	595.4
15.5	56.38	338	640

Mobil Therm Light After Test With Sodium

(TEST MI)
 RKH

<u>Distillation Range</u>		<u>Temperature</u>	
<u>Wt</u>	<u>%</u>	<u>°C</u>	<u>°F</u>
1st drop		232	450
13.9g	5.4	293	559
38.6	20.3	308	586
20.6	28.3	320.5	606
23.7	37.4	335	635
27.9	48.2	325.5	666
29.5	59.6	361	682
19.7	67.3	360	680

The temperature was determined at the intervals listed above using a mercury thermometer for the distillation of the original material and a voltmeter with an ice water cold junction for the material after the test with the sodium. All determinations were made at atmospheric pressure.

R. K. Hilliard
September 25, 1974
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Determination of Water

Visible drops of water were observed at the start of the distillation. It has been suggested that a sample of Mobil Therm taken from the bottom of the barrel be examined since the concentration of water would be maximized by this sampling technique.

Infrared Examination

Mobil Therm light was examined by infrared spectroscopy using a Perkin-Elmer Recording Spectrophotometer, Model 21. A thin film formed between two sodium chloride plates was the mode in which the examination was made.

The spectrum indicated a mixture of aromatic and aliphatic hydrocarbons. There were no significant amounts of alcohol, carbonyl, or amine groups present. A small peak at $1,036\text{ cm}^{-1}$ could be indicative of C-O-C linkages.

ECM:lm

APPENDIX C
THERMOCOUPLE LOCATIONS

Table C-1
THERMOCOUPLE LOCATIONS FOR INERT ATMOSPHERE TESTS

Thermocouple Number	Recorder Point			Thermocouple Location
	Test M1	Test M2	Test M3	
TC-1	F1	F1	F1	Inside beaker, 1" above bottom
TC-2	F2	F2	F2	Inside beaker, 1.75" above bottom
TC-3	F3	F3	F3	Inside beaker, 3" above bottom
TC-4	F4	F4	F4	Inside beaker, 3.75" above bottom
TC-5	F5	F5	F5	Inside beaker, 6" above bottom
TC-6	C1	C1	DR	Outside beaker, bottom center
TC-7	C3	C3	DB	Outside beaker, wall 7" high
TC-8	C5	C5	C5	Cabinet atmosphere, 12" above beaker top
TC-9	E1	E1	E1	Cabinet atmosphere, vent
TC-10	C7	C7	C7	Liquid metal dump line
TC-11	C9	C9	C9	Liquid metal dump tank

Table C-2
THERMOCOUPLE LOCATIONS FOR AIR ATMOSPHERE TEST (M4)

Thermocouple Number	Recorder Point	Thermocouple Location
TC-1	F1	Inside beaker, 1" above bottom
TC-2	F2	Inside beaker, 1.75" above bottom
TC-3	F3	Inside beaker, 3" above bottom
TC-4	F4	Inside beaker, 3.75" above bottom
TC-5	F5	Inside beaker, 6" above bottom
TC-6	F6	Outside beaker, bottom center
TC-7	C1	Atmosphere, 6" above beaker top
TC-8	C2	Atmosphere, 15" above beaker top
TC-9	C3	Atmosphere, 24" above beaker top
TC-10	C4	Atmosphere, 7 feet above beaker top
TC-11	C5	Atmosphere, 15 feet above beaker top
TC-12	C6	Atmosphere, 1" below beaker top, 6" from beaker
TC-13	A1	Liquid metal dump tank
TC-14	A2	Liquid metal dump line

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