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CALLERY CHEMICAL COMPANY

RESEARCH & DEVELOPMENT DIVISION • CALLERY, PA. • EVANS CITY 3510 • TWX-EVANS CITY 136

February 21, 1962

Aeronautical Systems Division
Air Force Systems Command
Wright-Patterson Air Force Base,
Ohio

Attention: Mr. M. W. Wannemacher ASRMFP-1

Gentlemen:

Enclosed are two replacement pages for Quarterly Progress Report No. 2 of Contract AF 33(616) 7224. Table IA was inadvertantly omitted when the report was assembled. Please replace page 10 of your copies of this report with the enclosed pages.

Very truly yours,

CALLERY CHEMICAL COMPANY

H. W. Wilson
H. W. Wilson

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Quarterly Report No. 2

Calorimetry

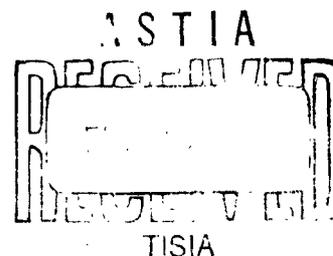
The high-temperature thermal properties of lithium metaborate, LiBO_2 , were found to have been measured by the Dow Chemical Company⁽¹⁾. The results of their measurements are shown in Table IA.

Drop-calorimetric measurements are being made on a sample of calcium silicide. The heat content, $H_T - H_{298}$, has been obtained to 470°C (878°F) and is shown graphically in Figure 3.

In the review of the current literature pertaining to thermal energy storage, it was found that Dow Chemical Company had reported the results of high-temperature thermal measurements on lithium metaborate. This was discovered before extensive measurements had been made, preventing unnecessary repetition.

The heat of fusion of lithium metaborate reported by Dow of 8092 ± 100 cal/mole or 293 BTU/lb is quite close to the estimated value of 310 BTU/lb based on entropy additivity considerations⁽²⁾.

The melting temperature reported by Dow of 844°C agrees with results of cooling curves run on a sample prepared at Callery from lithium hydroxide and boric acid.



(1) Dow Chemical Company, Quarterly Progress Report AR-2Q-61, Contract AF 33(616)-6149.

(2) Callery Chemical Company ASD Technical Report 61-187.

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Table IA
 HIGH TEMPERATURE THERMODYNAMIC PROPERTIES
 OF LITHIUM METABORATE - $\text{LiBO}_2^{(1)}$

T °K	C_p° cal/deg mole	$H_T - H_{298}$ cal/mole
298	14.29	0
300	14.36	26
400	16.99	1604
500	18.86	3400
600	20.41	5365
700	21.82	7478
800	23.16	9727
900	24.45	12108
1000	25.71	14616
1100	26.96	17249
1117 (s)	27.17	17709
1117 (1)	34.56	25801
1200	34.56	28670
1300	34.56	32130
1400	34.56	35580
1500	34.56	39040
1600	34.56	42490
1700	34.56	45950
1800	34.56	49410
1900	34.56	52860
2000	34.56	56320

(1) Dow Chemical Company, Quarterly Progress Report AR-2Q-61, Contract AF 33(616)-6149

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DETERMINATION AND ANALYSIS OF THE POTENTIALITIES
OF THERMAL ENERGY STORAGE MATERIALS

AF 33 (616)-7224

Quarterly Report No. 2



October 1, 1961 - December 31, 1961



Quarterly Report No. 2

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SUMMARY

Extensive tests have been conducted in an effort to find a metal suitable as a container for molten lithium metaborate (LiBO_2) at 1600°F . None of the metals tested thus far has been completely satisfactory. However, results to date indicate an increasing corrosion resistance with increasing chromium content of the alloys. Additional tests are scheduled with alloys containing a high percentage of chromium.

Drop-calorimetric measurements on calcium silicide have been started. The heat content, $H_T - H_{298}$ has been obtained to 470°C (878°F). Calorimetric work on the high temperature thermal properties of lithium metaborate (LiBO_2) will not be done since a survey of the recent literature revealed that these measurements have recently been made by the Dow Chemical Company.

The thermal conductivity apparatus has been modified several times to remedy difficulties that have been revealed during operation. A series of measurements has been made on molten sodium fluoride at 1020°C (1868°F). As a result of these measurements an approximate value of $5.7 \text{ BTU/hr ft}^2 \text{ }^\circ\text{F/ft}$ has been obtained for the thermal conductivity of molten sodium fluoride. Attempts to repeat and confirm this value have been delayed due to burn-out of the heating element caused by failure of the graphite crucible to contain the molten sodium fluoride. Installation of a new crucible and heating element is underway.

Construction of the furnace-calorimeter apparatus for measurement of heat release rates was completed except for the nickel calorimeter block. Delivery of the nickel stock was delayed and is now due the week of February 5, 1962. Testing

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DISCUSSION OF RESULTS

Material Compatibility:

A program is being conducted to find metals suitable for containing molten thermal energy storage materials during experimental measurements. A suitable container material must satisfy the following requirements: (1) no leakage through container walls and (2) no contamination of the storage material from the metal container. The program is to include lithium metaborate (LiBO_2), magnesium silicide (Mg_2Si), and calcium silicide (CaSi).

Lithium Metaborate

Although extensive tests have been conducted, none of the metals tested was found to be a suitable container material for molten lithium metaborate at 1600°F . Based on the results of these tests other metals have been selected for testing.

Lithium metaborate has been synthesized in high purity from lithium carbonate and boric acid according to the following reaction:



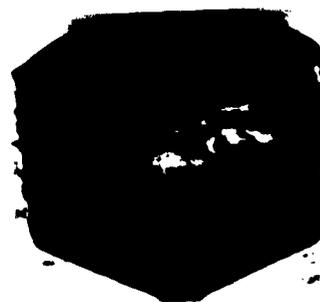
Stoichiometric quantities of these materials are mixed and heated to 1600°F in a porcelain dish. Porcelain is useful in the preparation of lithium metaborate where relatively short contact is required; however, porcelain is severely attacked after only a few hours (see Figure 1). Vycor is also severely attacked in a short time.

FIGURE I



Left: Porcelain Crucible with lithium metaborate, 16 hours.

Right: Magnesium silicide Test No. 1, 316 stn. stl. 35 hours in air.



Left: Metal test containers and lithium metaborate, before and after testing.

Right: Magnesium silicide Test No. 2, 316 stn. stl. 48 hours in argon (cross section view).

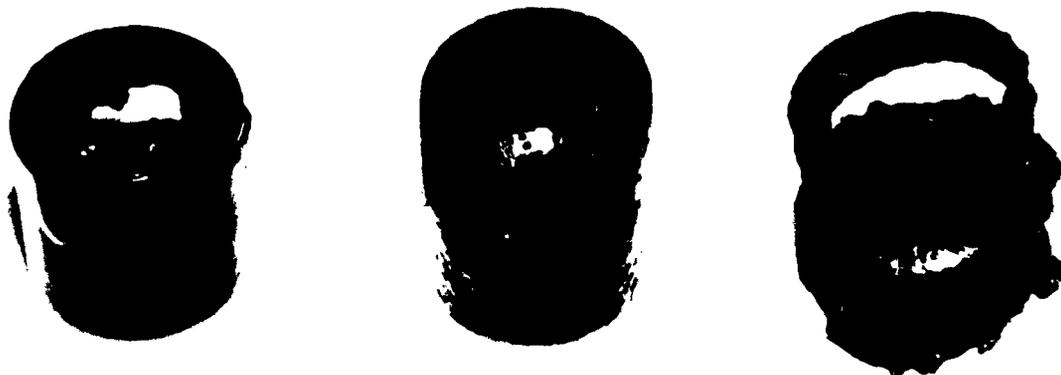
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As a result of the incompatibility of lithium metaborate with ceramic materials, the approach to these studies was revised. Instead of suspending metal coupons in the molten storage material in a ceramic container, test capsules were made from each of the metals studied. Initially a container consisting of a short length of pipe or tubing with a welded bottom plate was considered; however, this had two disadvantages: (1) the relatively thick walls required a long time for leakage to occur even though attack was occurring and (2) when leakage occurred it was difficult to determine whether it was through the wall or through the weld. As a result, the container geometry chosen for these tests is a thin-walled, weldless crucible machined from rod stock of each test metal. A 7/8 inch diameter hole is drilled in one inch diameter rod, and the outside diameter is machined to give a 15 mil wall thickness.

The tests made thus far are listed in Table 1. The test metals include four stainless steels, five high nickel alloys, Hastelloy X, and carbon steel. All of these were tested in air, and some of the most promising were tested in argon atmosphere. In most of these tests some leakage occurred, and in all cases the lithium metaborate was contaminated by the metal. In many cases the walls were badly blistered. Photographs of some of these test containers are shown in Figure 2.

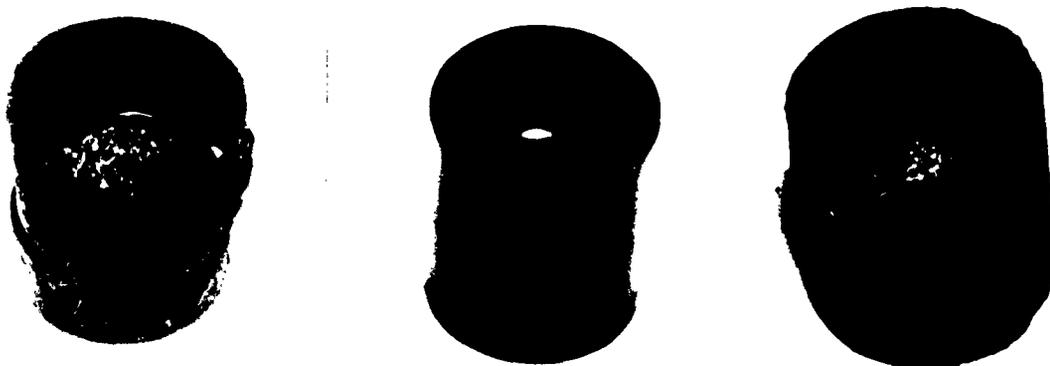
Type 310 stainless steel was the most corrosion resistant of the metals tested; type 309 stainless steel, Nionel 825, and Incoloy 800 were also only moderately attacked. These materials differ from the others mainly in their higher chromium content. In addition, type 310 stainless steel contains the highest chromium (25 percent). For this reason the next series of tests will

FIGURE II



Lithium Metaborate Tests (left to right)

Test Number 1 304 stn. stl. Air 20.5 hours.
Test Number 8 Nionel 825 Air 67 hours.
Test Number 10 Carbon Steel Air 16 hours.



Lithium Metaborate Tests (left to right)

Test Number 14 316 stn. stl. Argon 50 hours.
Test Number 17 310 stn. stl. Argon 70 hours.
Test Number 18 Hastelloy X Air 164 hours.

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include alloys containing up to 30 percent chromium, with relatively high nickel content. It is believed that Nionel 826 (30.0 Cr, 51.5 Ni), Incoloy 804 (29.3 Cr, 42.6 Ni), and type HP-stainless steel (28-32 Cr, 29-31 Ni) will be representative of this group of alloys. Haynes Metal 25 and Fansteel 80 will also be tested.

A sample of lithium metaborate held in Inconel 600 for 17 hours at 1600°F was submitted for chemical analysis to determine whether lithium nitride (Li_3N) is being formed and to check qualitatively what metallic impurities are present. No lithium nitride was detected. Iron and nickel were present in relatively large amounts; manganese and aluminum were also definitely present. Chromium was present in a small amount, and copper and carbon were not detected. It appears, therefore, that the attack is not selective except for the relative absence of chromium.

The metals now being considered for testing are not readily available from stock, and delivery will be several weeks on the high chromium alloys and the Fansteel 80. The Haynes Metal 25 is on hand, but special machine tools must be obtained to make the test containers. As a result of these delays, emphasis was shifted to the other thermal energy storage materials.

Magnesium Silicide

Compatibility tests between magnesium silicide and construction materials at 2250°F have been started. Commercial grade magnesium silicide from Var-Lac-Oid Chemical Company is being used. The purity of this material, based on X-ray analysis and elemental analysis, is very low; impurities are largely magnesium and magnesium oxide. Efforts are being made to find a source of high purity magnesium silicide.

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Two tests were made, both with type 316 stainless steel. The first test, for 35 hours in air, resulted in severe corrosion. In the second test, for 48 hours in argon atmosphere, the top part of the container was severely attacked; but near the bottom of the thin-walled section there was an irregular line below which the corrosion seemed to be negligible or totally absent (see Figure 1). After both tests the magnesium silicide consisted of two different layers. On the bottom was a gray packed powder, and on the surface was a hard lumpy white material. Both of these layers, however, contained principally magnesium oxide.

All future tests will be conducted in an inert atmosphere to prevent oxidation of the magnesium silicide at high temperature. If type 316 stainless steel is not suitable, the next metals to be tested are Incoloy 800 and Nionel 825, followed by type 310 stainless steel and Inconel 600. These were the metals which appeared to best withstand the required temperature based on a blank test using thin disks of the candidate metals.

Calcium Silicide

Commercial grade calcium silicide has been obtained from Var-Lac-Oid Chemical Company for use in compatibility tests. This material is also relatively low purity, although the supplier claims this to be the highest commercially available purity.

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Future plans in the materials compatibility program are as follows:

- (1) Continue lithium metaborate tests when test metal specimens arrive.
- (2) Try to obtain commercial magnesium silicide and calcium silicide of higher purity.
- (3) Continue testing commercial grade magnesium silicide, supplemented by chemical analyses, to further evaluate the effects of the second test. This should determine whether the magnesium silicide or an impurity is causing the attack.

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TABLE I

METAL COMPATIBILITY TESTS-LITHIUM METABORATE AT 1600°F

Test No.	Test Metal	Atmosphere	Test Duration Hr.	Result
1	304 S.S.	Air	20.5	Severe attack. Large hole in container wall.
2	316 S.S.	Air	65	Bad attack. Cracking in walls.
3	Monel 400	Air	17	Severe attack. Much metal in solution.
4	Nickel 200	Air	17	Severe attack. Some metal in solution.
5	Inconel 600	Air	17	Moderate attack. Contamination from metal.
6	Inconel 600	Air	17	Severe attack. Walls badly blistered.
7	Incoloy 800	Air	17	Moderate attack. Contamination from metal.
8	Nionel 825	Air	67	Severe attack. Walls badly blistered.
9	Incoloy 800	Air	75	Moderate attack. Some blistering on walls.
10	Carbon Steel	Air	16	Severe attack. Completely destroyed container.
11	Nionel 825	Argon	26.5	Moderate attack. Contamination from metal.
12	Incoloy 800	Argon	49	Indefinite. Contaminated with ceramic from furnace plug.
13	Incoloy 800	Argon	23	Moderate attack. Scale on walls.
14	316 S.S.	Argon	50	Severe attack. Walls totally damaged.
15	309 S.S.	Air	65.5	Moderate attack. Contamination from metal.
16	310 S.S.	Air	65.5	Moderate attack. Contamination from metal.
17	310 S.S.	Argon	70	Moderate attack. Contamination from metal.
18	Hastelloy X	Air	164	Severe attack. Walls badly blistered. Contents all leaked out.

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Calorimetry

The high-temperature thermal properties of lithium metaborate, LiBO_2 , were found to have been measured by the Dow Chemical Company⁽¹⁾. The results of their measurements are shown in Table II.

Drop-calorimetric measurements are being made on a sample of calcium silicide. The heat content, $H_T - H_{298}$, has been obtained to 470°C (878°F) and is shown graphically in Figure 3.

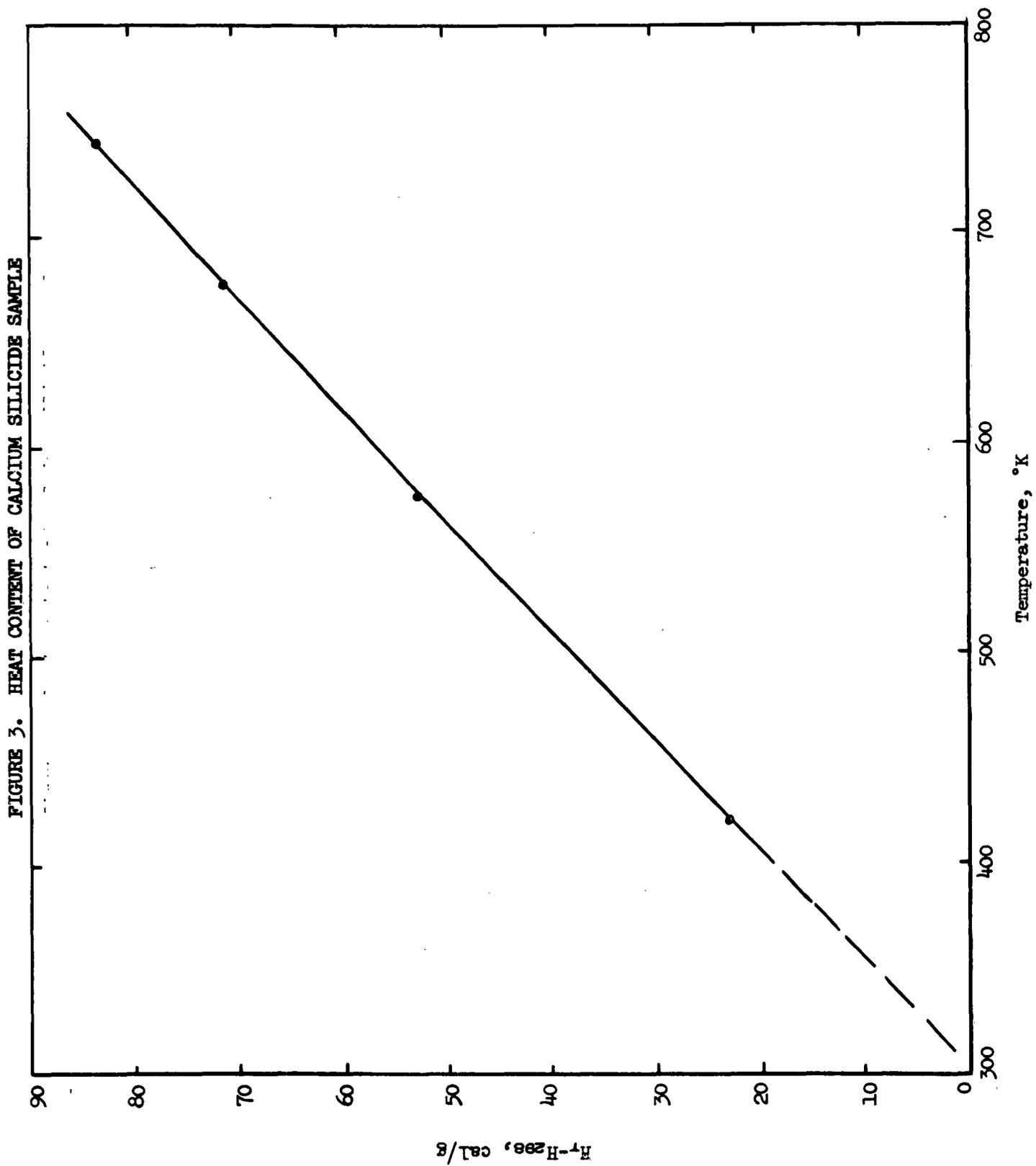
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The heat of fusion of lithium metaborate reported by Dow of 8092 ± 100 cal/mole or 293 BTU/lb is quite close to the estimated value of 310 BTU/lb based on entropy additivity considerations⁽²⁾.

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(1) Dow Chemical Company, Quarterly Progress Report AR-2Q-61, Contract AF 33(616)-6149.

(2) Callery Chemical Company ASD Technical Report 61-187.



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Drop-calorimetry measurements have been started on a sample of calcium silicide (Varlacoid Chemical Company), reported by the supplier to be principally CaSi. The sample, in the form of a powder of approximately 200-300 mesh, is contained in a platinum capsule. Chemical analyses of the calcium silicide sample are being made, but are not yet complete.

Several heat content measurements were made on the empty platinum capsule before loading with the sample. The results of these measurements, shown in Figure 4, agree satisfactorily with the reference values for pure platinum⁽³⁾.

The measurements on the calcium silicide sample have been completed up to 470°C (878°F). The heat content of the platinum capsule was subtracted from the gross heat content, yielding the heat content of the calcium silicide sample. The results of the measurements thus far are shown graphically in Figure 3.

Future Plans

Calorimetric measurements will be completed on the calcium silicide sample, to give thermal data through the melting transition.

Similar measurements will be made on a sample of magnesium silicide.

(3) K. K. Kelley, Bureau of Mines Bulletin 584, 1960

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Thermal Conductivity

The thermal conductivity furnace has been modified several times in order to remedy defects in design that have been revealed during operation. The extreme corrosivity of molten sodium fluoride plus the inherent difficulties of high temperature experimentation have made it necessary to use this method of arriving at a workable apparatus.

Before the latest changes were made, a series of measurements was made on molten sodium fluoride at 1020°C (1868°F). The temperature profiles of the sample and of the outer layer of insulation were measured at two-centimeter intervals with platinum - 6% rhodium vs platinum - 30% rhodium thermocouples. The profile indicated that the temperature of the sample decreased gradually toward the top. Also, the radial temperature drop across the insulation remained fairly constant over the lower section then increased markedly toward the top.

When the heater at the top of the sample was turned on, and equilibrium established, the temperature profiles were again determined. The effect on the sample profile was immediately evident in that the vertical temperature gradient was reduced to about one-third its value without the heater. The vertical gradient due to the heat added at the top was determined by correcting for the initial residual gradient. By assuming that a residual temperature gradient did not seriously affect the measurements, and that the change in temperature gradient, due to the heat added at the top, was constant, a value of 5.7 BTU/ft² hour-°F/ft was obtained for the thermal conductivity of molten sodium fluoride. This is to be considered as only an approximate value because of the assumptions involved in its calculation.

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Some of the more significant modifications to the apparatus are listed.

1. Crucible

The graphite crucible has been made 4 inches deeper to reduce the possibility of the melt overflowing, and to reduce the heat loss out the top. Graphite plugs were made for both the top and bottom of the crucible to hold the graphite thermocouple well vertical, and to reduce the heat loss out the top. The two heater windings on the crucible were increased to three to provide better control of the temperature gradient in the sample. This was found to improve the control considerably.

2. Outer furnace

The upper section of the furnace was extended another 7 inches, making a total of 18 inches of firebrick and Sil-O-Cel insulation above the top of the crucible. A wall of 4-inch firebrick was laid up without mortar outside the transite pipe. The space in the corners was packed with Kac-wool. As a result, the heat loss out the sides and top of the furnace has been reduced considerably, and much better control of the temperature gradient is possible.

Argon gas is now directed close to the top of the furnace through a stainless steel tube.

3. "D.C." heater

The small heater which sets up the temperature gradient in the sample has caused much trouble. It is necessarily compact, movable vertically, and must be supplied with current through leads at least 18 inches long. At high temperatures it is subject to stresses which lead to shorts and burn-outs.

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The original nichrome wire heater was replaced with a platinum wire to resist traces of sodium fluoride which seep through the graphite wall of the "D.C." heater. After the platinum burned out it was replaced with a flat spiral Kanthal ribbon as is used on the crucible. This was accidentally burned out when a defective transformer was used to supply it. Another attempt will be made to use a Kanthal heater.

4. Thermocouples

The platinum-rhodium thermocouples have repeatedly failed recently. There is evidence that the metal has crystallized and become brittle after service at high temperatures. Since a graphite thermocouple well is used in the sample, a chromel-alumel thermocouple would not be exposed to attack by the sodium fluoride, and should be satisfactory. In fact, the output of a chromel-alumel couple is 12 times that of a Pt-6% Rh vs Pt-30% Rh couple, and is preferable.

* * *

After a total of 140 hours of operation involving three heats, the molten sodium fluoride penetrated the walls of the high density graphite and caused the heater windings to fail. The graphite becomes porous after prolonged heating. Cooling sodium fluoride in the crucible may cause it to crack also. Furthermore, as the temperature of the sodium fluoride is increased much above its melting point its viscosity and surface tension probably are decreased enough to allow the melt to penetrate the graphite.

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For these reasons, it may be expedient to postpone measurements on molten sodium fluoride and try to measure the thermal conductivity of solid sodium fluoride at temperatures from 800 to 900°C. Many of the problems now present would be eliminated, and the results would be helpful in estimating other thermal conductivities. The experience in actual determinations would also be valuable for later work at higher temperatures.

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Density

The density of molten lithium metaborate was determined over the temperature range 947 to 1032°C, by the method of hydrostatic weighing. The results are listed in Table II . Densities of solid lithium metaborate at room temperature are also listed in Table II . It should be noted that the value for the specific gravity at 14.7°C is listed as 1.397 in the brochure published by Foote Mineral Company. The higher values obtained here are more consistent with the observed density of the molten salt. This discrepancy will be investigated further.

The lithium metaborate, prepared from lithium carbonate and boric acid, was melted in a platinum crucible. A platinum sphere, 1 cm. in diameter was weighed in argon just above the melt, then weighed immersed in the melt. The results, by this method of hydrostatic weighing are presented in Table II .

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TABLE II
DENSITY OF LITHIUM METABORATE

<u>Temperature</u> <u>°C</u>	<u>Density</u> <u>g/ml</u>	<u>Remarks</u>
947	1.87	Liquid, by hydrostatic weighing
970	1.87	Liquid, by hydrostatic weighing
992	1.86	Liquid, by hydrostatic weighing
1032	1.85	Liquid, by hydrostatic weighing
25	2.17	Approximate calculation of volume of cake in platinum crucible
25	2.24	Anhydrous LiBO_2 powder from Foote Mineral Company method of displace- ment of CCl_4
25	2.29	LiBO_2 glass prepared at Callery Chemical Company. Method of dis- placement of CCl_4 .

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Experimental Heat Release Rate Measurements

The objective of this part of the program is to determine and compare heat release rates of candidate thermal energy storage materials as an aid to design optimization of thermal energy storage systems.

Construction of the furnace-calorimeter apparatus was completed except for the nickel calorimeter block. Delivery of the nickel stock was delayed, and is now due on 7 February 1962. After delivery, an additional four weeks will be required to machine the rough cut nickel stock.

Upon completion of furnace construction, testing was started by heating slowly to the required temperature. When the temperature reached 2270°F, a short circuit between the heating element and the lead wire resulted in burnout of the heating element. The bottom lead wire ran through the insulation parallel to the heating element to the top of the furnace. In this position the electrical insulating properties of the thermal insulation and porcelain protection tube were inadequate to prevent the short circuit near the center of the furnace. The heating element will be rebuilt with the design modified to remove the lead wire at the bottom of the furnace.

Since the delivery of the nickel stock was delayed, the rebuilding of the furnace heating element will not affect the over-all construction completion date. The difficulty in finding a suitable container material for lithium metaborate will, however, delay the fabrication of test capsules and thus delay the start of experimental measurements. If a suitable container material is first found for magnesium silicide or calcium silicide, that material will be the subject of initial measurements while compatibility tests are continued on the others.

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