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TECHNICAL MEMORANDUM

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COMPILATION OF THE PROPERTIES OF LITHIUM HYDRIDE

By Roger L. Smith and James W. Miser

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Cleveland, Ohio

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
WASHINGTON January 1963

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

TECHNICAL MEMORANDUM X-483

COMPILATION OF THE PROPERTIES OF LITHIUM HYDRIDE*

By Roger L. Smith and James W. Miser

SUMMARY

The properties of lithium hydride, as published in the classified and the unclassified literature through February 1962, are presented. There are five main categories: chemical, physical, nuclear engineering, and thermodynamic properties, and manufacture and related subjects.

INTRODUCTION

Lithium hydride has properties of interest in many technologies. It is a salt-like white crystalline substance (face-centered cubic) physically resembling a halide and considerably more dense than lithium metal.

In the field of nucleonics, the high hydrogen content and light weight of lithium hydride make it an excellent candidate for the neutron shield and neutron moderator material in lightweight nuclear powerplants. For thermodynamic uses, the high heat of fusion of lithium hydride combined with its light weight makes it a good material to consider as a heat storage medium for solar powerplants on satellites. Another possible application is that of an expendable heat sink for the cooling of hypersonic aircraft (ref. 1(U)), again because of its light weight, and also because of its high heat of dissociation. These uses of lithium hydride require that its properties be known for design purposes, as well as for manufacture, handling, and containment. Of course, there are many other uses for lithium hydride, especially in the field of chemistry.

It was found that most of the available unclassified information is well summarized in the survey (ref. 2) by Dr. Charles E. Messer, done under a contract with the Atomic Energy Commission. However, sufficient additional information was found in both the classified and unclassified

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*Title, Unclassified.

literature to warrant the publication of this report. A large part of the report is composed of quotations from the references, a great many of which are unclassified.

Material is included from 267 references; wherever practical, primary references have been consulted. A portion of the data found in some of these references on the deuteride and the tritide of lithium is included in this report, but no attempt was made to cover thoroughly the properties of these materials. References 3 to 11 (all unclassified) and 12 (SRD), containing information similar to that mentioned herein, should be noted at this point. These references might be more readily available to the reader than those used in the text.

The report is divided into five parts: I. Chemical Properties, II. Physical Properties, III. Nuclear Engineering Properties, IV. Thermodynamic Properties, and V. Manufacture and Related Subjects. There is also an index.

The authors wish to thank the publishers and authors of many of the references for their kind cooperation, both in giving permission to quote and in furnishing additional data.

I. CHEMICAL PROPERTIES

I. CHEMICAL PROPERTIES

A. REACTIONS

1. Formation of Lithium Hydride (see also section V.A. entitled PREPARATION)

> "Lithium hydride can be prepared by reaction of lithium metal with hydrogen. The hydrogenation becomes appreciably rapid at temperatures near 600°C. Usually the reaction is carried out in iron vessels designed to be heated to the required high temperature and which can be kept under a reasonable pressure of hydrogen." (Ref. 13a(U))

Perlow made a study of the rate of reaction of hydrogen with lithium (ref. 14(U)). Her remarks regarding her results are as follows:

> "The most outstanding aspects of this rate study are that a reaction between lithium and hydrogen did take place at a temperature as low as 29° C. and that in those cases where the reaction temperature was 99° C. and 125° C. and the time was sufficient, the percentage of lithium hydride formed was as great as 60% and 85%. A careful inspection of the curves in Figure I-1 will show that the reaction rate for any given temperature varies due to the difference in surface area and condition of the surface of the lithium crystals. In the preparation of the recrystallized lithium the procedure followed was standardized as much as possible, but even then, it was not possible to reproduce crystals having exactly the same surface characteristics.
>
> "The writer's value for the reaction rate at room temperature is of the same order of magnitude as that reported by Remy-Gennette for the reaction between hydrogen, and lithium freshly distilled in the absence of air. Curve (2) in Figure I-1 is based on Remy-Gennette's data . . . at 22° C., and curve (1) on the writer's data at 29° C."

Note that Perlow does not give the temperatures at which the other curves were obtained, but they give a good indication of the order of magnitude of the time of reaction between lithium and hydrogen.

Reference 15(U) reports a rate of uptake of hydrogen of 1.5 cubic centimeters of hydrogen per square centimeter of surface per second at 1 atmosphere and 680° C.

Another study of the rate of reaction of hydrogen with lithium at temperatures between 29° and 250° C is reported in reference 16(U). The rate increased with temperature, and several days were required for complete reaction at the lower temperatures in this range. The variation of the reaction rate constant k with temperature was found to be: $\log k = -1380/T + 2.09$. This gives a heat of activation of 6300 calories per mole of hydrogen.

2. Reaction with Water and Air

> "The finely powdered hydride reacts rapidly with air of low humidity, forming $LiOH$, Li_2O, and Li_2CO_3. In moist air the powder may ignite spontaneously, when it burns fiercely, forming a mixture of products including some nitrogenous compounds. The lump material reacts with humid air forming a superficial coating which is a viscous fluid. This inhibits further reaction, and several hours' exposure of a one-inch cube does not cause appreciable loss of hydrogen therefrom, although the appearance of a film of 'tarnish' is quite evident. Little or no nitride is formed on exposure to humid air. The lump material, contained in a metal dish, may be heated in air to slightly below 200°C. without igniting, although it ignites readily when touched by an open flame. The condition of the hydride surface, presence of oxides on the metal dish, etc., have a considerable effect on the ignition temperature. Perfectly dry oxygen does not react with crystalline LiH unless heated strongly, when an almost explosive combustion occurs.
>
> "Both the powder and lump material generally ignite when moistened, the former sometimes giving a dust-explosion of some violence. Air-free steam reacts rather more slowly than might be expected, owing to the protective film which forms on the hydride.
>
> . . . .
>
> "The reaction with water appears to give lithium hydroxide at ordinary temperatures and possibly lithium oxide at higher temperatures by analogy with calcium hydride, which above 350° C. yields CaO rather than $Ca(OH)_2$ [ref. 17(U)]." (Ref. 2q(U))

The exposure of lithium hydride to moist air is reported as follows:

> "(1) Pellets of LiH exposed to moist air [react with the water and therefore] grow in dimensions and gain in weight at a rate proportional to the square root of the time of exposure.
>
> "(2) Dimensional increases appear to be proportional to the pressure. The pressure dependence of the weight increases is obscured by a density effect, and may be higher than first power. . . .
>
> "(3) Dimensional increases are independent of the bulk density of the pellet; weight increases have been shown semiempirically to be a function of $(\rho_0 - \rho)/\rho_0$ [where ρ_0 = crystal density of LiH and ρ = observed bulk density of the test specimen]. Linear dimensional increases are independent of the pellet area, whereas weight increases are proportional to the area.
>
> "(4) Different reaction mechanisms are found in different water vapor pressure ranges. At low pressures probably:
>
> $$LiH + H_2O \rightarrow LiOH + H_2$$
>
> At high pressures:
>
> $$LiH + H_2O \rightarrow LiOH + H_2$$
>
> $$LiOH + H_2O \rightarrow LiOH \cdot H_2O$$
>
> . . . .
>
> "(5) It is possible actually to slow down the reaction by raising the temperature within certain limits. Increasing the temperature from 24°C to 35°C raises the decomposition pressure of $LiOH \cdot H_2O$ and thereby moves the upper limit for the low pressure range reaction mechanism from 3.6 mm to 8.6 mm. Since the low range mechanism has a lower specific rate than does the high range mechanism, a rise in temperature from 24°C to 35°C in this pressure range will decrease the reaction rate.
>
> "(6) Pellets [1/2-in. by 1/2-in. cylinders] may be handled in air containing 2 mm H_2O vapor for as much as 24 hours without gaining as much as 1 mil in their linear dimensions." (Ref. 18(SRD))

"Exposure of lithium hydride to moist air at room temperature leads to the production of hydrogen in two stages:

(1) Fast at room temperature
$LiH + H_2O \rightarrow LiOH + H_2$ $< 15\%$ humidity
$LiH + 2H_2O \rightarrow LiOH \cdot H_2O + H_2$ $> 15\%$ humidity

(2) Slow at room temperature
$LiH + LiOH \rightarrow Li_2O + H_2$
$3LiH + LiOH \cdot H_2O \rightarrow 2Li_2O + 3H_2$"

(Ref. 19a(CRD))

See Section I.A.5. for reactions of lithium hydride with nitrogen.

3. Reactions with Acidic Substances

Acetylene:

"Lithium hydride reacts with acetylene to form lithium carbide and hydrogen [ref. 10(U)]." (Ref. 2r(U))

Acids, anhydrous organic:

"Anhydrous organic acids, phenols, acid anhydrides react slowly with lithium hydride, for the most part, giving hydrogen gas and the lithium salt of the acid." (Ref. 2r(U))

Acids, aqueous solutions of:

"Lithium hydride reacts more rapidly with aqueous solutions of acids than with water." (Ref. 2q(U))

Anhydrides, acid: see Acids, anhydrous organic

Carbon dioxide:

"In the presence of traces of moisture, it [lithium hydride] reacts slowly with carbon dioxide at room temperature, forming the carbonate.

. . . .

[redacted]

> "With dry carbon dioxide, the following reactions were reported by Moissan [ref. 20(U)]:
>
> $LiH + CO_2 = HCOOLi$; $2LiH + CO_2 = Li_2C_2O_4 + H_2$"
>
> (Ref. 2r(U))

Carbon monoxide:

> "$LiH + 2CO = HCOOLi + C$"
>
> (Ref. 2s(U))

Chlorine: see Hydrogen chloride

Glass: see Silicon dioxide

Hydrogen chloride:

> "In the absence of moisture, the [lithium] hydride reacts with surprising reluctance with HCl and Cl_2 gases, a temperature of 500°C. being required to complete the reaction." (Ref. 2r(U))

Hydrogen sulfide:

In a reaction similar to that with carbon dioxide, hydrogen sulfide yields lithium sulfide, etc. (ref. 2q(U)).

Phenols: see Acids, anhydrous organic

Quartz: see Silicon dioxide

Silicon dioxide:

> "Silicon dioxide reacts readily at red heat giving a brown glassy material presumably containing silicon or 'silicon monoxide' . . . and possibly lithium silicate. Lithium hydride shares with lithium the ability to crack glass, Vycor, or quartz on contact at temperatures only slightly above 180°C." (Ref. 2r(U))

Sulfur dioxide:

> "$2LiH + 2SO_2 = Li_2S_2O_4 + H_2$
>
> Above 50° C the sulfide is formed." (Ref. 2r(U))

[redacted]

4. Reactions with Basic Substances

Amines, tertiary:

"Tertiary amines are not reactive at ordinary temperatures." (Ref. 2r(U))

Ammonia:

"Ammonia reacts very slowly with lithium hydride at room temperature or below, but rapidly above 300°C., yielding the amide $LiNH_2$ quantitatively . . . ." (Ref. 2r(U))

Lithium carbonate:

"Frazer [ref. 21(U)] reported the following reaction with Li_2CO_3:

$$4\ LiH + Li_2CO_3 = 3\ Li_2O + CH_4$$

The reaction occurs in a solid compact at 350°-400°C. H_2 and CO_2 are also formed to a certain extent, but may be separated from the methane by cold trapping. Since the methane produced has little or no gaseous impurity, and since all of the H is from one source, Frazer patented the use of the reaction to produce methane in which only one definite isotopic species of C and one of H is present." (Ref. 2r(U))

Lithium hydroxide:

"Lithium hydroxide reacts irreversibly with lithium hydride, forming the oxide and hydrogen:

$$LiH + LiOH = Li_2O + H_2$$

This reaction sets in on heating at about 300-350°C., which could very well be the temperature of the LiH-LiOH eutectic. It seems likely that other hydroxides act similarly, although sodium hydroxide fails to react with sodium hydride and rather forms a solution [ref. 22(U)]. There is also a possibility of metathesis followed by the reaction of lithium hydride with the lithium hydroxide formed." (Ref. 2r(U))

[redacted]

Oxides, alkali metal:

> "Alkali metal oxides are presumed to exist in equilibrium with lithium hydride in the molten state, e.g.:
>
> $$2LiH + M_2O = 2MH + Li_2O$$
>
> This should also be the case for calcium group metal oxides. The reactions have not been studied, but since LiH is more stable than other alkali metal hydrides, the above-mentioned reaction should go to completion at temperatures where MH is largely dissociated. Continuous removal of hydrogen should force this reaction to go to virtual completion, viz:
>
> $$2LiH + M_2O = Li_2O + 2M + H_2$$
>
> The lithium oxide may coexist with lithium hydride, no water being formed." (Ref. 2r(U))

5. Reactions with Miscellaneous Inorganic Substances

> Lithium hydride "most resembles calcium hydride in its reactivity with inorganic materials, air, water, etc., but is considerably more covalent in nature, which aids in interpreting its differences from calcium and other binary saline hydrides. Thus, it is a less vigorous reducing agent than calcium hydride in metallurgical reactions . . . ." (Ref. 2q(U))

The inorganic compounds with which reactions have been attempted and reported are as follows:

Carbide, lithium:

$$Li_2C_2 + 4LiH + 6H_2O = C_2H_2 + 4H_2 + 6LiOH$$ (Ref. 23(U))

Carbon:

> "Carbon was reported by Peters [ref. 24(U)] to react with lithium hydride, to form lithium carbide, but the reaction has not been well studied. Hydrogen and possibly methane are also formed. Massive graphite, which is commonly used in the electrolytic cells in which lithium is prepared, is only slightly attacked by the hydride. Carbide or graphite inclusions in metals are thought to react with molten hydride, but this has not been unequivocally established." (Ref. 2r(U))

[redacted]

[redacted]

Chlorides, metal:

> "The older literature cites a reaction with lithium chloride at red heat yielding a 'subchloride' [ref. 25(U)], which was later shown to be an equimolar mixture of LiH and LiCl [ref. 26(U)]. The alkali metal chlorides are reduced in part, at red heat, owing to removal of the volatile components, metal and hydrogen [ref. 27(U)]. Molten systems containing, for example, Na, Li, H, LiCl, and NaCl are presumably formed first. Magnesium chloride is probably first reduced to magnesium hydride, which then decomposes. Palladium chloride reacts almost explosively. Metals which form hydrides stable at red heat (Ti, Zr, Ta) may yield hydrides via these reactions.
>
> "The reductions by oxides and chlorides are analogous:
>
> $$MO + 2LiH = Li_2O + M + H_2 \quad \text{(Cr-Cu, Mo-Sb)}$$
>
> $$MCl_2 + 2LiH = 2LiCl + M + H_2"$$
>
> (Ref. 2s(U))

Diborane, gaseous:

> "The over-all reaction of gaseous diborane with an ether suspension of lithium hydride involves not only diffusion and reaction at the solid surface but also the transport of diborane from the gas phase to the liquid phase. . . . The whole process may be considered as the transport of diborane from an initial state in the bulk of the gas phase to a final state as dissolved or suspended lithium borohydride monoetherate in the bulk of the liquid phase." (Ref. 28(C))

Iron: see Magnesium

Magnesium:

> "Powdered magnesium reacts with calcium hydride on heating, presumably by forming unstable MgH_2 which decomposes as rapidly as it is formed, at dull red heat. It is possible that this reaction also occurs with lithium hydride. No evidence exists for an analogous reaction between massive iron or nickel and lithium hydride at or below 800° C." (Ref. 2s(U))

[redacted]

Mercury:

> "Mercury reacts slowly with lithium hydride forming an amalgam and evolving hydrogen. This reaction is probably general for low melting metals which do not form stable hydrides and which do form metallic compounds with lithium (e.g., Tl, Sn, Pb, Sb, Bi)."
> (Ref. 2s(U))

Metals, low melting: see Mercury

Nickel: see Magnesium

Nitrogen:

> "The complex series of reactions between lithium hydride and nitrogen gas was investigated by Dafert and Miklauz [refs. 29(U), 30(U), and 31(U)]. (This set of reactions is closely related to those between Li_3N and H_2.) Reaction began at 160° C., and became rapid at 200°-250°. Li_3NH_4 was reported to be formed here [ref. 31(U)], decomposing to Li_3NH_2 on further heating to 340°, and to lithium imide, Li_2NH, on heating to 600°-800°C. The imide may be formed directly on heating lithium hydride with nitrogen to these temperatures:
>
> $$4LiH + N_2 = 2Li_2NH + H_2 \text{ [ref. 30(U)]}$$
>
> Ruff and Goerges [ref. 32(U)] disputed Dafert and Miklauz's interpretations of these reactions, postulating no intermediates other than Li_2NH and $LiNH_2$, but Dafert and Miklauz [ref. 33(U)] defended their postulation of the 'trilithium amide'." (Ref. 2r(U))

Oxides, metal: see also Chlorides, metal

> "Pearce, Burns, and [Gantz (ref. 27(U))] studied the reaction of LiH with a series of transition metal oxides. TiO_2 was reduced to Ti_2O_3, ZrO_2 to Zr (some ZrO, Zr_2O_3 ?), and GeO_2 to GeO (some Ge), in the solid state at 600° C. ThO_2 was not reduced by LiH. V_2O_5 was reduced to VO, Nb_2O_5 to Nb_2O_3, and Ta_2O_5 to TaO_2, by LiH in fused LiCl-KCl eutectic at 350° C."
> (Ref. 2s(U))

6. Reactions with Miscellaneous Organic Substances

Acid chlorides: see also Benzyl chloride

> Lithium hydride "effects the reduction of acid chlorides (RCOCl) or thioesters (RCOSR') to the aldehydes (RCHO), on boiling in benzene, toluene, or xylene [ref. 34(U)]." (Ref. 2s(U))

Acid group: see Acids - carboxylic, sulfonic, etc.

Acids - carboxylic, sulfonic, etc.:

> Lithium hydride "reacts with carboxylic, sulfonic, etc. acids, forming salts. Lithium hydride is therefore not generally compatible with plastics containing hydroxyl, aldehyde, ketone, acid, or ester groups. Its long-term reaction with anhydride, amide, nitro, and possibly nitrile groups may be suspected. Thus, only hydrocarbons or ethers are definitely known to be inert." (Ref. 2s(U))

Alcohols, higher:

> "Lithium hydride reacts . . . slowly with higher alcohols and phenols." (Ref. 2s(U))

Alcohols, lower:

> "Lithium hydride reacts vigorously with lower alcohols . . . ." (Ref. 2s(U))

Aldehyde group: see Acids - carboxylic, sulfonic, etc.

Aldol and Claisen condensations:

> "Lithium hydride is a poor reducing agent and seems to act more like a strongly basic reagent. Thus, it is a powerful condensing agent for aldol and Claisen condensations in the same sense as the sodium alcoholates:
>
> $2\ CH_3COCH_3 + LiH = CH_3COCH_2C(OLi)(CH_3)_2$, etc. $+ H_2$
>
> $2\ CH_3COOEt + 2LiH = (CH_3COCHCOOEt)Li + LiOEt + 2H_2$"
>
> (Ref. 2s(U))

Aluminum triethylate:

> "Aluminum triethylate reacts with LiH to form $((EtO)_2AlH_2)Li$ and LiOEt, and finally $LiAlH_4$. These salt-like addition compounds are colorless, crystalline and ether soluble. They give H_2 in the presence of water . . . ." (Ref. 35(U))

Amide group: see Acids - carboxylic, sulfonic, etc.

Anhydride group: see Acids - carboxylic, sulfonic, etc.

Benzoyl chloride:

> "LiH when heated in petroleum ether with benzoyl chloride yielded benzylbenzoate . . . ." (Ref. 35(U))

Benzylalcohol:

> "Lithium phthalocyanine has been prepared . . ., using lithium hydride, benzylalcohol and phthalonitrile." (Ref. 35(U))

Benzyl chloride:

> "With benzyl chloride at 150-210° C., benzoyl benzoate is formed, with traces of benzaldehyde . . . ." (Ref. 2s(U))

Boron trihalide ether complexes:

> "The preparation of diborane from lithium hydride and boron trihalide ether complexes is described [in refs. 36(U) and 37(U)]." (Ref. 35(U))

Catalyst:

(1) "The use of lithium hydride as a catalyst to produce a filament or clear-film forming high melting polymer from e-caprolactam has been claimed." (Ref. 38(U))

(2) "Lithium hydride has been claimed as a catalyst promoter. The catalyst is a solid MoO_3 supported on an inert oxide. Lithium hydride greatly increases the yield of polymer by reducing the MoO_3 to a lower oxide. Ethylene was polymerized to a series of homopolymers

ranging from greases to tough resins. Propylene was also polymerized but to a rubber-like material. The conditions used were fairly gentle . . ..

"Many other investigations on the use of lithium hydride to reduce catalysts of this general type have been carried out using not only lithium hydride but also mixed metal hydrides such as $LiBH_4$ . . . ." (Ref. 35(U))

(3) "Polyethylene terephthalates with filament and film forming properties are made by first using LiH as the esters interchange catalyst between dimethylterephthalate and ethylene glycol with a concurrent polymerization of the product with Sb_2O_3 . . . ." (Ref. 35(U))

(4) "Any substituted 1, 3 butadiene or 1,3 butadiene itself may be reacted with ammonia with LiH as a catalyst to prepare mixtures repellent to Periplaneta Americana [an insect]." (Ref. 35(U))

(5) "It was shown that LiH catalyzed the condensation of two molecules of benzaldehyde to benzyl benzoate." (Ref. 2s(U))

(6) "Synthetic rubbers have been made by the polymerization of diolefins using lithium hydride as a catalyst . . . ." (Ref. 2t(U))

(7) A study has been made of "the use of unsupported lithium hydride as a catalyst in the formation of polyethylene." (Ref. 2t(U))

(8) "LiH will catalyze . . . the side chain ethylation of toluene . . .:

$$C_6H_5CH_3 + CH_2 = CH_2 = C_6H_5CH_2CH_2CH_3$$ [<u>sic</u>]"

(Ref. 2t(U))

Cyanogen: $LiH + C_2N_2 = LiCN + HCN$ (Ref. 2s(U))

Dimethylterephthalate: see Catalyst (3)

Ester group: see Acids - carboxylic, sulfonic, etc.

Ethers: see Acids - carboxylic, sulfonic, etc.

Ethylene glycol: see Catalyst (3)

Glycol with esters of H_3PO_4 and H_3PO_3:

"Glycol with esters of H_3PO_4 and H_3PO_3 are transesterified by LiH . . . ." (Ref. 35(U))

Halogen atoms, labile:

> Lithium hydride "may, under rigorous conditions, react with labile halogen atoms, especially iodine, to form lithium halide. In general, such reactions are feasible only in the presence of aluminum chloride or lithium aluminum hydride. Carson and Carter [ref. 39(U)] have used the smoothness of the reaction: $RI + LiH = RH + LiI$ in ether in the presence of dissolved $LiAlH_4$ to determine the heats of reaction of LiH with alkyl iodides. From these data, the heats of formation of the organic iodides were obtained. [The values for these heats of formation are not given in ref. 39.]" (Ref. 2s(U))

Hydrocarbons: see Acids - carboxylic, sulfonic, etc.

Hydroxyl group: see Acids - carboxylic, sulfonic, etc.

Iodine: see Halogen atoms, labile

Ketone group: see Acids - carboxylic, sulfonic, etc.

Methyl chloride: $LiH + CH_3Cl = CH_4 + LiCl$

Molybdenum trioxide: see Catalyst (2)

Nitrile group: see Acids - carboxylic, sulfonic, etc.

Nitro group: see Acids - carboxylic, sulfonic, etc.

Petroleum ether: see Benzoyl chloride

Phenols: see Alcohols, higher

Phthalocyanine:

> "Phthalocyanine reacts with lithium hydride, yielding lithium phthalocyanine . . . ." (Ref. 2s(U))

Phthalonitrile: see Benzylalcohol

Plastics: see Acids - carboxylic, sulfonic, etc.

Polyvinyl chloride:

> "[Cotman, ref. 40(U)] has reduced polyvinyl chloride with LiH in boiling tetrahydrofuran containing

[redacted]

some $LiAlH_4$, to yield a polyhydrocarbon similar to polyethylene. Some atmospheric oxygen is incorporated into the polymer as OH groups." (Ref. 2s(U))

Rubbers:

> "The compatibility of hydrides with rubbers, etc. has been studied to some extent [ref. 41(U)]. Presumably no reaction occurs with silicone rubbers, but this has not been established experimentally."
> (Ref. 2s(U))

Thioesters: see Acid chlorides

Toluene:

> "At 200° lithium hydride is completely inactive [in the ethylation of toluene] . . .. [Lithium hydride is] active for ethylation [of toluene] and polymerization [of ethylene] at 300° . . .. In the presence of toluene, both alkylation and polymerization take place over lithium hydride. . . . In the absence of toluene [lithium hydride is] active for polymerization."
> (Ref. 42(U))

7. Reactions Leading to Hydrides of Metals and Metalloids

Aluminum chloride:

> ". . . the well-known reaction of LiH with aluminum chloride in diethyl ether under anaerobic conditions [forms] lithium aluminum hydride, $LiAlH_4$ . . . [ref. 43(U)]:
>
> $$4\ LiH + AlCl_3 = LiAlH_4 + 3\ LiCl$$
>
> The lithium chloride, insoluble in ether, is filtered off.
>
> "The $LiAlH_4$ produced is a more versatile agent for hydride synthesis than LiH." (Ref. 2s(U))

The conditions for optimum yields in this reaction have been studied (ref. 44(U)). The use of iodine to initiate this reaction has been reported (ref. 2s(U)).

> "A change in the conditions of the reaction leads to the formation instead of polymeric, ether solvated aluminum hydride, $(AlH_3)_x$ [ref. 43(U)]:
>
> $$3\ LiH + AlCl_3 = (AlH_3)_x + 3\ LiCl"$$
>
> (Ref. 2t(U))

[redacted]

[redacted]

Aluminum ethoxide:

> "Aluminum ethoxide reacts with lithium hydride in ether to give $(Et_2O)_2 \cdot AlH_2Li$, and finally $LiAlH_4$ [ref. 45(U)]." (Ref. 2t(U))

Beryllium chloride:

Polymeric beryllium hydride $(BeH_2)_x$ has been prepared from LiH and $BeCl_2$ in ether solution (ref. 2t(U)).

Boron compounds:

> "The formation of several substituted borohydrides by addition of lithium hydride to boron compounds has been reported:
>
> $LiH + B(OR)_3 = Li^+ [HB(OR)_3]^-$ [ref. 46(U)]
>
> $LiH + B(C_2H_5)_3 = Li^+ [HB(C_2H_5)_3]^-$ [ref. 46(U)]
>
> $LiH + B(C_6H_5)_3 = Li^+ [HB(C_6H_5)_3]^-$ [ref. 47(U)]
>
> It is probable that many reactions of LiH involve this kind of addition as a first step." (Ref. 2t(U))

Boron halides:

Hurd (ref. 48(U)) reports a general reduction of gaseous boron halides to diborane above 200° C, and gives evidence for diborane formation in the explosive reaction of lithium hydride with powdered B_2O_3.

Boron trifluoride:

Diborane, B_2H_6, may be synthesized from lithium hydride and boron trifluoride in ether solution (refs. 37 and 49 to 51, all unclassified):

$$6LiH + 8BF_3{:}(C_2H_5)_2O = B_2H_6 + 6LiBF_4 + 8(C_2H_5)_2O$$

The following reaction is also reported (refs. 37(U) and 51(U)):

$$6LiH + 2BF_3 = B_2H_6 + 6LiF$$

[redacted]

Diborane:

Lithium borohydride, $LiBH_4$, may be prepared from lithium hydride and diborane in ether solution:

$$2LiH + (BH_3)_2 + 2(C_2H_5)O \rightarrow 2LiBH_4 \cdot (C_2H_5)_2O$$

where the $(C_2H_5)_2O$ is driven off at 70° to 100° C in vacuo (ref. 52(U)).

Diethyl dichlorosilane:

Diethyl silane was prepared (ref. 53(U)) from the reduction of diethyl dichlorosilane with lithium hydride, but it was found that in general lithium hydride was poorer than $LiAlH_4$ for reduction of silanes:

$$Et_2SiCl_2 + 2LiH = 2LiCl + Et_2SiH_2$$

However, the reduction of several chlorosilanes to silanes (ref. 54(U)) was accomplished by long refluxing in isoamyl ether.

Diethyl ether complex of diethylchloroaluminum:

> "With $Et_2AlCl \cdot Et_2O$, LiH in ether suspension yields Et_2AlH in ether solution [ref. 55(U)]." (Ref. 2t(U))

Halides, metal:

The syntheses of additional hydrides from reactions of lithium hydride with metal halides in ether solution that have been reported are $LiGaH_4$(refs. 43(U) and 56(U)), $(InH_3)_x$ (ref. 57(U)), $LiInBr_3H \cdot 6Et_2O$ and $LiInI_3H \cdot 6Et_2O$ (ref. 58(U)), ZnH_2 and ZnHI from ZnI_2 (ref. 59(U)), and SbH_3 from $SbCl_5$ (ref. 60(U)). A hydride of Tl could not be synthesized from $TlCl_3$ and lithium hydride (ref. 61(U)).

Magnesium bromide:

The preparation of MgH_2 from LiH and $MgBr_2$ in ether solution has been reported (ref. 2t(U)).

Trimethyl borate: see also Diborane

> Lithium borohydride "may also be prepared from lithium hydride and $B(OCH_3)_3$ [ref. 62(U)]." (Ref. 2t(U))

$$4LiH + B(OCH_3)_3 \rightarrow LiBH_4 + 3LiOCH_3$$

[redacted]

8. Explosive Reaction

"Brisance [explosion-damage] tests were performed with mixtures of lithium hydride (<325 mesh) and liquid oxygen containing 1 mole lithium hydride and 1/2 mole oxygen. This corresponds to one of the following reactions:

(a) $LiH_{(s)} + \frac{1}{2} O_{2(g)} \rightarrow LiOH_{(s)} + 94.9 \text{ Kcal.}$ . . .

or

(b) $LiH_{(s)} + \frac{1}{2} O_{2(g)} \rightarrow \frac{1}{2} Li_{2(s)}O + \frac{1}{2} H_2O$

$+ 78.3 \text{ Kcal}$ . . .

depending upon the temperature and pressure of the reaction." (Ref. 63(U))

The conclusion reached is that this reaction is considerably more powerful per pound of reactants than is that for TNT. Theoretically, it produces four times as much heat of reaction per gram as TNT (ref. 63(U)).

9. Electrolysis

"Electrolysis of the solid at dull red heat gives hydrogen at the anode." (Ref. 2a(U))

The electrolysis experience of several workers (refs. 24 and 64 to 66, all unclassified) confirms this statement. Varying percentages of the theoretical amount of hydrogen were obtained.

Reference 67(U) also extracts from the previously mentioned references.

10. Isotope Exchange

The reactions and their equilibrium constants found in reference 2(U) are summarized in table I-I, and the notes that apply to each item in the table are found below it.

Reference 68(U) describes and discusses two methods for the recovery of tritium from dilute solutions of lithium tritide in lithium.

[redacted]

[redacted]

"One is based on the crystallization of LiT at temperatures near the melting point of lithium. The other is based on sudden cooling from 700° C. to 300° C. of the vapors over the melt, with the Li metal condensing and the T_2 remaining in the gaseous phase. The yield would depend on the relative rates of cooling and of combination of Li with T_2." (Ref. 2o(U))

Reference 69(U) considers "the exchange of H and D and of H and T between the alkali metal hydrides and the hydrides of nonmetals, with the largest effects for the heavier alkali hydrides." (Ref. 2o(U))

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11. Reducing Reactions

"The reaction of lithium hydride with water and alcohols provides molecular hydrogen rather than atomic or 'nascent' hydrogen. For this reason, it is a much less powerful reducing agent than lithium when dropped into water, alcohols, etc., containing reducible solutes. This is true of all the binary saline hydrides. The reducing action is similarly less than that of lithium towards organic reactants such as esters. The insolubility, high lattice energy (218 kcal.), and stability of lithium hydride render it incapable of exhibiting the high reduction potential of the hydride ion ($E^{0}_{298} = -2.2$ volts for $1/2\ H_2 + e^- = H^-$) shown by e.g. aluminum hydride and lithium aluminum hydride in their organic reactions." (Ref. 2q(U))

See also Boron halides in section I.A.7., and section I.E. REACTIVITY.

[redacted]

B. KINETICS OF FORMATION AND DISSOCIATION

Messer wrote on rate of formation as follows:

"The qualitative variation of rate of hydrogen uptake of lithium with temperature was observed by many workers, and the pattern seems to be about the same for all. The reaction of hydrogen with bulk lithium begins at a fairly low temperature, and proceeds at a moderate rate until a certain amount is taken up. This amount varies from a few percent up to 24% of the theoretical. The rate then slows down, and it is necessary to raise the temperature to complete the reaction. [The table] below summarizes the observations of the various workers.

	Temp. of first reaction [°C]	Temp of rapid reaction [°C]
Guntz [Ref. 70(U)]	Just before red heat	Full red heat
Dafert and Miklauz [Ref. 30(U)]	440°	700°
Ephraim and Michel [Ref. 71(U)]	300°	580-660°
Soliman [Ref. 72(U)]	500°	---
Bode [Ref. 73(U)]	450-500°	700°
Albert and Mahé [Ref. 15(U)]	450°	680-690°

"The interpretation of this behavior is clear in terms of the dissociation pressure behavior to be described in [section IV.O.]. During the initial low temperature uptake, the hydrogen is dissolving in molten lithium. When saturation is reached, either throughout the bulk or on the surface, solid lithium hydride forms, slowing down further absorption. The temperature must then be raised to increase the rate, which cannot reach its maximum until the lithium hydride melts at 688° [C].

"There is some evidence that lithium metal will take up hydrogen at a lower temperature under certain circumstances. Remy-Gennetè [ref. 74(U)] reports that lithium metal, distilled out of contact with air, took up about 9% of the theoretical amount of hydrogen at [20° C] in 24 hours, and about 36% in one month. No significant uptake of hydrogen was noted for the undistilled metal. Huettig and Krajewski [ref. 75(U)] report that the finely divided form of lithium metal, prepared by dissolving it in liquid ammonia, reacts with hydrogen at room temperature.

"[Hüttig] and Krajewski [ref. 75(U)] have studied the rate of decomposition of lithium hydride from 100° to 270° C. The solid sample was introduced into a high vacuum and the rate of pressure increase followed until the pressure became constant. The first significant rate of hydrogen loss was observed at 100° C. Sublimation to cooler parts of the container caused complications above 190°. (In view of the fact that the vapor pressure of lithium metal is only [1.41×10^{-10} mm at 453.7° K (ref. 76(U))], this sublimate could not have been lithium metal.)

"The rates varied widely and in an unsystematic way, the times for achievement of the final constant pressure varying from 60 to 885 minutes, most of the values falling between 120 and 240 minutes. The results were further complicated by the fact that in most runs there was an interruption of the heating period, during which the sample was allowed to cool. The pressures involved were mostly between 0.05 and 0.22 mm. Hg.

"Quantitative data on the rate of reaction of lithium with hydrogen are relatively scarce. Albert and Mahé [ref. 15(U)] report a rate of uptake of hydrogen of 1.5 cm.^{3} H_2 per cm.^{2} of surface per second, at 1 atm. and 680° C. Perlow [ref. 14(U)] also gives some quantitative data on the rate of this reaction, and obtained an activation energy for the reaction of 12.0 ± 0.1 kcal./mol. . . .

"Swain and Heumann [ref. 16(U)] studied the rate of reaction of hydrogen with lithium at temperatures from 25° C. to 250° C. The initial rate of reaction was directly proportional to the mass of lithium and to the pressure of hydrogen. The rate then fell off, and apparently became diffusion-controlled. Irregularities in rate were shown to be due to the presence of nitrogen in the lithium.

"The variation of specific reaction rate constant with temperature was: $\log k = -1380/T + 2.09$. This gives a heat of activation of 6300 cal/mole H_2. [ref. 16(U)]" (Ref. 2j(U))

C. ANALYSIS

Several different methods of analysis of lithium hydride are presented by Messer as follows:

> "The material is commonly assayed by measuring the volume of hydrogen evolved by hydrolysis. This is best performed with a sufficiently large apparatus to permit samples weighing a gram or more to be used. The best technique is to allow the evolved hydrogen to displace water contained in a large flask kept at a constant head of pressure. The displaced water is allowed to flow into a beaker which is weighed on a platform balance. The evolution flask should contain nitrogen or argon, not only to prevent premature hydrolysis by moist air, but also to prevent an explosive reaction. The latter may be moderated by adding purified dry dioxane before adding water to the sample.
>
> "Hydrogen has also been determined in LiH by using a metal [such as mercury, lead, or tin] which readily alloys with lithium to decompose the hydride. [Bergstresser] and Waterbury [ref. 77(U)] have decomposed the hydride with lead at 600° C., the evolved hydrogen being oxidized to water by CuO, and the water absorbed and weighed. Frazer, Schoenfelder, and Tromp [ref. 78(U)] have decomposed lithium hydride with boiling mercury and measured the evolved hydrogen.
>
> "Total lithium may be determined by various methods [refs. 79(U) and 80(U)] but in view of the precision of the determination of total hydrogen by evolution, lithium determination does not appear to be too useful. The difference between total hydride by evolution and total base by titration of the hydrolysis residue permits a rough calculation to be made of Li metal and LiH contents, but this is subject to obvious errors if a correction is not made for other bases present.
>
> "If it is assumed that the only substances present are Li, LiH, and Li_2O, a useful approximation of the relative 'contents' of the three components may be made by the solution of these three simultaneous equations:

$$(\text{Wt. LiH}/7.94) + (\text{Wt. Li}/6.94) + (\text{Wt. Li}_2\text{O}/14.94) = \text{moles base}$$

$$(\text{Wt. LiH}/7.94) + (\text{Wt. Li}/13.88) = (\text{Volume H}_2\text{ S.T.P.})/22{,}400$$

$$\text{Wt. LiH} + \text{Wt. Li} + \text{Wt. Li}_2\text{O} = \text{Wt. sample}$$

"A similar set of equations could be derived assuming the basic impurity to be LiOH. A sample contaminated by air at room temperature would probably contain both LiOH and Li_2O, and a simple titration could not distinguish between these. A sample which had been heated above 300° C. and protected from air contamination thereafter would contain only Li_2O.

"Lithium hydroxide has been independently determined by Friedman [ref. 81(U)], by reacting the sample in methanol with benzoic acid, followed by a titration with Karl Fischer reagent. The Li_2CO_3 content must be known and corrected for, since this substance also reacts with benzoic acid. Frazer, Schoenfelder, and Tromp [ref. 78(U)] have determined LiOH from the measurement of the hydrogen evolved on heating by the reaction: $LiOH + LiH = Li_2O + H_2$.

"Lithium oxide could presumably be independently determined by a method used for pxide [sic] in lithium [ref. 82(U)], namely dissolving in absolute ethyl alcohol and filtering off the insoluble oxide, which is then determined by titration with standard acid. The nitride usually present must be determined and subtracted off.

"Lithium Carbonate was determined [to within ±0.01% (ref. 83(U))] by hydrolysis of the sample (presumably the aqueous residue from hydrolysis) in constant boiling HCl, the evolved CO_2 being measured as a gas. Li_2CO_3 can also be determined (assuming only Li_2CO_3, Li_2O, and LiOH in the hydrolysis residue) from the difference between the titration of the basic solution with standard acid to the bromthymol blue (pH 7) end point and the methyl orange (pH 4) end point.

"Nitrogen is determined by Kjeldahl distillation, the solid hydride being placed in the distillation flask to which ice water is added anaerobically through the condenser. The mixture is kept as cold as possible until the reaction abates. A very brief boiling completes the transfer of ammonia to the condensate. Due precaution to avoid carry-over of alkaline spray must be taken even to the extent of redistilling after addition of sodium hydroxide. The mixed indicator mannitol-boric acid method is recommended.

"Carbon as carbide is determined colorimetrically by passing hydrogen evolved by hydrolysis into ammoniacal cuprous chloride solution with gum arabic as a stabilizer. The red cuprous acetylide suspension is determined in a photoelectric photometer. Careful standardization is necessary.

"Total carbon has been determined by combustion, but this is not recommended. The entire silica combustion tube must be protected from Li_2O smoke by a liner of stainless steel foil.

"*Chloride* is best determined in the aqueous residue from hydrolysis by addition of nitric acid, filtration (use a weighed medium sinter) and precipitation with silver nitrate. This procedure is not entirely reliable, owing to the high concentration of lithium ions. Distillation of the acidified solution might be preferable but has not been thoroughly studied.

"The *acid-insolubles* are dried on the above-mentioned sintered filter and weighed. Fragments of graphite are occasionally noted.

"No simple chemical procedure has been developed for *sodium* and *potassium*. The amounts vary widely in the commercial hydride from a few parts per million to 1% or more. The spectrographic technique of Owen [ref. 84(U)] is probably the best method, although flame photometry appears promising. The spectrographic technique has the advantage that it also reveals the presence of trace impurities such as iron, nickel, chromium, magnesium, boron, [beryllium, cobalt, niobium, manganese, molybdenum, tantalum, titanium, uranium, vanadium, tungsten, and zirconium. Most of these elements were determined in the range 0.0001 to 0.1% (ref. 84(U))].

"[*Cobalt* - see section II.A.4. for a magnetic method.]

"Friedman [ref. 81(U)] has worked out techniques for the determination of various elements in lithium hydride, lithium hydroxide, or lithium metal. The above reference lists the reports in which full details are given for the following procedures:

> "*Iron* is determined photometrically, with o-phenanthroline at a controlled pH. If the amount is small, zirconium hydroxide may be needed as a carrier. [See also section II.A.4. for a magnetic method.]
>
> "*Nickel* is determined with dimethylglyoxime. [See also section II.A.4. for a magnetic method.]
>
> "*Copper* is determined with diethylthiocarbamate following removal of the nickel. [The residue from the nickel extraction may be used for the determination of copper. The carbonate procedure can be adapted to determine copper, if the copper carbonate is extracted with isoamyl alcohol (ref. 81(U)).]
>
> "*Chromium* is determined with diphenylcarbazide, following precipitation on $Al(OH)_3$ as a carrier.
>
> "*Aluminum* is determined colorimetrically [in the range of 0 to 20 p.p.m.] with 'aluminum reagent'.

> Li^+ must be present in the standard in the same concentration as in the sample.
>
> "Silicon is determined [up to 100γ] by a procedure based on the ammonium molybdate method." (Ref. 2v and w(U))

It was stated that there is no known method for determining the amount of free lithium in a sample of lithium hydride (ref. 85(U)).

It has been emphasized that standardization of methods of analysis is greatly to be desired so that standards of purity will have better meaning. David Coghlan of the Foote Mineral Co. stated that the two impurities which need improved analysis the most are lithium oxide and lithium hydroxide (ref. 85(U)).

A study of lithium hydride analysis is reported in reference 86(U). The purpose of the investigation was to determine the available hydrogen and the lithium on the same lithium hydride specimen as a pilot-type test procedure outside an analytical laboratory.

The method is based on the evolution of hydrogen from lithium hydride in the presence of water. Hydrogen is collected by displacement, and the resulting alkaline solution is titrated with standard 0.1 N hydrochloric acid as a measure of the lithium content of the sample.

Reference 87(CRD) mentions methods for the analysis of Li_2O, LiOH, $LiOH \cdot H_2O$, Li_2CO_3, and Li_3N in lithium hydride. The hydride itself was determined by measuring the hydrogen evolved with hot mercury.

D. PHOTOCHEMICAL BEHAVIOR (see also section II.C. OPTICAL PROPERTIES)

There is a very slight reversible reaction between lithium and hydrogen which is affected by visible and ultraviolet light, as well as by ionic bombardment. Exposure to these radiations causes slight decomposition of lithium hydride into its elements, and the lithium metal gives the mixture varying colors, according to the amount of the metal present. This reaction can be reversed by heating to the vicinity of the melting point. References 15, 30, and 88 to 90 (all unclassified) were the sources for this information.

> An orange-red luminescence was observed (ref. 91(U)) "when lithium hydride was irradiated with the 3650 A mercury vapor line. Three maxima were found at 5970 A, 6550 A, and 7180 A. Chemical analysis showed 0.4% excess Li, to which the luminescence was attributed. The production of the effect in crystals activated by Au or Mg is also discussed." (Ref. 2g(U))

E. REACTIVITY

> "The reactivity of lithium hydride above red heat is similar to that of lithium metal which, of course, it forms by endothermic dissociation. The free energy of formation of lithium hydride approaches zero at about 800-900° C. Thus, its reactions become similar, thermodynamically, to those of lithium. The heat of formation, however, is still considerable at this temperature; hence, the reactions are usually less vigorous than those of lithium. The heat of reaction of: $MO + 2Li = Li_2O + M$ is greater than that of: $MO + 2LiH = Li_2O + M + H_2$ by the heat of the reaction: $2LiH = 2Li + H_2$. As noted above, the latter heat is quite significant, being endothermic by 44 kcal. at room temperature and by somewhat less than this for metallurgical temperatures. Lithium hydride may therefore react (in the practical sense) only incompletely with oxides, halides, etc. which are rapidly reduced by lithium metal. The heat of formation of Li_2O is about 10 kcal. less than that of CaO, and accordingly it might be expected that lithium is a less powerful reducing agent than calcium. This is also true of the hydrides despite the higher heat of formation of calcium hydride." (Ref. 2q(U))

[redacted]

TABLE I-I. - ISOTOPE EXCHANGE REACTIONS AND THEIR EQUILIBRIUM CONSTANTS

Reaction	Temperature, ^{o}K							Comments
	200	273	298.1	400	500	600	700	
	Equilibrium constant, K							
$LiD(s) + H_2(g) = LiH(s) + HD(g)$			1.34	1.65	1.7	1.8	1.9	(a)
$LiH + \frac{1}{2} D_2 = LiD + \frac{1}{2} H_2$		2.301	2.160	1.807	1.611	1.484		(b)
$Li^7D + Li^6 = Li^6D + Li^7$		1.033	1.029	1.018	1.012	1.009		(b) and (c)
$HT + LiH = H_2 + LiT$	3.7							(d)
(Method of Urey for same reaction)	3.66							(d)
$Li^6H + Li^7D = Li^7H + Li^6D$		1.005	1.004	1.002	1.001	1.000		(b)
$Li^6 + Li^7H = Li^7 + Li^6H$		1.028	1.025	1.016	1.011	1.008		(b)
$2LiH + D_2O = 2LiD + H_2O$		8.851	7.241	4.134	2.964	2.373		(b)

[a] Reference 92(U) indicates the results of "a series of thermodynamic estimations on lithium hydride and deuteride, using Ubbelohde's [ref. 93(U)] zero point energy and Debye θ values, along with known information on lithium metal, H_2 gas, and HD gas, to estimate the free energy of formation and the dissociation pressure of the hydride and deuteride up to 900° K." (Ref. 2o(U))

[b] "Urey [ref. 94(U)] has calculated the equilibrium constants of a large number of exchange reactions involving isotopic species of lithium hydride, both for lithium isotopes and hydrogen isotopes." (Ref. 2o(U))

[c] Calculations were made (ref. 95(U)) of the equilibrium constants of exchange reactions involving isotopic species of lithium hydride by statistical mechanical methods, for gaseous lithium hydride molecules. The difference in the equilibrium constant for solid molecules should only be about 1%.

[d] The rate and the equilibrium constant were measured for the exchange between lithium hydride and tritium. "The rate of exchange was noticeable at room temperature, and complete within 24 hours at 200° C. The kinetics were difficult to interpret, the authors [ref. 95(U)] suggesting a molecular diffusion process, possibly as H_3^- . . . . (Ref. 2o(U))

[redacted]

[redacted]

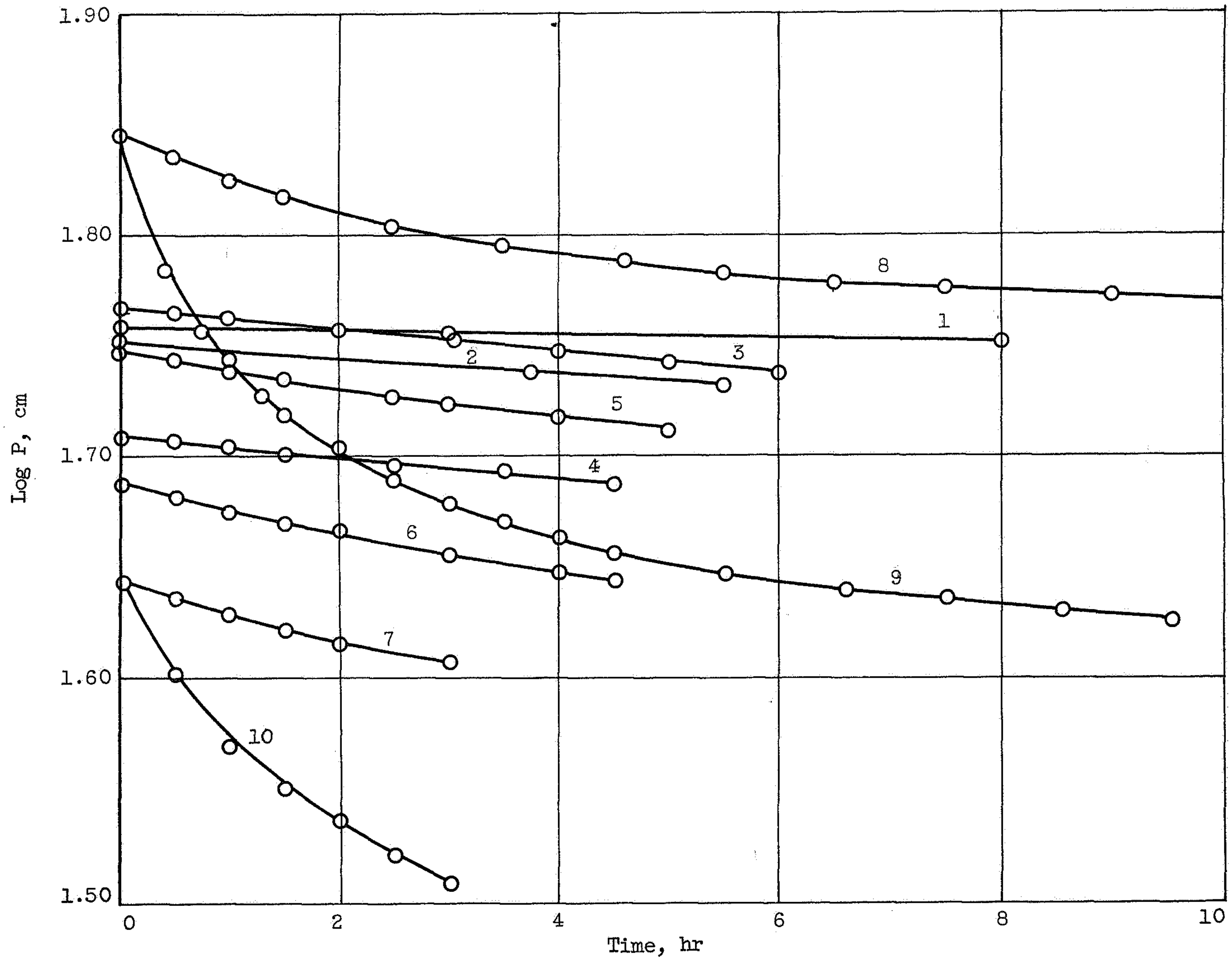


Figure I-1. - Rate of reaction of lithium with hydrogen (ref. 14(U)).

[redacted]

[redacted]

II. PHYSICAL PROPERTIES

[redacted]

[redacted]

II. PHYSICAL PROPERTIES

A. ELECTRICAL AND MAGNETIC PROPERTIES (see also section II.C. OPTICAL PROPERTIES)

1. Dielectric Constant and Dipole Moment

> The dielectric constant "has not been measured, but may be calculated from other properties. The high frequency dielectric constant is 3.60 from the refractive index at infinite wave length [ref. 96(U)]. The static dielectric constant ϵ_0 = 12.5-13.0 from observed infrared optical frequencies [refs. 97(U) and 98(U)]." (Ref. 2f(U)) It is assumed these values are for room temperature.

The dipole moment of lithium hydride at room temperature has been calculated and reported by at least seven different sources. The values are all clustered about 6.0 Debyes (refs. 2p(U) and 99(U)). Reference 100(U) presents a curve of the dipole moment as a function of internuclear distance, which is presented in figure II-1.

2. Electrical Conductivity (see also section I.A.9. Electrolysis)

> Lithium hydride "is probably less ionic than . . . sodium chloride, owing in part to the strong polarization of the negatively charged hydride ion by the smaller lithium ion. Although less ionic, it is a better conductor of electricity than sodium chloride well below melting temperature." (Ref. 2a(U))

Figure II-2 gives electrical conductivity data for lithium hydride crystals as a function of temperature (ref. 101(U)).

Figure II-3 gives the electrical resistivity of lithium hydride at room temperature as a function of pressure (ref. 102(U)).

The information that Messer found on this subject is contained in the following paragraphs:

> "G.N.Lewis [ref. 103(U)] first suggested, on the basis of his electronic theory of valence, that the hydrogen in lithium hydride should be anionic, taking on an electron to form the hydride ion, H^-, with the two electron stable configuration of the helium atom. Hence, lithium hydride should be salt-like in nature. Preliminary experiments under his direction indicated that fused lithium hydride (m.p. 688° C.,) is a good conductor of electricity.

[redacted]

[redacted]

"Moers [ref. 88(U)] measured [and calculated] the electrical conductivity of lithium hydride at temperatures from 443° C. to 754° C., using both direct and alternating current, but obtaining consistent results only with the latter. [The following table shows his results:]

Temperature, °C	Specific conductance, k, $\text{ohms}^{-1}\ \text{cm}^{-1}$
443	2.124×10^{-5}
507	2.154×10^{-4}
556	9.633×10^{-4}
597	3.665×10^{-3}
657	.01811
685	.03607
725	.09213
754	.1796

These data may be represented to ± 5% by the empirical formula:

$$k = 2.065 \times 10^{-4} + 1.8 \times 10^{-7}(T-500)^2 + 1.95 \times 10^{-11}(T-500)^4 + 0.5 \times 10^{-21}(T-500)^8. \quad [\text{where } T = {}^\circ C]$$

The temperature coefficient varies from 0.1-0.2% per °C. No discontinuity is noticeable at the melting point of 688° C." (Ref. 2f(U))

3. Paramagnetic Resonance of Color Centers (see also section II.C.2. Optical Absorption by Color Centers)

Lewis and Pretzel (ref. 104(U)) have studied the paramagnetic resonance of color centers of lithium hydride. They produced concentrations of F-centers in excess of 10^{17} per cubic centimeter, in single crystals of lithium hydride by neutron irradiation at 78° K. (See refs. 105(U) and 106a(U) for a description of F-center formation.) The electron spin resonance absorption of the F-centers was observed at an electron spectroscopic splitting factor g of 2.004±0.001. There were two rather narrow peaks of free-radical electron spin resonance absorption (approx. 10 to 15-G maximum slope width, where G is the unit of magnetic field strength). One peak occurred near the center of the F-center resonance at 2.002 G, and the other peak was about 110 G on the low-field side of the F-center.

[redacted]

[redacted]

The saturation parameter of the electron spin resonance absorption, that is, the mean value of the relaxation time product $(T_1T_2)^{1/2}$, was found to be $1\times10^{-5}\left\{\begin{matrix}+2.0\times10^{-5}\\-0.7\times10^{-5}\end{matrix}\right\}$ seconds in lithium hydride at 78° K.

The root-mean-square line widths of the F-center resonance in lithium hydride of various isotopic compositions are shown in the following table:

Isotopic composition	Li^7H	Li^7D	Li^6H	Li^6D
Rms width (G)	30.6	28.5	14.3	10.1

From these line width measurements, spin densities of the F-center electron at the Li^+ and H^- sites were estimated. Spin densities $|\psi(Li)|^2$ of 0.118×10^{24} per cubic centimeter and $|\psi(H)|^2$ of 0.025×10^{24} per cubic centimeter were chosen, where $\psi(i)$ is the value of the normalized F-center wave function at the nucleus i.

4. Magnetic Susceptibility

> "The diamagnetic susceptibility of a mole of atoms or atomic ions may be expressed by means of the equation
>
> $$[X] = -\left(\frac{N\ e^2}{6\ MC^2}\right)\sum_i \overline{r_i^2}$$
>
> where [X] is the molar susceptibility, N is [Avogadro's] Number, e the electronic charge, M is atomic mass, C is the velocity of light, and $\overline{r^2}$ is the mean square distance of the ith electron from the nucleus." (Ref. 13b(U))

Reference 107(U) reports the measurement of the diamagnetic susceptibility X at 300° K to be -4.65×10^{-6} per mole, and at 78° K to be -4.60×10^{-6} per mole, an increase of 1.1%. This increase can be partly accounted for by an increase in the susceptibility of a paramagnetic impurity of about 0.35%, because paramagnetic susceptibility usually varies almost inversely as the absolute temperature.

[redacted]

"The conclusion was reached that calculations of ionic radii using measured values of $[\chi]$ were probably not too good, due to:

1) variation in spin constant. (Van Vleck para magnetism).
2) differences in vapor phase, dissolved, and crystal-state values of susceptibilities.

"It should be pointed out that the variation in paramagnetic susceptibilities introduced by inclusion of even very small amounts of ferromagnetic materials will affect the measured $[\chi]$ value, so that it should be possible to use the evaluation of diamagnetic susceptibilities as an extremely sensitive method for detection of such materials as nickel, cobalt, and iron." (Ref. 13b(U))

The experimental value is considerably lower than theoretical predictions. Reference 108(U) gives a calculated value of $\chi = -11.8 \times 10^{-6}$ per mole, based upon the molar susceptibility as the sum of the ionic susceptibilities in a lattice with only He-type ions.

[redacted]

B. MECHANICAL PROPERTIES

1. Compressibility

The compressibility of LiH was calculated by Kasarnowsky (ref. 109(U)) to be 2.36×10^{-12}, and a value of 2.32×10^{-12} square centimeter per dyne is reported in reference 110(U).

> "This is of the same order of magnitude as the values for the alkali halides: e.g., 4.18×10^{-12} for NaCl and 1.53×10^{-12} for the more closely comparable LiF." (Ref. 2f(U))

2. Compressive Creep

The compressive creep reported in reference 111(U) of specimens cold pressed at 30,000 pounds per square inch and tested under a load of 350 pounds per square inch in an inert gas atmosphere is given in the following table:

Temperature, °C	Compressive creep in 100 hr, percent
350	<1
375	1
400	4
425	16
450	47
475	>100

> "Pressed lithium hydride pellets have been tested for compression creep in argon at 400, 500, and 600F for approximately 200 hours under a load of 825 psi. No creep was observed at 400 and 500F, but a creep rate of 3.5×10^{-6} in./in./hr was measured at 600F [312 °C]. A second specimen saturated with Santowax TEM at 500F and tested in TEM for 500 hours at 500F with a load of 350 psi showed no creep. Increasing the load on the same piece to 825 psi resulted in a creep rate of 4.5×10^{-5} in./in./hr after 500 hours." (Ref. 112(CRD))

> "In Figure [II-4], it will be seen that the rate of creep becomes quite high above 500° F under a pressure of 350 psi. Thus, the salt cannot be depended on at high temperature to lend structural support to its container." (Ref. 113a(SRD))

[redacted]

Pratt & Whitney's results to date on the compressive creep of pressed lithium hydride are listed in the following table:

COMPRESSIVE CREEP OF LITHIUM HYDRIDE (Ref. 114(CRD))

Test environment	Temperature, °F	Time, hr	Load, psi	Creep rate, in./in./hr
Argon	400	282	825	Nil
	500	107	825	Nil
	600	164	825	3.6×10^{-6}
Tertiary eutectic [polyphenyl] mixture	500	500	350	Nil
	500	500	825	4.5×10^{-5}
	600	450	825	7.8×10^{-5}

3. Compressive Strength

Some of the data on the compressive strength of lithium hydride is shown in the following table (ref. 111(U)):

Strength, psi	Comments
14,600±485	Cold pressed
19,670±5,020	Cold pressed and sintered, 3 cycles*
15,950±4,370	Cold pressed and sintered, 5 cycles*
24,300±915	Cold pressed and sintered, 10 cycles*
(95-percent confidence interval - room-temperature data)	*Blocks thermal cycled from room temperature to 593° C, specimens machined from sound, uncracked portion of blocks

The compressive strength of samples isostatically cold-pressed at 30,000 psi is reported by Waldrop to be 10,860±380 psi. Similar tests of samples sintered at 600° C gave 24,300±915 psi (ref. 113b (SRD)).

Note the coincidence of these numbers with those of reference 111(U). They may come from the same test. Pratt & Whitney gives the following values of compressive strength (ref. 115(CRD)):

10,500 psi (95 percent dense)
15,950 psi after sintering at 600° F
[Authors' note: Perhaps this 600° F was actually 600° C.]

> "Compression strengths of nonsintered lithium hydride were measured . . .. Values in the range of 8056 to 8950 psi were obtained for Pratt & Whitney Aircraft prepared material of 97.8 percent theoretical density." (Ref. 116(CRD))

E-1502

Pratt & Whitney has done further work with pressed lithium hydride and reports more compression strength data as a function of temperature as shown in table II-I.

4. Modulus of Elasticity (Young's Modulus)

Welch (ref. 111(U)) reports Young's modulus of elasticity for lithium hydride bars $1\frac{3}{4}$ by $1\frac{3}{4}$ by $16\frac{1}{2}$ inches measured at room temperature in the following table:

Modulus of elasticity, psi	Comments
7.1×10^6	Cold pressed (30,000 psi)
10.9×10^6	Cold pressed and sintered at 593° C

5. Modulus of Rigidity

MODULUS OF RIGIDITY OF LITHIUM HYDRIDE

AT ROOM TEMPERATURE (Ref. 111(U))

Modulus of rigidity, psi	Comments
$2.9\pm0.2\times10^6$	Cold pressed (30,000 psi)
$4.6\pm0.4\times10^6$	Cold pressed and sintered at 593° C

Almost the same values of 2.7×10^6 - 3.1×10^6 psi as isostatically cold pressed, and 4.3×10^6 - 5.0×10^6 psi after sintering at 600° C (1112° F) are given by Waldrop (ref. 113b(SRD)).

6. Modulus of Rupture

Welch reports a value of 1798±108 psi at room temperature with a 95-percent confidence interval. Seven cold-pressed specimens (30,000 psi) were tested (ref. 111(U)).

Waldrop gives the modulus of rupture of a 7- by 4- by $2\frac{1}{2}$-inch piece, as isostatically cold pressed at 30,000 psi, to be 1798±160 psi. A test on a similar piece, pressed and then sintered at 600° C, was tried, but it cracked before any data were obtained (ref. 113b(SRD)).

Pratt & Whitney reports the modulus of rupture at room temperature of pressed lithium hydride as shown in the following table (ref. 114(CRD)):

Modulus of rupture, psi	Comments
2570	Pressed at Pratt & Whitney Aircraft
2455	Pressed at Union Carbide Nuclear Corporation
1800	Pressed at Union Carbide Nuclear Corporation and also measured by UCNC

7. Tensile Strength and Hardness

Property	Value	Reference
Tensile strength	4000 psi	19b(CRD)
Hardness	3.5 (Moh's scale)	117(U)
	60 Kg per mm^2 DPHN	19b(CRD)
Bending strength	9000 psi	19b(CRD)

C. OPTICAL PROPERTIES (see also section I.D. PHOTOCHEMICAL BEHAVIOR)

1. Refractive Index

> "[Staritzky] and Walker [ref. 96(U)] have measured the refractive index of lithium hydride by the refraction of prisms ground from crystal fragments. Their value at 25° C. is:
>
> $$n_D = 1.9847$$
>
> This is much higher than Bode's [ref. 73(U)] earlier value of [1.615±0.002] by the immersion method." (Ref. 2f and g(U))

References 96(U) and 118(U) by the same authors give additional values of refractive index and molar refractivity. The values are shown in the following table:

REFRACTIVE INDICES n AND MOLECULAR REFRACTION R

OF LITHIUM HYDRIDE AND LITHIUM DEUTERIDE

(Ref. 96(U))

Wave-length, A	n		R, cc	
	LiH	LiD	LiH	LiD
Hg 4358	2.0694±0.0005	2.0659±0.0005	5.357	5.287
Hg 5461	2.0002±0.0005	2.0010±0.0005	5.127	5.073
Na 5892	1.9847±0.0005	1.9856±0.0005	5.074	5.021
	1.900	1.905	4.769	4.735

These values for infinite wavelengths have been extrapolated from the linear relation $1/R = a + b\nu^2$, where ν is the frequency of light vibration. The value of the constant a is 0.20968 for lithium hydride and 0.21121 for lithium deuteride; values of the constant b are $0.0004863 \times 10^{-28}$ for lithium hydride and $0.0004666 \times 10^{-28}$ for lithium deuteride.

2. Optical Absorption by Color Centers (see also section II.A.3. Paramagnetic Resonance of Color Centers)

> "The optical properties of color centers involving trapped electrons in LiH show that properties of the crystalline media other than the lattice parameter are important in relation to the energy of the optical absorption bands. The F band in LiH is found at 2.4 ev instead of at 4.8 ev, as predicted by the Ivey formula for F bands in the alkali halides. Other properties of the F band in LiH are consistent with expectations from the F-center model and with the high vibrational frequency and high cation-vacancy mobility in LiH crystals. The M band, Li-colloid band and other bands in LiH are also shifted to lower energy; therefore, the sequence of bands in LiH is comparable to the familiar sequence found in KCl.
>
> "The principal V band in LiH is at 3.5 ev. The properties of the center responsible for the V band are consistent with those of an H_2 molecule trapped at an anion site. This is the same as one proposed for the V_1 center in KCl. [See refs. 105(U) and 106(U) for descriptions of F- and V-bands.]
>
> "'Impurity' bands are found both in 'pure' and in Mg-doped LiH crystals. Bands due to Mg colloid and Z-type centers are found, along with a series of fine-structure lines like those previously reported only in LiF." (Ref. 119(U))

3. Spectra

a. Infrared

INFRARED SPECTRA OF LITHIUM HYDRIDE AND LITHIUM DEUTERIDE

Material	Wavelength range tested, μ	Wavelength of peak	Type of peak	Reference
LiH	From 2 to 50 on crystals	Min. at 1260 cm^{-1}; Max. at 550 to 1100 cm^{-1}	Reflection	97(U) and 2g(U)
LiH	12.5 to 25	17.0 μ	Absorption	120(U)
LiD	12.5 to 25	22.4 μ	Absorption	120(U)

[redacted]

[redacted]

Notes that apply to the data in the preceding table are as follows:

Reference 2g(U): The authors of reference 97(U) interpreted the data as indicating a longitudinal optical frequency of 1120 cm^{-1} for lithium hydride.

Reference 120(U): The data were for the absorption spectrum of thin films of LiH and LiD at room temperature. The peaks were broad but definite.

> "The observed ratio of the wavelengths is 1.32±0.02, in excellent accord with the ratio of the square root of the reduced masses, 1.33. This agreement is a confirmation of the elementary Born theory of lattice vibrations." (Ref. 120(U))

Klemperer observed the infrared emission spectrum of LiH up to 1400° K in the region from 950 to 1500 cm^{-1}. (Ref. 121(U))

b. Ultraviolet

Absorption maximums have been observed in solid lithium hydride at about 2517 A and 1900 to 2000 A, and one has been predicted to exist at 1630 A. For gaseous lithium hydride, an absorption band between 2880 and 3080 A was found for both the hydride and the deuteride. Further details concerning these data are as follows:

Three references reported the absorption maximums at about 2517 A (refs. 101(U), 122(U) and 123(U)), while two observed the one at 1900 to 2000 A (refs. 89(U) and 101(U)).

An absorption maximum at 1630 A corresponding to the band maximum energy of 7.61 ev was predicted (ref. 124(U)). A study was made of the ultraviolet absorption of a thin film of lithium hydride made by bombarding a thin film of lithium metal with atomic hydrogen. There was no absorption above 2500 A. From 2500 A to the lowest wavelength studied, 1860 A, the absorption rose more and more steeply.

> "When lithium metal or lithium hydride is heated in a closed tube with hydrogen above 750° C., a characteristic absorption spectrum is noted in the violet and near ultraviolet. This spectrum, appearing only in the presence of hydrogen, has been shown to be due to the presence of gaseous LiH molecules. Some emission lines may also be found.
>
> . . . .

[redacted]

"Velasco [ref. 125(U)] observed the ultraviolet spectrum of LiH and LiD [assumed to be in the gaseous form] in the region from 2000-3200 A., with a grating spectrograph of high resolving power. He discovered a new band between 2880 and 3080 A. in each case. Continuous regions of absorption were found on the low wave length side of these bands, with absorption maxima at 2720 A. for LiH and 2690 A. for LiD. This band was attributed to the second higher electronic level (B^1II). Rotational and vibrational analyses were made." (Ref. 2o(U))

An ultraviolet absorption maximum has also been reported for lithium deuteride. References 122(U) and 123(U) report a value of 2482 A.

c. Miscellaneous

"The absorption and emission spectra of gaseous LiH were observed by Nakamura [refs. 126 to 128, all unclassified], and by Crawford and Jorgensen [refs. 129 to 131, all unclassified], who also observed the spectrum of gaseous lithium deuteride. These workers observed over 1000 lines, in 26 bands, covering the range from 3100 A. to 4500 A. Most of these lines could be assigned to definite quantum transitions between energy levels in the ground state $(X^1\Sigma^+)$ and the first excited state $(A^1\Sigma^+)$ of the molecule." (Ref. 2o(U))

[redacted]

D. THERMAL PROPERTIES

1. Thermal Conductivity

Tables II-II and II-III and figures II-5(a) to II-5(c) include all the data reported. Notes concerning these data are as follows:

> "Vetrano [ref. 132(U)] has reported measurements from 50° C. to 600° C. on two kinds of samples: (a) compacted 3" × 3/4" specimens of density 0.775 g/cm^3, and (b) a specimen of cast material. The two sets of results differ considerably, especially at lower temperatures.
>
> "Fieldhouse [ref. 133(U)] has also measured the thermal conductivity of . . . material [assumed to be compacted] from 62° to 513° C, measuring radial heat flow from inside to outside of a series of stacked annular rings of 5/8" i.d. and 3" o.d.
>
> "The results of these researches are shown in Table [II-II]. The values shown are smooth-curve values at selected temperatures." (Ref. 2i(U))

Vetrano's data come extremely close to the solid-line curves in figures II-5(a) and (b); therefore, they are presented in table II-II only. Fieldhouse's data are also presented in table II-III.

The reason the conductivities of the compacts and the cast samples approach each other at the higher temperatures may be that the powdered compacts tend to sinter, and perhaps some crystal growth occurs, causing the properties to become more like those of a casting.

Welch's comments, which apply to all the curves in figure II-5(b) except the solid line, are:

> "Crystalline lithium hydride in heat transfer type test apparatus. Cylindrical specimen, 6 inch diameter by 36 inches long, heated with electric furnace. Heat balance established by measuring temperature increase in air passing through tube in core of lithium hydride." (Ref. 111(U))

Further details of the experimental procedure and apparatus used for the data reported by Welch can be found in references 111(U), 134(SRD), and 135(U).

[redacted]

[redacted]

2. Thermal Expansion Coefficient and Density

Figure II-6 presents all the thermal expansion data for lithium hydride found by the authors. The coefficient varies from 0 cm/(cm °C) at approximately -250° C to around 7×10^{-5} cm/(cm °C) at 600° C. Further information concerning the data and the methods by which they were obtained may be found at the end of this section.

A thermal expansion coefficient of 5.7×10^{-5} cm/(cm °C) for lithium deuteride, and a predicted value of 6.3×10^{-5} cm/(cm °C) for lithium tritide have been reported (ref. 136(U)).

Table II-IV gives the density and lattice constant of lithium hydride from 25° to 950° C. Further density information is as follows:

> "The solid densities at 600° and 680° were adjusted for the fact that the coefficient of expansion tends to increase more rapidly than the extrapolated estimate at temperatures approaching the melting point.
>
> "The percentage increase in volume on heating from 25° C. to the melting point is 12. This is in the range of values for the alkali halides, 12-16% [ref. 137(U)].
>
> "From considerations on volume changes on melting as a function of chemical bonding in the crystal, the density of the liquid at 688° C. is estimated at 0.58 ± 0.03 g/cm^3." (Ref. 2e(U))

The values from 700° to 950° C were taken from "smoothed" curves and were measured using a National Bureau of Standards type dip cell (ref. 111(U)).

The volume change on melting is in the range of 16 to 20 percent (refs. 2(U), 117(U), and 115(CRD)).

Part (e) of section V.C.2. discusses the variation in density of hot-pressed samples at various temperatures and pressures. Section V.C.3. has similar information for cold-pressed compacts.

Figure II-7 gives percent theoretical density as a function of compacting pressure for isostatically cold-pressed powder compacts.

Reference 2(U) also mentions several methods of determining the density of lithium hydride at room temperature, and then gives a recommended value of 0.780±0.007 gram per cubic centimeter.

[redacted]

[redacted]

"Lithium hydride has a bulk density . . . of approximately 27 pounds per cubic foot for class VI material." (Ref. 117(U)). See section V.E. for the class VI specification.)

Notes on thermal expansion coefficient data (fig. II-6):

> "Laquer [ref. 138(U)] measured the coefficient of expansion of lithium hydride over the range from 20° K to 300° K by a dilatometric technique, determining the change in length of a pressed specimen relative to its length at 273° K. [These specimens were pellets compacted from 20-mesh powder at 400° C and 20,000 psi in an Aquadag lubricated steel mold.] His results were uncertain to ±10%, due apparently to exposure of the sample to atmospheric moisture during handling." (Ref. 2e(U))

Zalkin (ref. 139(U)) determined the coefficient of expansion at high temperatures from the lattice constant, which he measured from 25° to 525° C by X-ray diffraction, based on the high-angle reflection 333 line with a beryllium window. His results may be expressed by the equation:

$$a = a(25^\circ\ C)[1 + 4.2\times10^{-5}(T-25) + 1.9\times10^{-8}(T-25)^2]$$

where a is specimen length and T is the temperature in °C.

> "This gives a linear coefficient of expansion of $4.2 \times 10^{-5}/^\circ C$. at 25° C., or a cubical coefficient of [$1.26 \times 10^{-4}/^\circ C$]. These agree closely with a previous estimate of [Nordman] and Lipscomb [ref. 136(U)] of 3.9×10^{-5} [$/^\circ C$ for the linear coefficient] based on lattice frequency considerations. They are also very close to the values for sodium chloride (cubical coeff. 1.21×10^{-4}).
>
> "Fieldhouse, Hedge, and Lang [ref. 133(U)] have measured directly the linear coefficient of expansion of a rod of lithium hydride, from room temperature to 650° C. Their results agree closely with those of Zalkin up to Zalkin's highest temperature of 525° C. At higher temperatures their expansion coefficients seem to decrease rather than increase in the normal way, suggesting possible sintering of their sample." (Ref. 2e(U))

Table II-V lists the data from reference 133(U), giving linear thermal expansion from 81° to 1160° F.

[redacted]

Welch gives a mean linear coefficient of expansion of 34.8×10^{-6} inch per inch per ^{o}C ($\pm0.5\times10^{-6}$) for the temperature range of 30° to 580° C. Three cold-pressed specimens were used, and a total of 22 determinations were taken. There was a 95-percent confidence interval (ref. 111(U)).

Tannenbaum and Ellinger determined the linear thermal expansion by X-ray diffraction photographs based on 422 and 420 lines. The sample of powdered lithium hydride was sealed inside a nickel-plated fused quartz capillary (ref. 140(U)).

Walters used a dial dilatometer. The type sample he used is unknown to the authors (ref. 140(U)).

> "The linear coefficient of expansion for lithium hydride has been determined from room temperature to 600F. Measurements were made on pieces as pressed, sintered for 16 hours at temperatures to 1000F, soaked in Wantowax MIPB or Santowax TEM. None of these treatments had discernable [sic] effect on the measured expansion. The average linear coefficient [sic] obtained from 23 measurements are:
>
> 300F 21.2×10^{-6} in./in./F
> 400F 22.4×10^{-6} in./in./F
> 500F 23.7×10^{-6} in./in./F
> 600F 25.2×10^{-6} in./in./F"

(Ref. 112(CRD))

3. Thermal Cycling and Thermal Gradient Tests

Welch relates General Electric's thermal cycling experience with lithium hydride in sealed containers as follows:

> "Tests were conducted on both cold pressed and cast lithium hydride in containers of several different alloys, on bare cold pressed blocks of lithium hydride (with and without stainless steel honeycomb), and on special test assemblies . . . to determine the effects of thermal cycling on lithium hydride and on the container material.
> "a. Canned Lithium Hydride
> "The first group of tests were [sic] made using T-shaped cans 12 inches by 6 inches by 24 inches . . . and containing machined blocks of cold pressed lithium hydride. Each unit was instrumented with two thermocouples inside the lithium hydride and one thermocouple on each of the two broad sides. The cans were equipped with a tube through which the unit was outgassed, evacuated and sealed. . . .

[redacted]

[redacted]

"Two cans were thermally cycled together by being alternately lowered into and raised out of the furnace . . ..

"Figure [II-8] shows a typical heating and cooling plot (time versus temperature) of a container during one complete thermal cycle from about room temperature to 1100 F.

"1. Effects of thermal cycling on canned lithium hydride

"The test cans were radiographed after outgassing and periodically during the thermal cycling. The radiographs . . . show the general progression of cracking of a cold pressed block during a 1100 F cycling test. In general, cracking of cold pressed lithium hydride from thermal shock starts in the first cycles and continues throughout the test. [Figure II-9 shows a typical container] sectioned after . . . thirty six thermal cycles . . . from room temperature to 1100 F.

"A carefully controlled heating and cooling rate test was made on cylinders of cold pressed lithium hydride 9 inches in diameter by 16 inches long. The cylinder heated to 1100 F at 30 F/hr and cool [sic] to room temperature at 15 f/hr [sic] did not show cracks when radiographed, although a cylinder heated to 1100 F at 130 F/hr and cooled to room temperature at 20 F/hr showed small cracks in the transverse and in the longitudinal directions.

"The normal thermal cycle (room temperature to 1100 F and return in about 24 hours) given the T-shaped containers was about 140 F/min (heating) and 55 F/min (cooling). This is a more rapid thermal cycle than those given the cylinders, and cracking occurs as would be expected.

"Castings were made into T-shaped containers using the reservoir method [described in section V.C.5.c.]. The castings were made both with and without stainless steel honeycomb. Outgassing was not required with cast lithium hydride since the LiOH-LiH reaction occurs during the melting. . . . Cracking [in a casting after 13 thermal cycles to 1100° F] was not found to be as severe [as in the cold-pressed samples], although separation of the casting from the container was evident. Addition of a stainless steel honeycomb to the casting [fig. II-10] eliminates major cracks and provides an effective means of providing a more macroscopically uniform material.

"2. Effects of thermal cycling on container materials in contact with lithium hydride

"T-shaped cans were prepared of various alloys and filled with cold-pressed or cast lithium hydride. . . .

"In general, thin-walled (0.037 inch thick) Inconel containers are not satisfactory and fail in about 200 hours. Increasing the wall thickness to 0.078-0.090 inch

[redacted]

[redacted]

increases the life of the container to 1000 or more hours at 1100 F.

"Types 304 and 316 stainless steel and Hastelloy C containers (0.031 - 0.037 inch thick wall) did not show appreciable resistance to lithium hydride when thermally cycled to 1100 F, although Type 347 showed considerable resistance as evidenced by four tests ranging from 360 to 1460 hours duration. Failure in all cases was along welds.

"Type 301 (1/4 hard) stainless steel initially thermal cycled from room temperature to 700 F for about 285 hours with no evidence of failure, failed after an additional 200 hours of thermal cycling to 1000 F." (Ref. 141(CRD))

Welch says about uncontained lithium hydride:

"Two groups of tests were conducted on uncanned lithium hydride: (1) Blocks with metallic additions, and (2) Thin slabs containing perforated stainless steel honeycomb. In both cases the lithium hydride was thermally cycled by placing the steel reactor containing the specimens in a furnace at temperature, heating the reactor and specimens to temperature, removing the reactor from the furnace after about eight hours, and allowing the reactor and specimens to cool. The reactor contained a hydrogen atmosphere to prevent deterioration of the lithium hydride during the cycling. The reactor was opened periodically to visually examine the lithium hydride.

"1) Effects of thermal cycling on blocks of lithium hydride

The first group of specimens consisted of four blocks (4 by 6 by 8 inches) as follows:

a. Stainless steel honeycomb, 0.001 inch foil, one inch square cell, 11/16 inch holes on 3/4 inch centers [fig. II-11].

b. Same, except 1/2 inch holes on 3/4 inch centers [fig. II-11].

c. Same, except 0.002 inch thick foil with no perforations.

d. Chopped steel fibers, (about one inch long)

"All blocks were given ten thermal cycles from room temperature to 700 F, followed by ten cycles to 1000 F.

. . . .

"Thermal cycling to 700 F of non-perforated honeycomb compacts caused serious breakout of external sections while perforated honeycomb compacts were virtually unaffected. The addition of chopped steel fibers offered no advantage as the block cracked seriously.

[redacted]

"Continued cycling to 1000 F showed the non-perforated honeycomb compact to loose additional external sections and the lithium hydride to shrink leaving exposed metal foil. The perforated honeycomb compacts were again almost unaffected except for a slight breakout along one edge of the block, although one block (honeycomb with the 11/16 inch perforations) showed the start of a fracture tra[ns]verse to the cell direction. Cracking of the block containing steel fibers continued until four almost equal-sized pieces were formed.

"A second group of four blocks was given ten thermal cycles to 800 F and eleven cycles to 1100 F. The blocks containing the non-perforated stainless steel and the chopped steel fibers suffered the same damage as described before. All blocks underwent more shrinkage from the 'sintering effect' at the higher temperatures. The major difference encountered in the test was the complete fracturing of the block containing the honeycomb with the 11/16 inch perforations . . . probably because the honeycomb with the larger holes (less metal foil between holes) was weaker and more susceptible to trans-cell rupture.

"The compact containing the honeycomb with 1/2 inch perforations on 3/4 inch centers was selected as the most resistant to cracking under thermal cycling . . ..

. . . .

"(2) Effects of thermal cycling on thin slabs of hydride containing stainless steel honeycomb (1/2 inch perforations).

"Slabs (3/4 inch thick) of lithium hydride, cold pressed in perforated stainless steel honeycomb (1/2 inch holes), were prepared and thermally cycled to establish the relative merit of cell direction in reducing cracking of thin sections.

"[One slab was tested] with the cell depth in the same direction as the short dimension and [another was tested] with the cell depth perpendicular to the short dimension.

"The slabs were thermally cycled in the same manner as were the rectangular blocks [10 thermal cycles to 700° F and then 10 additional cycles to 1000° F]. . . . Neither slab was seriously affected by thermal cycling from room temperature to 700 F, although additional cycling to 1000 F produced separation of the lithium hydride compact along the metal foil . . ., and the slab with the cell depth perpendicular to the short dimension . . . suffered loss of lithium hydride slivers from the surface and cracking across the cell depth indicating tearing of the steel foil.

[redacted]

[redacted]

> "It was concluded that in the case where the slab thickness is about the same as the cell dimension (1 inch square cell) the honeycomb should be positioned with the cell depth in the same direction as the short dimension to provide a more thermally stable configuration." (Ref. 141b(CRD))

Further work by Welch has been reported as follows:

> "The method of carrying out dynamic air tests at temperature of lithium hydride contained in cylinders with defective (cracked) welds was modified to better simulate the temperature conditions that are present under operating conditions. This was achieved by supplying heat at the center of the lithium hydride and allowing the temperature to vary from 950°F in the center to about 750°F at the periphery. Ten 10-hour soaking cycles plus five rapid cycles from room temperature to 950°F in the center of a specimen were completed. No exudate was observed along the weld crack." (Ref. 142(SRD))

> "The lithium hydride casting made . . . by the Union Carbide Nuclear Company was adapted to a thermal-stability test assembly. The lithium hydride was cast in a hexagonal container that is 17 inches long by 12 inches across flats and has 19 cooling tubes. Heating elements were installed in the interior circle of six tubes, and air lines were connected to the outer circle of 12 tubes and to the center tube. In the 1000 hours the test assembly has operated, the power input, air velocity, mass airflow, and temperatures were monitored. Temperatures recorded were for heater tubes, entrance and exit air, and skin. The testing consisted of temperature-cycling the unit through a series of 8-hour slow cycles followed by a series of 1-hour rapid cycles. No significant change in the operating characteristics of the assembly has been noted." (Ref. 143(SRD))

Another of General Electric's thermal experiments is related as follows:

> "The importance of maintaining small thermal gradients in a system containing lithium hydride near its melting point can be shown by the following experiments . . .:
>
> "A cylindrical casting of lithium hydride was heated at the bottom to a temperature above its melting point, while the top was maintained below the melting point. As a result of the large changes in volume which occur during the solid-liquid transformation, the bottom of the stainless

[redacted]

> steel container surrounding the molten zone was bulged almost to the rupture point. Thermal expansion studies on cast lithium hydride have shown a softening of the hydride at approximately 900°F. For the case where the lower end of the casting was heated to the melting point, and the thermal gradient along the length of the casting maintained at approximately 10 F per inch, gradual melting of the lithium hydride occurred without distortion of the container.
>
> "Thermal cycling experiments have shown that cast lithium hydride sealed in an Armco container may be continuously heated in the range of 200-1200° F without noticeable breakdown of the material." (Ref. 19a(CRD))

Pratt & Whitney has performed thermal shock testing which is reported as follows:

> "In further tests of the degassing and thermal shock behavior of lithium hydride, two 8 inch cubes pressed at Union Carbide Nuclear Company and canned in type 316 stainless steel were heated to 500F at an average rate of 24F per hour. One gallon of monoisopropylbiphenyl ([Wantowax] MIPB) at 300F was introduced rapidly to fill the void space in the cans. One cube was then cooled to room temperature at an average rate of 17F per hour. The second cube was reheated to 500F and held for 200 hours before cooling to room temperature. Post-test examination showed only hairline cracks across four faces of the former cube and insignificant surface cracks on the latter cube. There were no significant dimensional changes and the MIPB penetration of the surface did not exceed 1/8 inch." (Ref. 114(CRD))

4. Method for Calculating Heat Transfer Involving Melting or Freezing

Murray and Landis give a good heat-transfer method background, and additional references for those interested in any material for heat storage where the latent heat of fusion is to be utilized (ref. 144(U)).

E. MISCELLANEOUS PHYSICAL PROPERTIES

1. Crystal Structure and Bonding in Crystalline Lithium Hydride

Lithium hydride has a face-centered cubic structure with a measured room-temperature lattice constant a_o of 4.0834±0.0005 A (ref. 2d(U)). (See table II-IV for lattice constants at different temperatures.) Reference 145(U) gives a calculated value of 4.42 A, assumed to be at room temperature.

Although some say the structure is of the sodium chloride type, the position of the hydrogen atoms has not been determined (ref. 2d(U)).

Concerning bonding in crystalline lithium hydride, Messer draws the following general conclusions from the work of 25 authors:

> "The high melting point, hardness, electrolytic conductance, and electrolysis behavior of lithium hydride clearly show its ionic nature. However, the results of certain theoretical calculations show a considerable amount of covalency in the Li-H bond in this solid." (Ref. 2m(U))

Messer indicates that the bond is 41% ionic in character (ref. 2q(U)). However, reference 146(U) reports that solid lithium hydride has 25% ionic character, with limiting values of 50% or complete covalency.

Reference 147(U) gives a cation radius of 0.68 A and an H^- radius of 1.40 A, with an internuclear distance, both calculated and observed, of 2.04 A.

> "Behringer [ref. 148(U)] calculated the dependence on pressure of the energy gap between the last filled band and the first empty [energy] band of LiH, and concluded that metallic lithium hydride would be produced at 35 megabars. He found that charge transfer from Li to H would affect this pressure by less than 1 megabar. However, a transition to the denser CsCl structure might lower the energy gap and produce the transition at a lower pressure." (Ref. 2n(U))

A great many quantum mechanical studies of both crystalline and gaseous lithium hydride have been reported. Some of the information is listed in the preceding paragraphs of this section, and in section IV.L. ELECTRONIC ENERGY. Much of this information does not agree with experimental results, and many of the references do not agree with each other. The references are: 82, 100, 122, 145, and 149 to 173, all unclassified.

[redacted]

2. Solubility

Reference 174(U) reports that lithium hydride is insoluble in hydrocarbons, ethers, and tertiary amines. However, reference 2 reports a trace solubility in polar organic solvents, such as ether, due to a slightly covalent nature. It also states that there is no appreciable solubility in inert solvents.

> "Lithium hydride dissolves in molten halides such as the LiCl-KCl eutectic and in certain molten saline carbides. . . . Molten lithium hydride is also a solvent for some carbides and halides. There appears to be no inert liquid solvent, although vestigial solubility in ethers has been suggested as a mechanism for the reactivity of lithium hydride in certain reactions [ref. 175(U)]." (Ref. 2b(U))

3. Alloying Potential

> "Of all metal hydrides, lithium hydride is unique in that the molten hydride is stable at its melting point . . .. Alloying of this material, N_H approximately 6.2, with either zirconium or yttrium hydride may produce some interesting results in regard to both stability and hydrogen content. To determine the alloying potential of lithium hydride with both hydrided zirconium and yttrium, specimens of the latter, 1" diameter by 1/2" high, were prepared with a 3/8" diameter cavity. This cavity was filled with lithium hydride and both specimens were heated to 1300° F in hydrogen. Both specimens exhibited a very slight weight increase indicating that decomposition of the lithium hydride did not occur. Examination of the cross section of these specimens revealed no indication of alloying with the yttrium hydride but some attack, penetration to a depth of approximately 1/8", had occurred with the hydrided zirconium. Additional specimens similar to the above were prepared and were heated in hydrogen at 1500° F for three hours-the vapor pressure of lithium hydride reaches 760 mm between 1540° and 1700° F. Appearance of these specimens after this treatment indicated that the lithium hydride had melted and climbed over the edges of both specimens. Once again examination of the specimens revealed that alloying of the lithium hydride and hydrided zirconium had occurred but no alloying was accomplished with yttrium hydride. Spectrographic analysis of a midway sample confirmed this showing 0.1% lithium in the zirconium with none in the yttrium." (Ref. 176(CRD))

[redacted]

[redacted]

4. Melting Point (see also section IV.M. LITHIUM - LITHIUM HYDRIDE SOLID-LIQUID EQUILIBRIUM)

Messer gives 688°±1° C, or 1270° F, as the melting point of pure lithium hydride. This number is probably quite reliable, as Messer has done a great deal of work with lithium hydride in the region of the melting point (refs. 2i(U) and 177(U)).

5. Viscosity and Surface Tension

Welch's data show that the viscosity of molten lithium hydride between 1252° and 1578° F varies little, and appears to be described by a straight line between 0.38 centipoise at 1252° F and 0.24 centipoise at 1578° F. This sample melted at a temperature less than that given in section II.E.4., perhaps indicating supercooling or a sample containing less than 100% LiH. The measurements were made with a Brookfield viscometer under a pressure of 20 pounds per square inch gage (ref. 178(U)).

The experiences related in reference 85(U) indicate that molten lithium hydride is very apt to creep up the sides of its container, to a degree dependent on the container material and cleanliness, and on the purity of the lithium hydride. The purer the lithium hydride is, the less it tends to creep. For zero-gravity, heat-transfer applications this creeping tendency appears to be a very desirable characteristic. (See also sections V.C.5.c. and V.D.8).

[redacted]

TABLE II-I. - ULTIMATE COMPRESSION STRENGTH

(Refs. 114(CRD) and 179(SRD))

Temperature, °F	Pratt & Whitney Aircraft pressed, psi	Union Carbide Nuclear Corp. pressed, psi
75	14,900 15,800	14,100 14,000
200	12,700 13,900	11,800 13,000
400	9,500	
600	5,470 5,070	4,690 4,975
700	4,465 3,950	3,815 3,880
800	2,465 2,335	3,615 2,955

TABLE II-II. - THERMAL CONDUCTIVITY OF LITHIUM HYDRIDE (Ref. 2i(U))

(w)(cm)/(cm^2)($^\circ$C)

Temperature, $^\circ$C	Vetrano		Fieldhouse
	Crystals	Compact	Unknown (assumed to be compact)
50	0.125	0.0695	0.0750
100	.106	.0652	.0650
200	.074	.0575	.0576
300	.055	.0515	.0510
400	.042	.0469	.0453
500	.036	.0432	.0414
600	------	.0409	------

TABLE II-III. - THERMAL CONDUCTIVITY OF LITHIUM HYDRIDE (Ref. 133(U))

Mean temperature, $^\circ$F	Thermal conductivity, Btu/(hr)(ft)($^\circ$F)
143	4.22
214	3.75
342	3.42
456	3.21
510	3.10
579	2.93
666	2.73
766	2.60
840	2.52
955	2.36

TABLE II-IV. - DENSITY AND LATTICE CONSTANT OF LITHIUM HYDRIDE

Temperature, °C	Density, g/cc	Lattice constant, a_o, A	Comments
25	0.775	4.083	Calculated (ref. 2e(U))
200	.757	4.116	↓
400	.733	4.159	↓
525	.718	4.188	↓
600	.705	4.210	Estimated (ref. 2e(U))
688(s)	.690	4.240	↓
688(l)	.58±0.03	-----	↓
700	.550		Measured (ref. 111(U))
750	.546		↓
800	.540		↓
850	.532		↓
900	.518		↓
950	.492		↓

TABLE II-V. - EXPERIMENTAL DATA FOR LINEAR THERMAL EXPANSION OF LITHIUM HYDRIDE (Ref. 133(U))

Temperature, °F	$\frac{\Delta L}{L} \times 10^3$
81	0
116	.76
189	1.87
272	3.55
326	5.16
368	5.94
416	7.55
501	10.05
593	12.28
671	15.52
742	18.07
799	19.99
856	22.00
911	24.08
957	25.38
1025	27.26
1090	28.81
1160	30.26

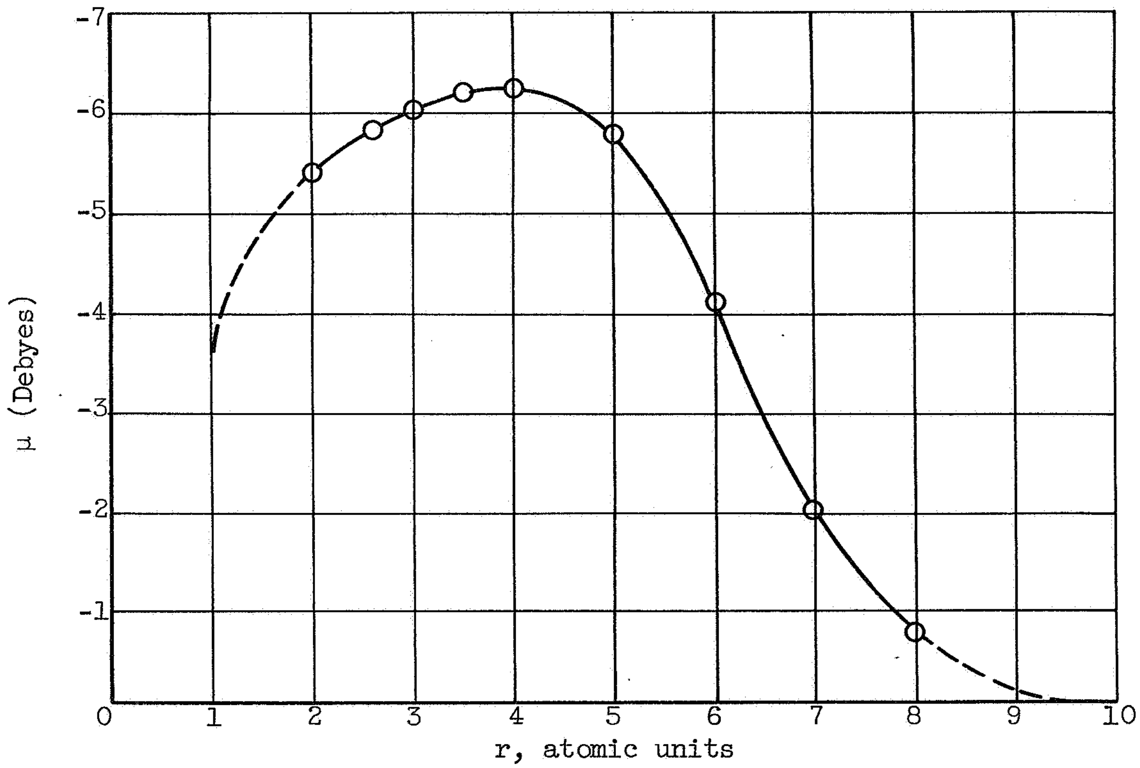


Figure II-1. - Dipole moment for LiH as function of internuclear distance (ref. 100(U)).

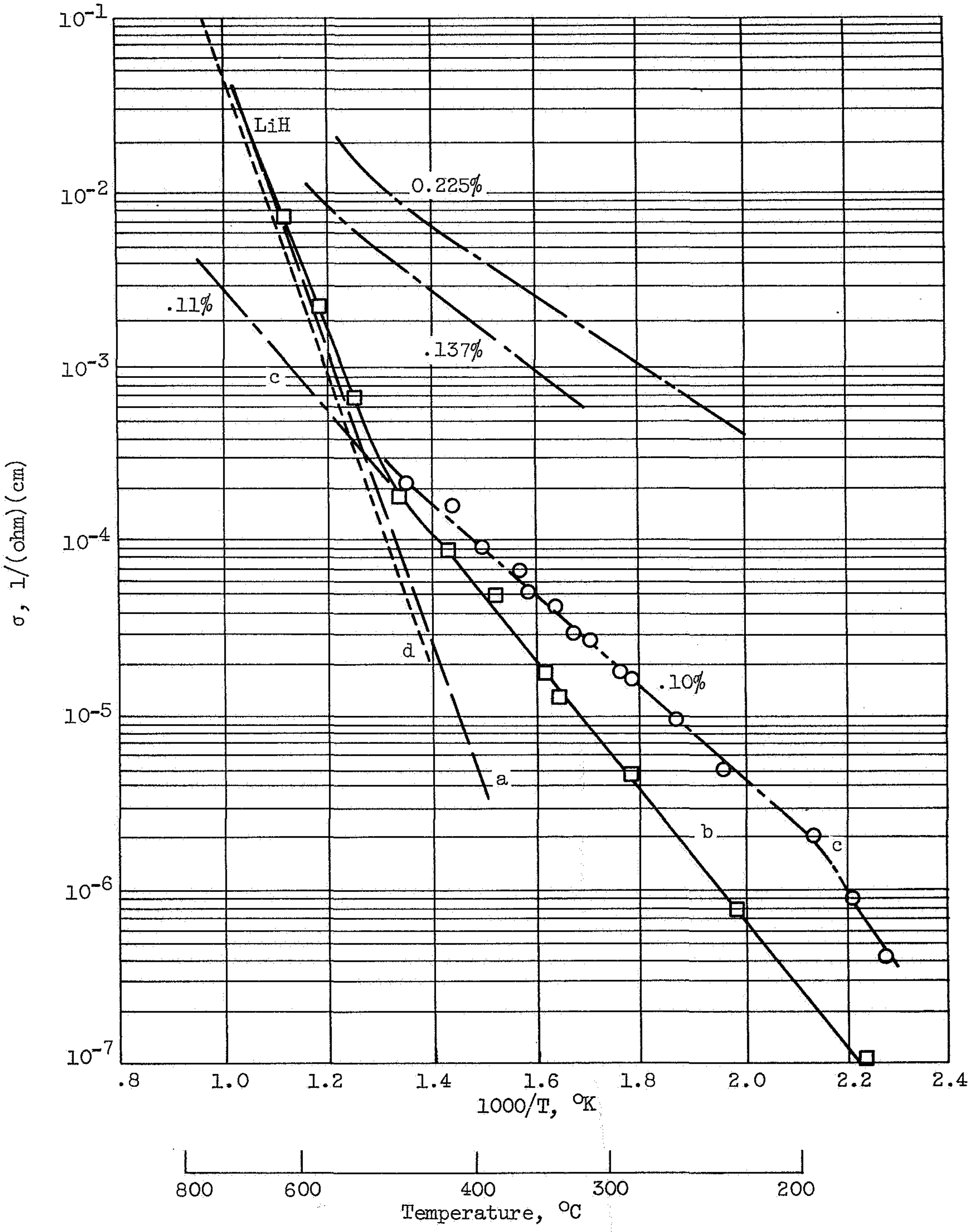


Figure II-2. - Electrical conductivity of "pure" and Mg-doped LiH as function of temperature (ref. 101(U)). (a) Intrinsic conductivity of an ideal crystal; (b) measured conductivity of pure crystal; (c) conductivity of a Mg-doped crystal with the indicated impurity content; (d) measured conductivity of a crystal containing oxide. (LiH after K. Moers.)

[redacted]

[redacted]

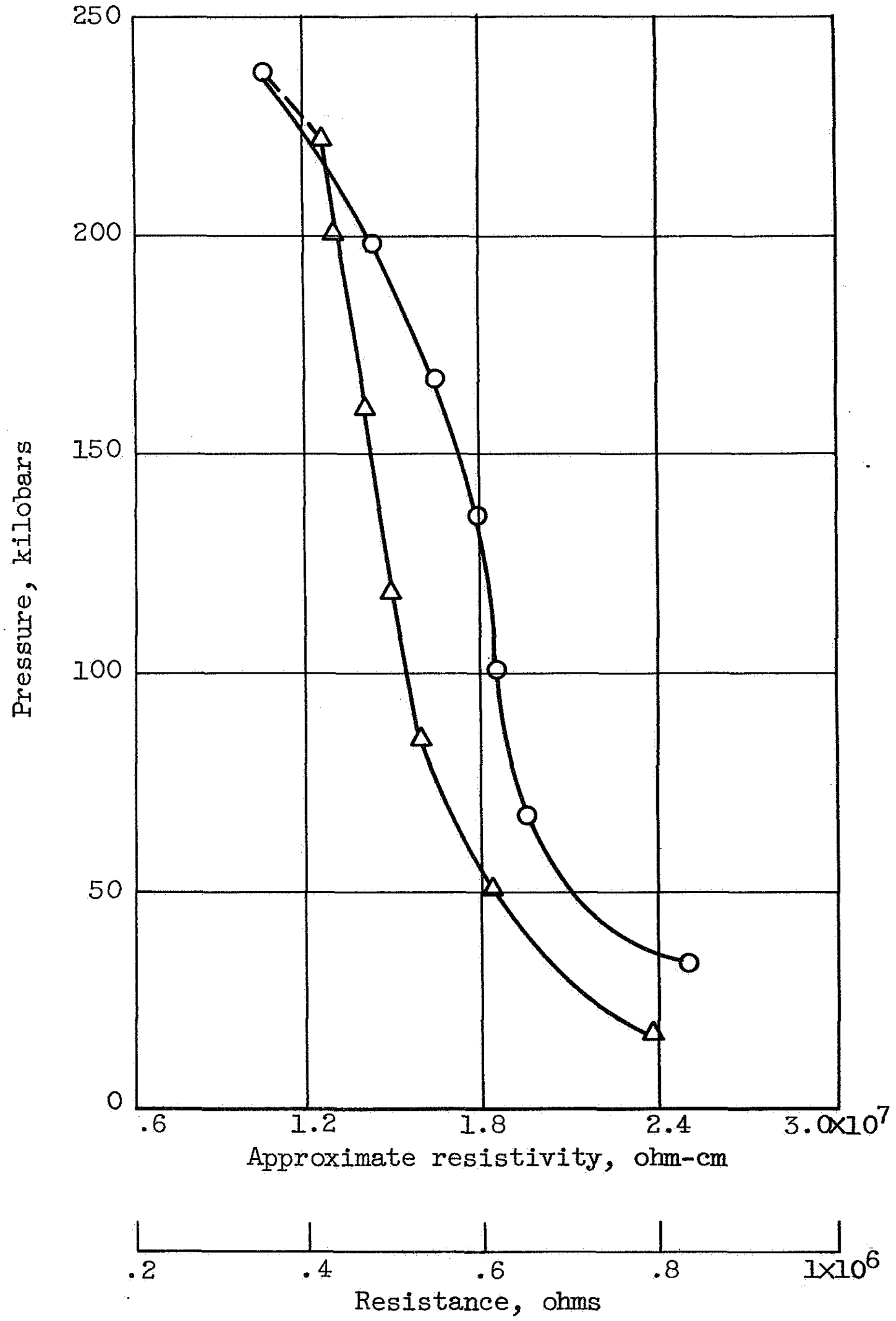


Figure II-3. - Approximate resistivity of LiH at room temperature (ref. 102(U)).

[redacted]

[redacted]

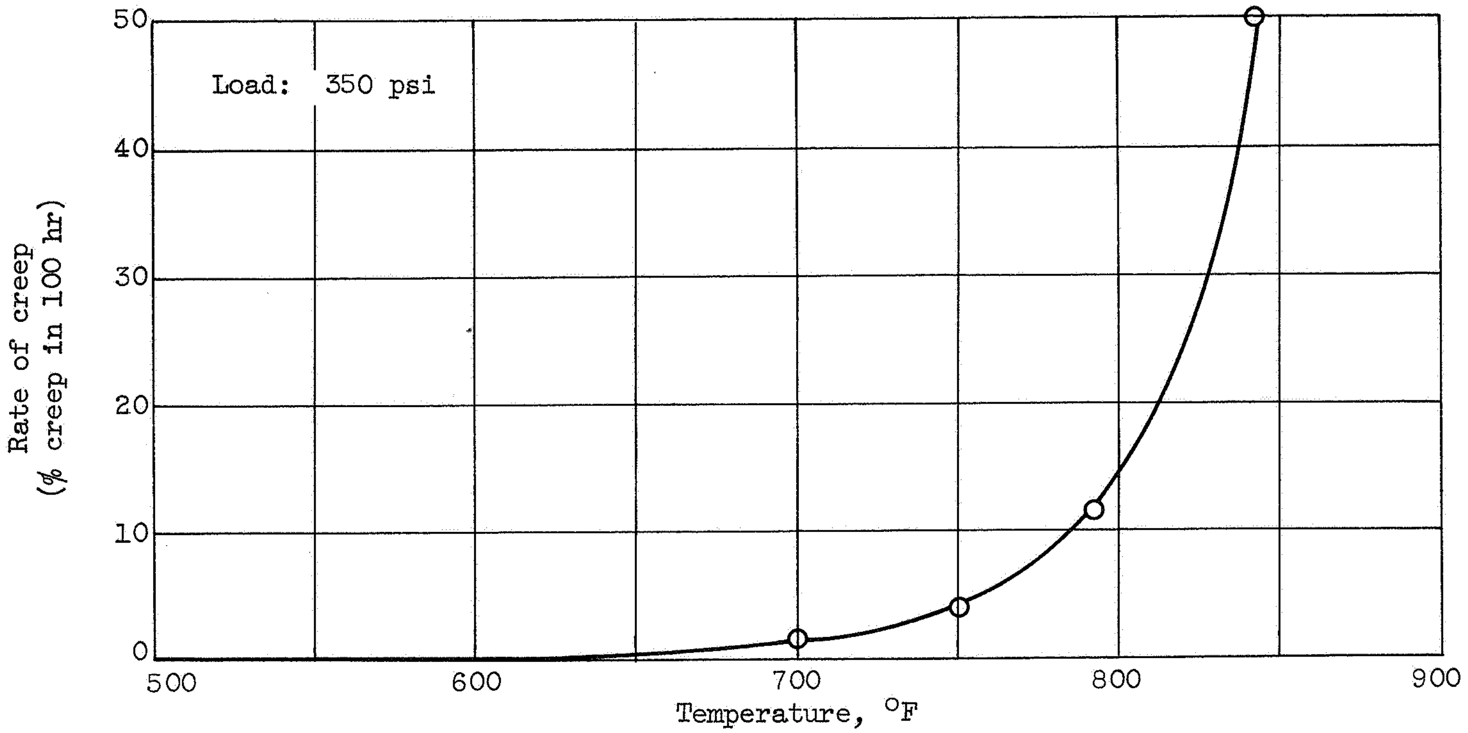
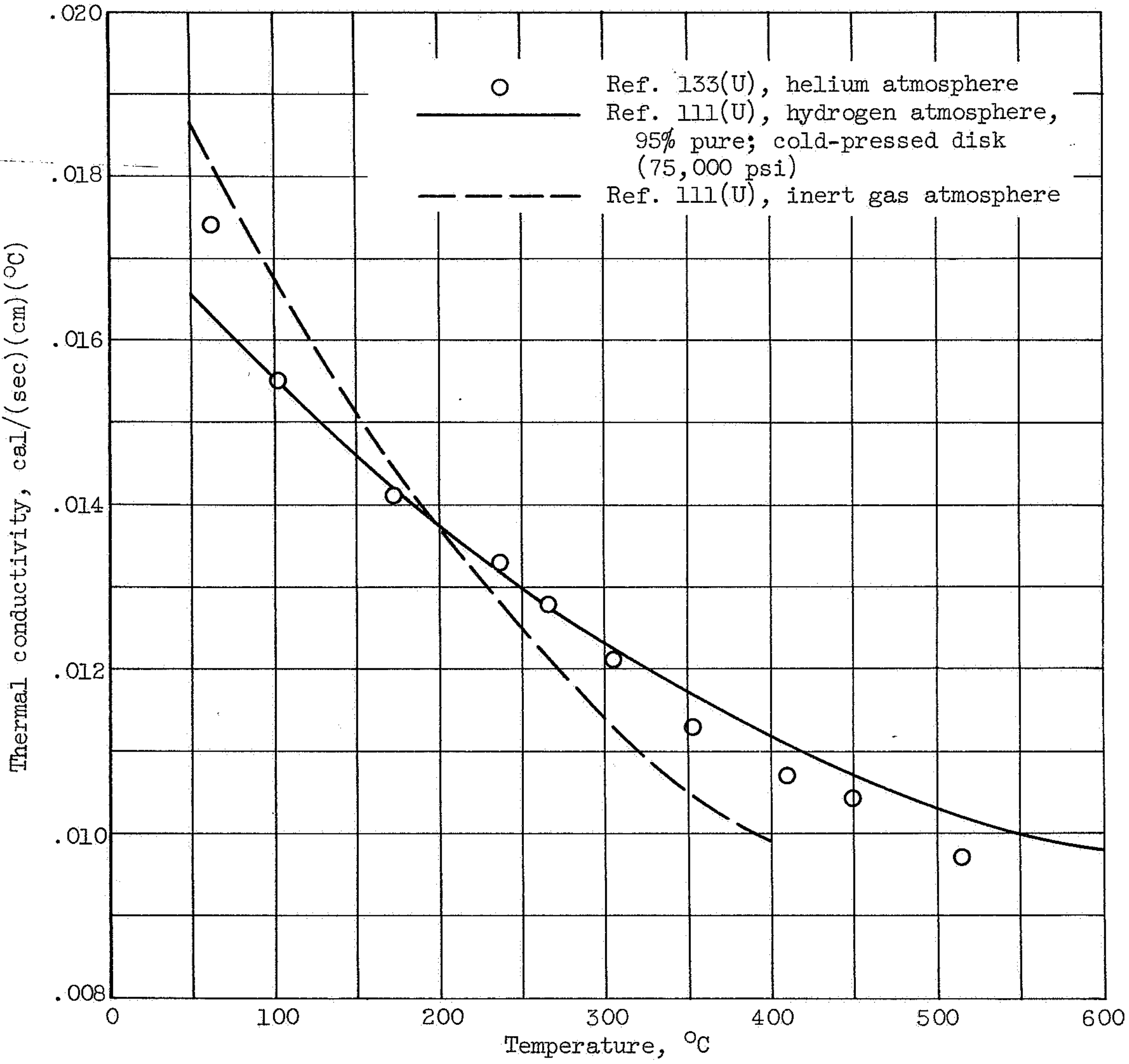


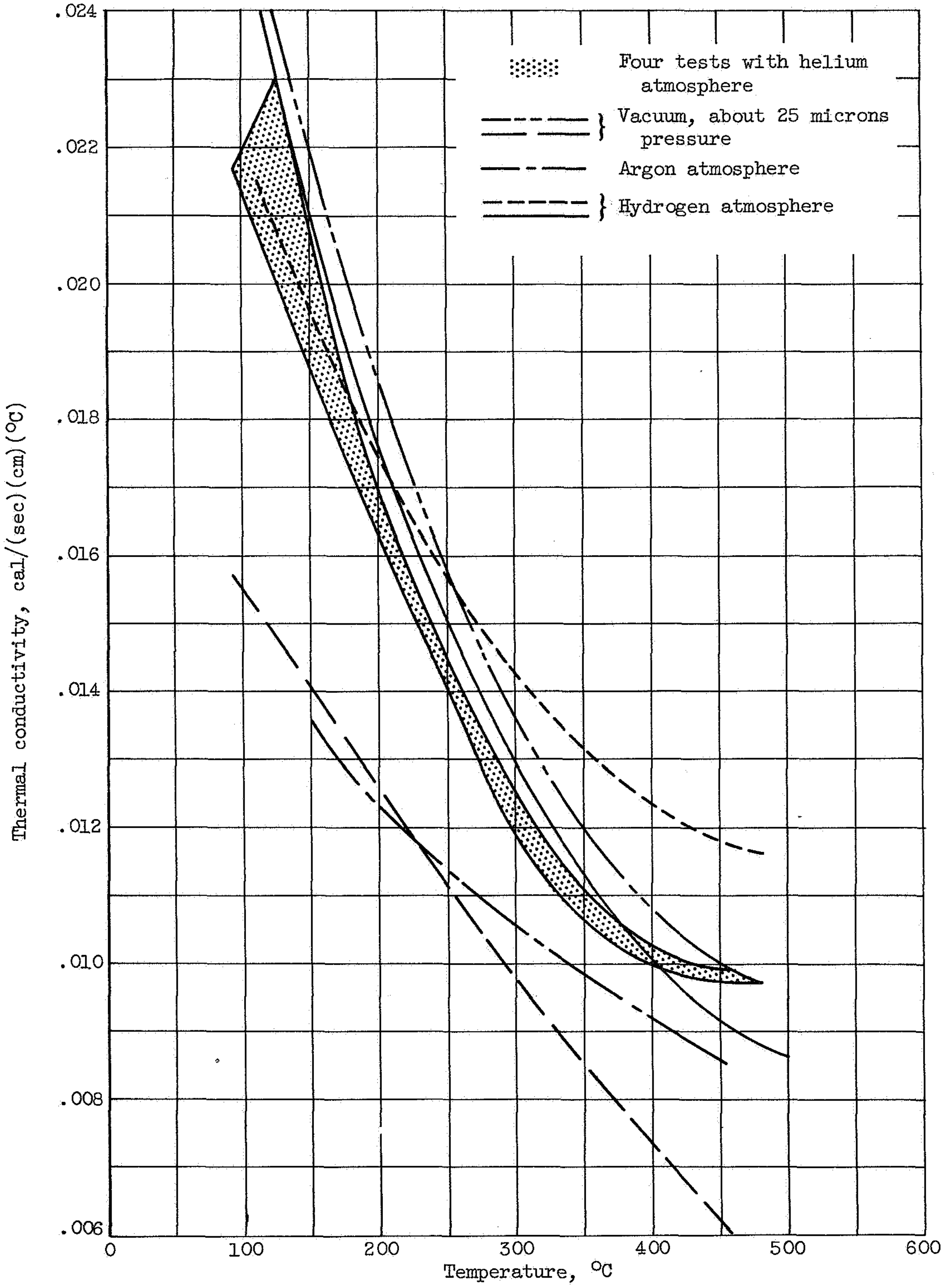
Figure II-4. - Rate of creep of lithium hydride powder compacts prepared by isostatic pressing at 30,000 psi (ref. 113c(SRD)).

[redacted]



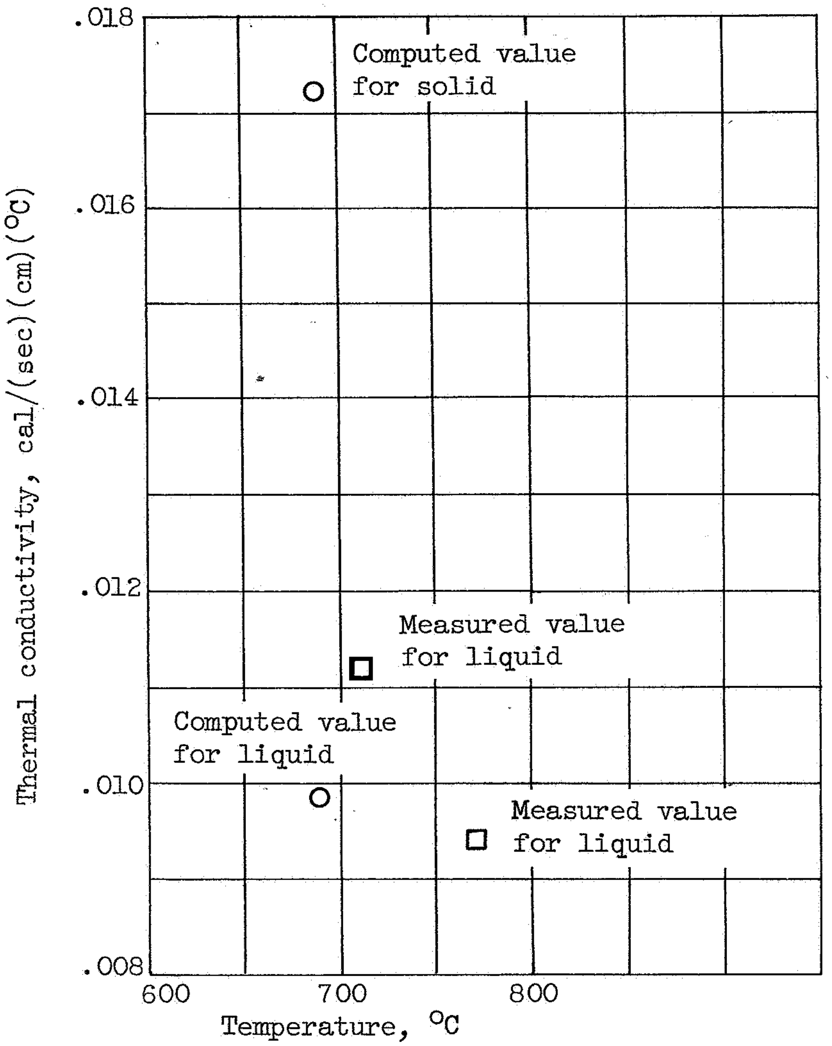
(a) Compacts.

Figure II-5. - Thermal conductivity of lithium hydride.



(b) Castings. (Curves drawn through smoothed-curve data from ref. 111(U). See also ref. 135(U)).

Figure II-5. - Continued. Thermal conductivity of lithium hydride.



(c) At and above its melting point (ref. 180(U)). Reference 180(U) indicates that the measured values are probably in error on the high side because of errors introduced by the apparatus used.

Figure II-5. - Concluded. Thermal conductivity of lithium hydride.

[redacted]

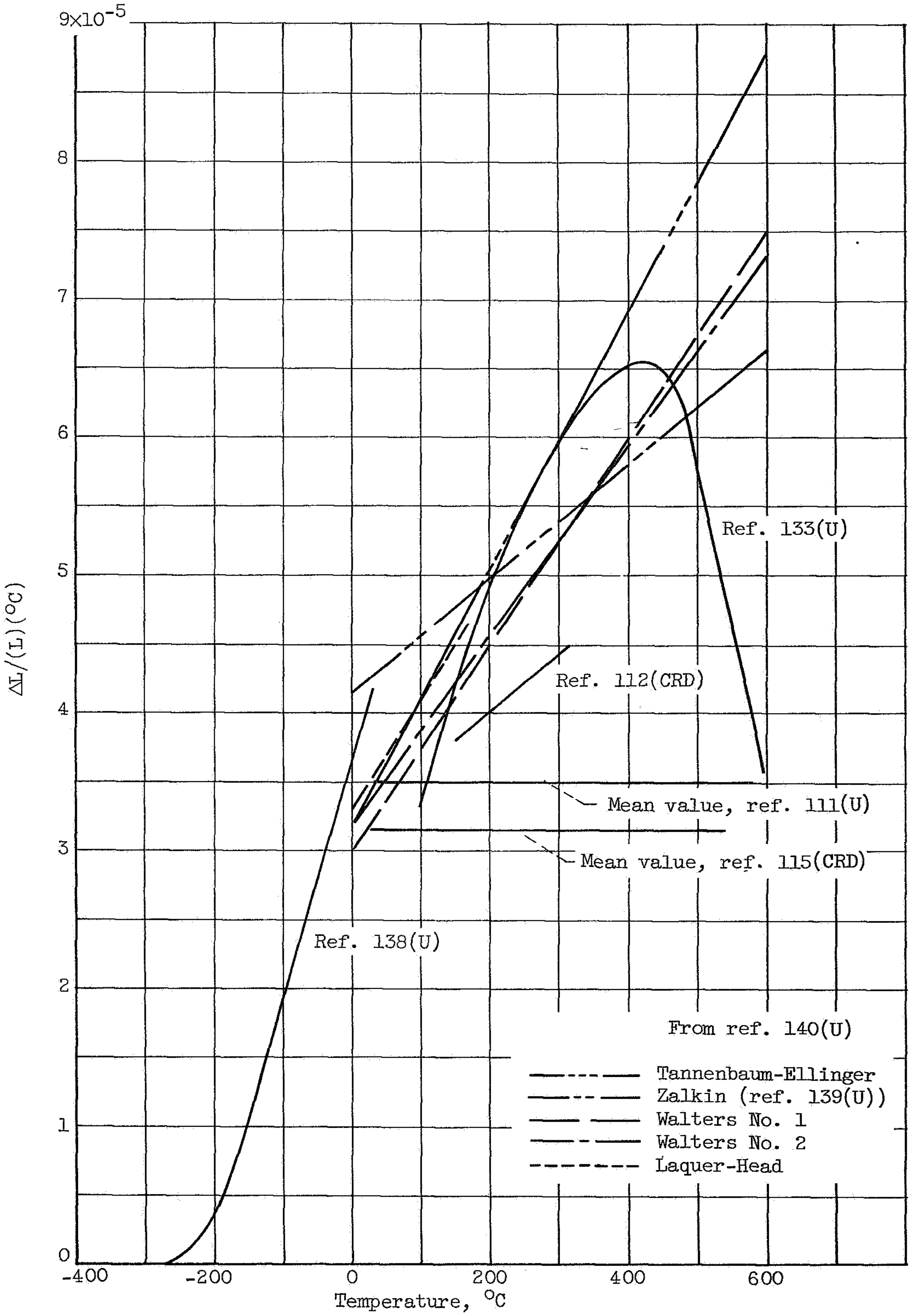


Figure II-6. - Linear thermal expansion coefficient of lithium hydride.

[redacted]

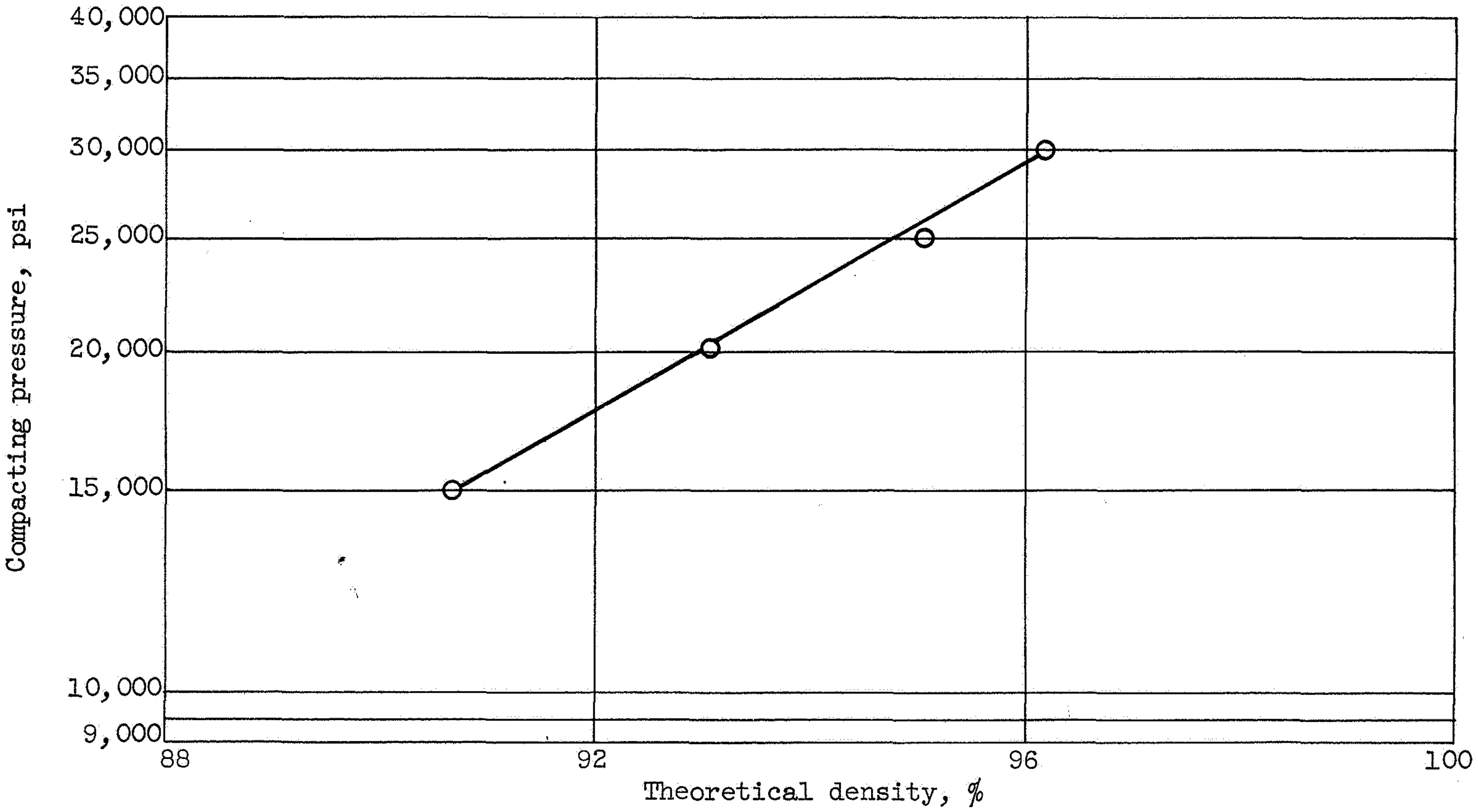


Figure II-7. - Density of lithium hydride powder compacts prepared by isostatic pressing (ref. 113(SRD)).

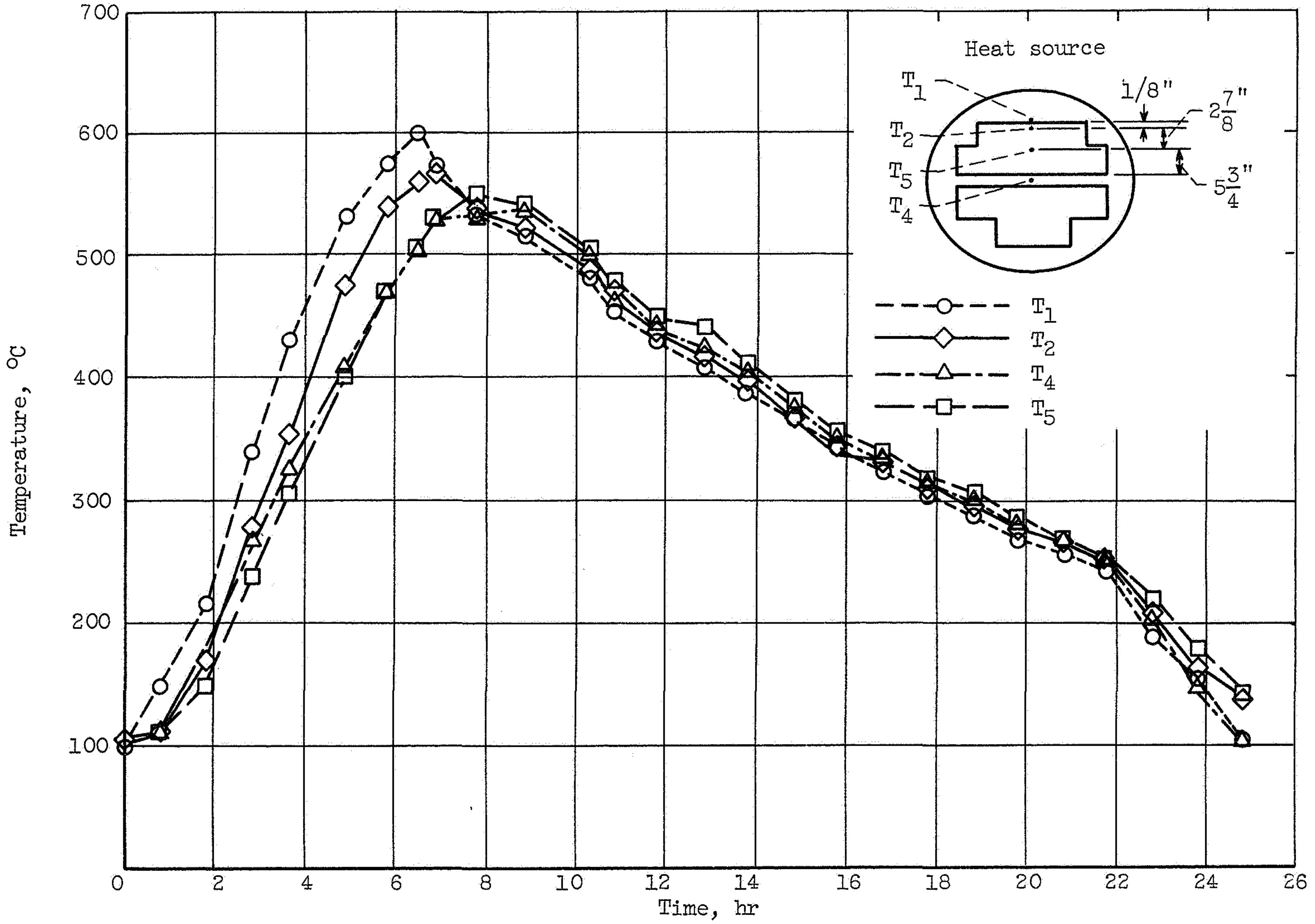


Figure II-8. - Typical thermal cycle of a can containing a cold-pressed block of lithium hydride (ref. 141 (CRD)).

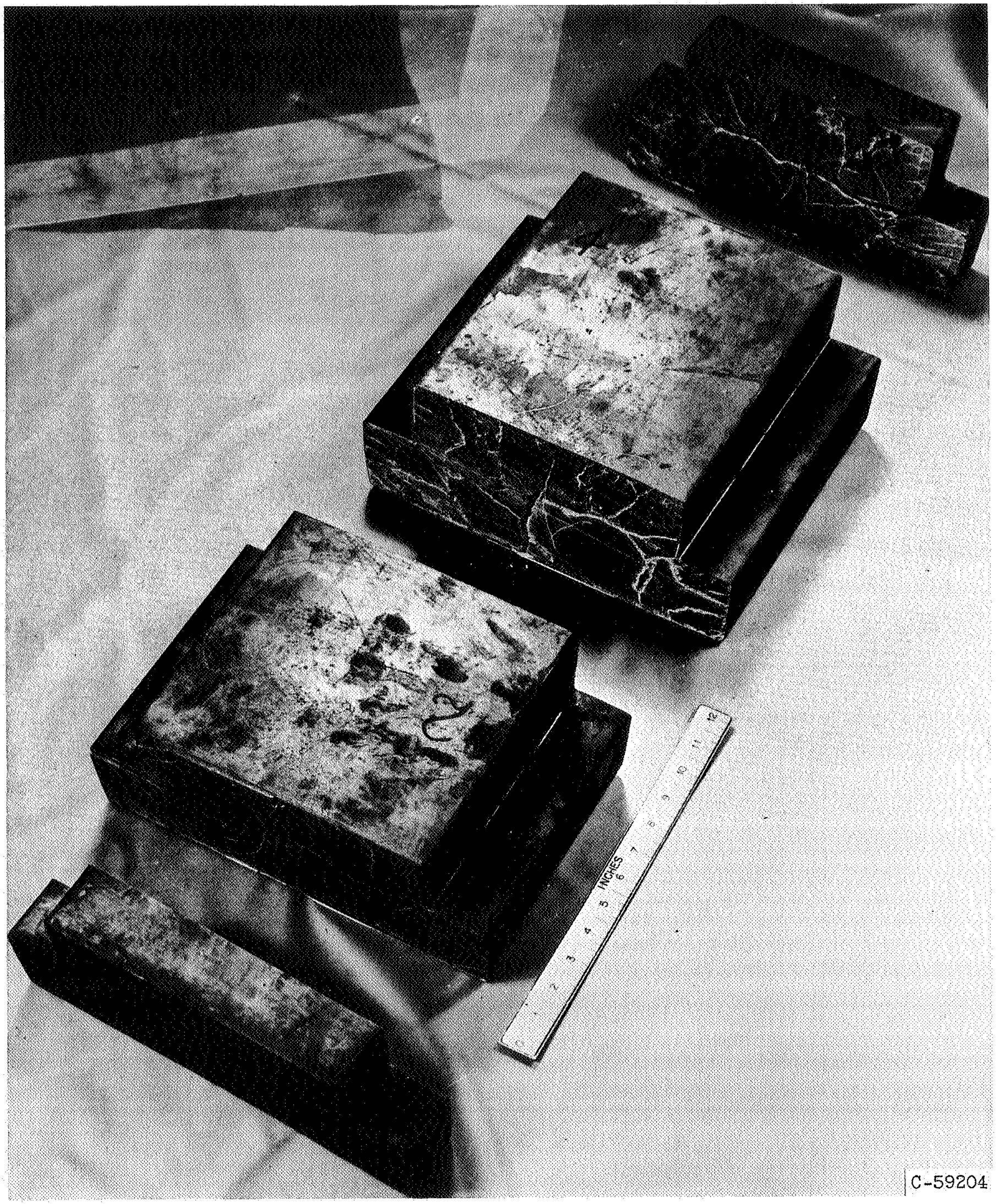


Figure II-9. - Test can containing cold-pressed lithium hydride sectioned after 36 thermal cycles from room temperature to 1100° F. (Ref. 141(CRD)).

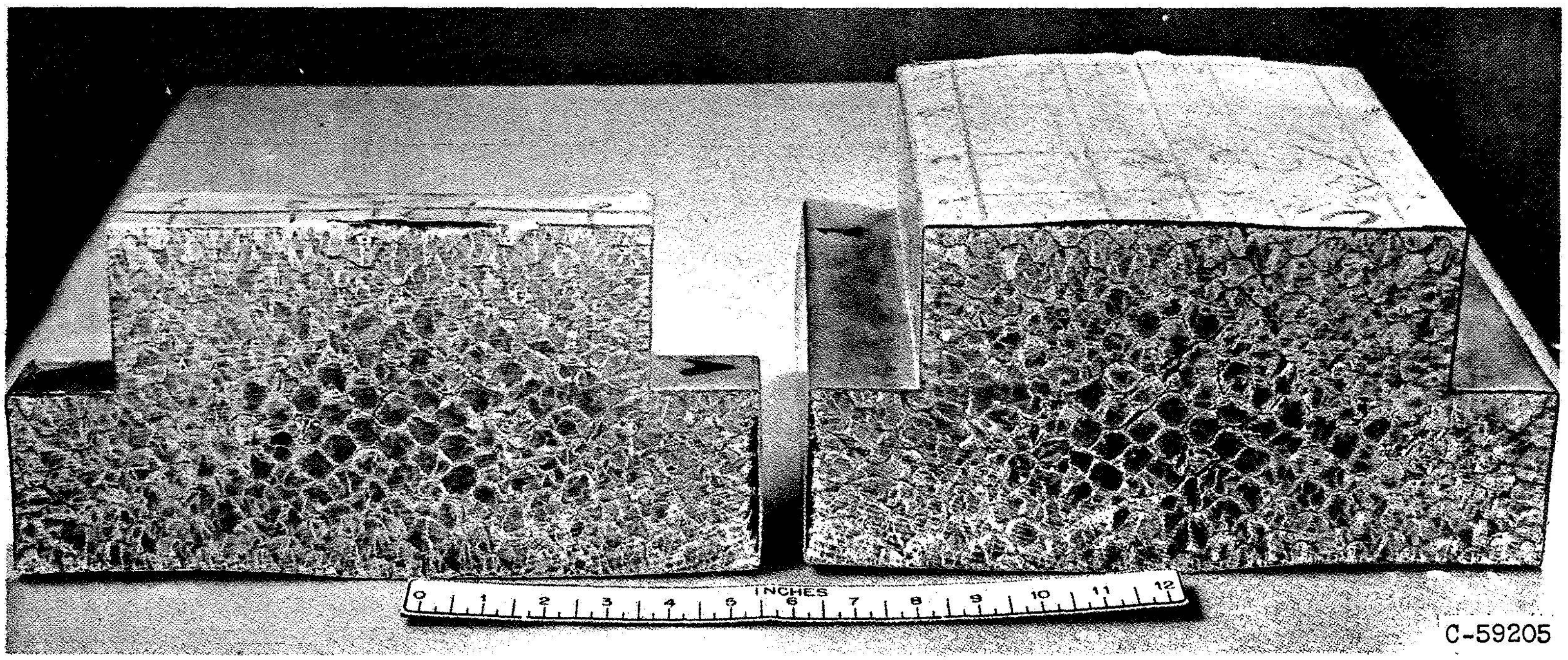


Figure II-10. - Control of cracking by casting lithium hydride in a stainless steel honeycomb. (Ref. 141(CRD)).

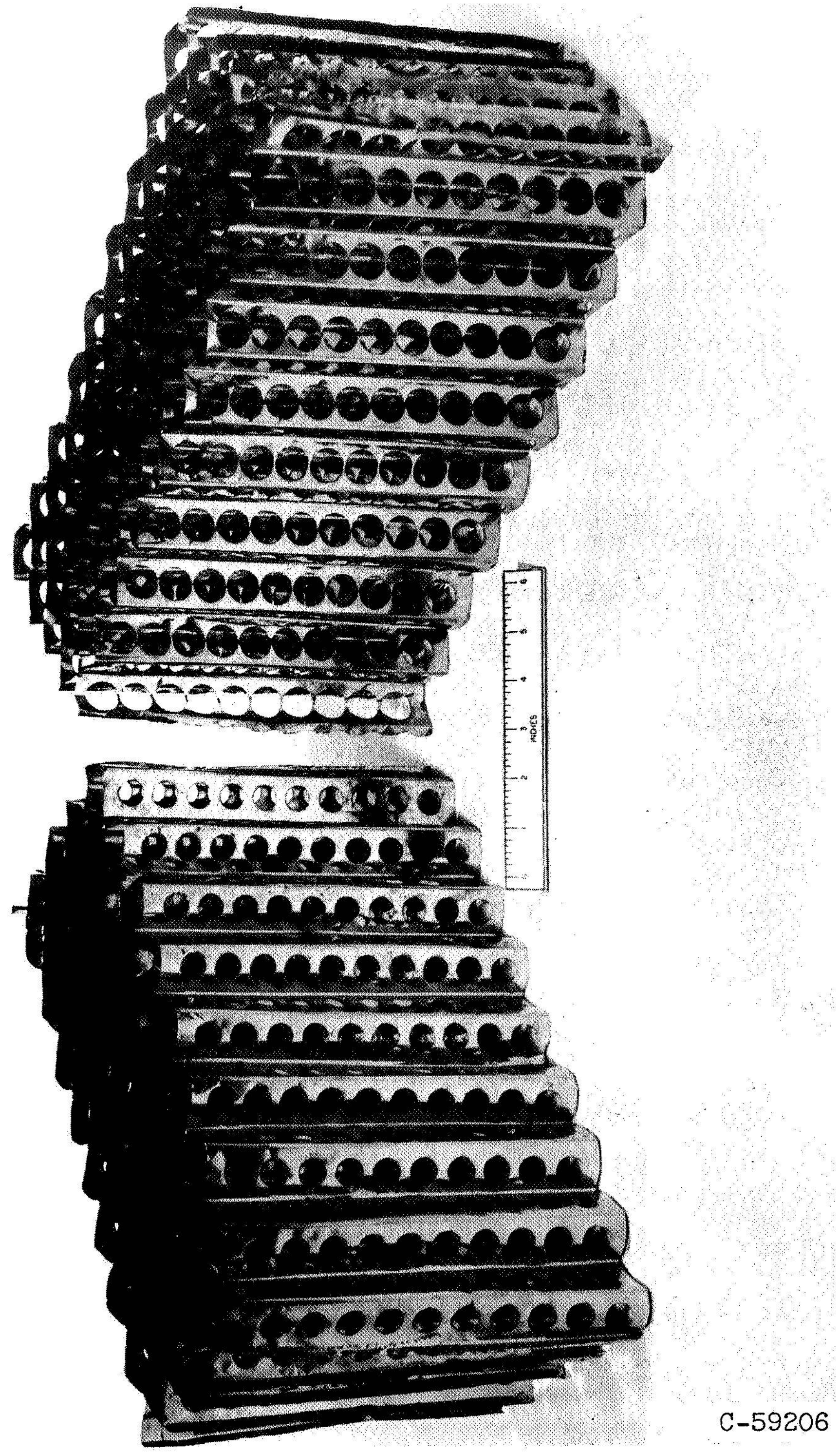


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Figure II-11. - Perforated 0.001-inch-foil stainless steel honeycomb. Left: 1/2-inch holes; right: 11/16-inch holes. (Ref. 141(CRD)).

III. NUCLEAR ENGINEERING PROPERTIES

III. NUCLEAR ENGINEERING PROPERTIES

A. NEUTRON CROSS SECTIONS AND OTHER NUCLEAR INFORMATION

The cross sections and resonance parameters are given in tables III-I, III-II, and III-III. The natural form of lithium contains approximately 7.5 percent lithium-6 and 92.5 percent lithium-7. Cross-section data for lithium and its isotopes over all energy regions are presented in figure III-1 (refs. 181(U) and 182(U)). Additional data on nuclear properties are presented in table III-IV, figures III-2 to III-8, and in the notes that follow.

Casper (ref. 183a(CRD)) made some measurements in water behind arrays of LiH and Li^6H. The abstract of his report is as follows:

> "This report presents the fast neutron dose rates, the gamma dose rates, and the thermal neutron flux measurements made in water behind arrays of LiH and Li^6H. The measurements were made in the pool at the GE-ANPD Source Plate Facility at Battelle Memorial Institute. Fast neutron dose rates, gamma dose rates, and thermal neutron flux measurements behind equal thicknesses of LiH and Li^6H are compared. The effect of varying the LiH and Li^6H thicknesses are [sic] also presented. Point kernel programs were used to compute the fast neutron and gamma dose rates and these values are compared with the measurements."

Excerpts from the results of reference 183b(CRD) are as follows:

> ". . . both LiH and Li^6H are more effective fast neutron shielding materials than water. . . . LiH and Li^6H are both less effective gamma shielding materials than water.
>
> ". . . Li^6H is as effective and perhaps slightly more effective than LiH. . . . the substitution of Li^6H for ordinary LiH would lead to some weight saving.
>
> ". . . the actual fast neutron removal cross section of Li in LiH must be somewhat larger than the value of 0.057 cm^{-1} which was used in these calculations."

Fast-neutron penetration in lithium hydride has been studied and is introduced as follows:

> "Studies of fast-neutron penetration in an infinite medium of lithium hydride have been done by Goldstein and Aronson . . . using the moments method and by Wells . . .

> using Monte Carlo techniques. In order to supplement these studies, Monte Carlo calculations were done using monoenergetic sources of 2, 4, 6, 8, 10, and 14 Mev emitting 1 neutron/sec for penetration distances of 10-120 gm/cm^2.
>
> "Two thousand histories were followed for each input energy and the variance-reduction techniques of forced first collision, stratified sampling, and the exponential transform were employed . . . . The biasing parameter was taken as 0.9 of the total macroscopic cross section at the initial energy." (Ref. 184a(SRD))

The results are summarized as follows:

> "The spectra [for a 2-Mev source] are relatively smooth, with a minimum occurring about 1 Mev and another minimum at 1.6 Mev past 30 gm/cm^2. As might be expected, the relative contribution of neutrons at 1.75 Mev is smaller at the greater penetration distances because of less unscattered flux.
>
> ". . . A minimum occurs [for the 4-Mev source] at 3.25 Mev with a corresponding lessening of the flux at 3.75 Mev with increasing penetration distance. The maximum penetration distance is 60 gm/cm^2 for the 4-Mev case. At this penetration distance the values below 1.5 Mev were unreliable . . . .
>
> ". . . the results for a 6-Mev source out to 120 gm/cm^2 [are given]. The minimum occurs at approximately 4 Mev with a secondary minimum appearing at 1.75 Mev.
>
> ". . . the spectra for an 8-Mev source [are given]. Here, the minimum occurs around 5 Mev. . . . Notice that the higher energy spectra (10- and 14-Mev) are relatively flat and a high percentage of the flux contribution even at distances of 90 and 120 gm/cm^2 are [sic] from high-energy neutrons.
>
> ". . . the total $4\pi r^2$ dose rate [is given] in units of mrem/hr × cm^2 as a function of penetration distance for the various source energies. As expected, the dose rate falls off faster the smaller the source energy, with the 2-Mev dose reduced 6 decades through 50 gm/cm^2 of LiH, while it takes the 14-Mev dose over 120 gm/cm^2 to fall off the same amount." (Ref. 184(SRD))

Hanchon and Haffner (Ref. 185(SRD)) report measured values of fast neutron dose rate and gamma ray dose rate behind 1- and 2-foot thicknesses of lithium hydride. They correlated very well with the values calculated using a modified Albert-Welton point kernel for the attenuation function. The measurements were made in oil (C_7H_{12}; density, 0.882 g/cc) behind the test shield adjacent to the source plate

[redacted]

of uranium enriched to 20 percent uranium 235. The source plate was a disk 28 inches in diameter, 60 mils thick, encased in aluminum, which produced 5.2 watts ± 5 percent. The neutron and gamma radiations were believed to be identical to radiations resulting from a thermal reactor having the same materials and geometry.

For the 1-foot thickness of lithium hydride the log of the fast neutron dose rate varied almost linearly from about 370 millireps per hour at 40 centimeters from the source plate to 0.15 millirep per hour at 100 centimeters. Behind the 2-foot thickness of lithium hydride, the fast neutron dose rate variation with distance from the source plate was the same as that for the 1-foot thickness, but identical dose levels occurred 4 centimeters closer to the source plate.

For the 2-foot thickness of lithium hydride the log of the gamma ray dose rate also varied almost linearly from about 6000 milliroentgens per hour at 80 centimeters from the source plate to about 300 milliroentgens per hour at 147 centimeters.

A great many other shield combinations using other materials with lithium hydride were also tested and reported (refs. 185(SRD) and 186(SRD)).

A computer program that is a multiregion, multilevel, one-dimension diffusion code was used to compute absolute fluxes and foil activations through lithium hydride (and several other materials). Correlations with outside tank tests were made (ref. 187(CRD)).

Some work on removal cross section is reported in reference 188(CRD). See also table III-IV.

Correlation functions were found (ref. 189(U)) for resonance scattering of quanta with excitation of the 5/2-level (7.46 Mev) in Li^7 for two cases of excitation: single-particle and rotational. Figure III-9 gives these results.

The lifetime of the half-energy (0.477 Mev) state of the Li^7 nucleus was experimentally determined to be 1.09×10^{-13} second. This is in good agreement with the value obtained when calculated on the assumption that the half-energy level (0.47 Mev) of Li^7 is the result of a rotation of the triton spin and not of a nucleon (ref. 189(U)).

> "Measurements of the capture γ-radiation processes, mass $3+\alpha \longrightarrow$ mass $7+\gamma$ and nucleon$+Li^6 \longrightarrow$ mass $7+\gamma$, give information about the cluster structure of the mirror nuclei Li^7 and Be^7. The cluster model predicts that the ground state and low excited states of these nuclei should have

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large reduced widths $\theta_{3+4}{}^2$ for the configuration mass 3+α particle and small reduced widths $\theta_{1+6}{}^2$ for the configuration nucleon+Li^6. Scattering experiments provide accurate initial, capturing, wave functions, and an assumption of the cluster nature of the final, bound, states allows the electromagnetic capture cross sections to be calculated and compared to experiment. The reduced widths deduced show that $\theta_{3+4}{}^2$ is large, $\theta_{1+6}{}^2$ is small, and that the ground states and first excited states of Li^7 and Be^7 are primarily of the two-body cluster form mass 3+α particle." (Ref. 190(U))

B. RADIATION EFFECTS

1. Damage

Reference 191(U), concerning radiation effects on lithium hydride, is quite a comprehensive report, with 107 references of its own. Actually, most of the report concerns mixtures of the hydride with the deuteride and the tritide, but the conclusions and results would probably be similar for any of these materials. Excerpts from the abstract of this report are as follows:

> "Extensive measurements of the effects of radiation have been made on various compositions of Li hydrides containing combined T. The isothermal expansions of samples exposed to a flux of tritium β radiation equivalent to 16.8 Mr/hr have been observed at 12 temperatures between -196° and $400^{\circ}C$ for maximum exposures up to 7 years. The results of these measurements are correlated with other data and are used to formulate a model for radiation damage in the Li hydrides.
>
> . . . .
>
> "Mechanical properties, phase separation, gas evolution, and other properties of the samples exposed to intense β radiation from combined T were also observed and correlated with the isothermal expansion data to formulate a model for the radiation effects. . . . One interesting conclusion obtained from these studies is the result that little dissociation and cavitation occurs in samples stored at temperatures below $-7^{\circ}C$, whereas these effects are extensive and tend to lead to the disintegration of samples stored at $23^{\circ}C$ or higher." (Ref. 191(U))
>
> "It seems most reasonable to assume that the net effect of radiation damage on LiH will result in the chemical dissociation of the material with the possibility of recombination by a back reaction as indicated in the equation:
>
> $$\mathrm{LiH} \overset{\beta}{\rightleftharpoons} \left(\begin{array}{l}\text{Intermediate}\\ \text{Metastable}\\ \text{Products}\end{array}\right) \rightleftharpoons \mathrm{Li} + \frac{1}{2}\,\mathrm{H_2}$$
>
> "The extent to which the back reaction occurs in the first stage of the latter equation is most important to the resistance of the material to permanent radiation damage. If recombination is rapid and the steady-state concentration of metastable products is comparatively small, the net effect of the radiation on the material may be nearly negligible because all of the energy is dissipated as heat. On the other extreme, all of the energy could go to form

[redacted]

dissociation products which do not recombine at all under the conditions of the irradiation; this could lead to the dissociation of about 300 molecules of LiH per particle, assuming that 20 ev is required per dissociated molecule." (Ref. 191b(U))

"Radiation damage tests made on lithium hydride have not presented any serious behavior that might impair its usefulness as a shield material. . . .

"Large crystals of lithium hydride retain their gross structure when exposed to pile radiation in the order of 10^{17} nvt and no change in the lattice constants of unirradiated and irradiated samples have [sic] been detected when examined by X-ray diffraction. However, X-ray diffraction studies have shown the formation of polycrystalline aggregates resulting presumably from the stresses caused by thermal spikes. The fracture of the material into polycrystalline aggregates can be demonstrated by the relative ease of powdering irradiated lithium hydride in contrast to the unexposed material.

"The normally clear crystal is darkened at exposures as low as 10^{15} nvt. Paramagnetic resonance studies . . . on lithium hydride crystals irradiated with gamma rays from a cobalt-60 source at liquid nitrogen temperature have revealed the presence of F-centers. Attempts to anneal out the color completely have been unsuccessful.

"Irradiations performed by GE-ANPD on cast and powder pressed lithium hydride, confined tightly in stainless steel containers resulted in negligible pressure or expansion effects after 6 × 10^{18} nvt exposure at approximately 1000°F. During irradiation the samples were heated entirely by neutron effects, and the temperature of lithium hydride maintained at approximately 1000°F by adjusting the flow of cooling helium gas. The results are summarized in Table [III-V]. The lithium hydride was blackened - but remained otherwise unchanged.

"Experiments performed by NDA [ref. 192(SRD)] have indicated that powder-pressed lithium hydride will expand when unconfined after exposures as low as 10^{16} nvt, and at temperatures up to 600°F. The results are shown in Table [III-VI]. Their conclusions are that growth and disintegration of the cold-pressed pellets is caused by reactor radiation rather than elevated temperatures, whereas gas evolution is caused by reaction of impurities in the lithium hydride at the elevated temperatures, and is only slightly affected by radiation." (Ref. 19a(CRD))

C[redacted]

[redacted]

Further work (ref. 193(SRD)) indicates very little radiation damage to contained lithium hydride irradiated under the following ranges of conditions: 200° to 1000° F, up to 744 hours, 1.9×10^{15} to 2.1×10^{19} nvt dose, and 5.2×10^{9} to 2.3×10^{13} neutrons per square centimeter flux. There were 14 cold-pressed samples and 9 cast samples.

Two specimens showed powdering or swelling during irradiation. A leak was found in one and suspected in the other. These were the samples that were tested for the longest times and sustained the highest dosage. In all other cases the lithium hydride was recovered as solid, crystalline material either whole or in large chunks. The conclusions reached from this test are as follows:

> "In general, no deleterious effects to LiH are noted from 100 hours of exposure to thermal neutrons of 10^{16} - 10^{18} nvt at temperatures of 200 - 1000° F. It is believed, although not established, that the entrance of air or water vapor during irradiation or the presence of LiOH in non-outgassed or improperly outgassed LiH contributed to the swelling or powdering as evidenced in the two cases emphasizing the necessity of maintaining the LiH in a hermetically sealed container or an inert atmosphere."
> (Ref. 193(SRD))

Aitken and Henry describe their radiation damage experiments in reference 194(CRD). The lithium hydride samples, both pressed powder and cast, were placed in stainless-steel 304 containers and were kept at approximately 1000° F during the test. The thermal dosage was 4×10^{18} nvt, and the fast dosage was 10^{18} nvt. The time of the test was 100 hours. Excerpts from the summary and conclusions follow:

> "The in-pile experiments on lithium hydride at 1000°F revealed no measurable effects with regard to gas evolution or dimensional change. . . . Nuclear heating from thermal capture was rather extensive, and the activity arising from impurities in the lithium hydride was minor when compared with the activity induced in the stainless steel container.
>
> "The negative results obtained . . . conflict with those observed by Minushkin. [Note that Minushkin's highest observed temperature was 600° F. Self-annealing would probably not take place at this temperature as it would at 1000° F. Also, the cohesive energy of the powder-pressed material used by Minushkin is relatively poor, making radiation damage more severe.]

[redacted]

> ". . . it is concluded that the effects of radiation on lithium hydride are not serious enough to present difficult problems with respect to contamination or growth in high-temperature reactor-shield assemblies contemplated . . .. However, it does appear that some microscopic radiation damage effects are produced by radiation, such as microcrystalline fracturing and helium retention in vacant lattice sites, and annealing effects." (Ref. 194(CRD))

2. Positron Annihilation

> "The angular correlation of photons from positron annihilation in LiH . . . has been measured. The data yield a wave-function product density distribution much wider than the outer-shell electron density around the negative ion. This result is in contrast with the observations for other alkali halides for which these two distributions are much alike." (Ref. 195(U))

3. Atomic Disintegration Reactions

Reference 196(U) reports several atomic disintegration reactions which have been performed and which involve lithium, hydrogen, and deuterium. They are:

$$ {}^{9}_{4}\mathrm{Be} + {}^{2}_{1}\mathrm{D} = {}^{7}_{3}\mathrm{Li} + {}^{4}_{2}\mathrm{He} $$

$$ {}^{7}_{3}\mathrm{Li} + {}^{2}_{1}\mathrm{D} = {}^{8}_{4}\mathrm{Be} + {}^{1}_{0}\mathrm{N} $$

$$ {}^{6}_{3}\mathrm{Li} + {}^{1}_{1}\mathrm{H} = {}^{4}_{2}\mathrm{He} + {}^{3}_{2}\mathrm{He} $$

$$ {}^{6}_{3}\mathrm{Li} + {}^{1}_{0}\mathrm{N} = {}^{3}_{1}\mathrm{H} + {}^{4}_{2}\mathrm{He} $$

[redacted]

[redacted]

C. HYDROGEN NUMBER

> "For some possible applications of metal-hydrogen compounds, an important consideration is the number of hydrogen atoms per cubic centimeter. The factor is usually designated by N_H." (Ref. 13d(U))

E-1332

The following table shows the calculated N_H of pure lithium hydride at various values of density (ref. 13d(U)):

Density, g/cc	$N_H \times 10^{-22}$
0.65	4.967
.70	5.349
.75	5.732
.78	5.961
.80	6.114

[redacted]

[redacted]

TABLE III-I. - THERMAL CROSS SECTIONS OF LITHIUM, ITS ISOTOPES, AND HYDROGEN (Ref. 181(U))

Element	Isotope (percent)	Reaction cross sections (2200 m/sec)		Scattering cross sections, barns		
		Absorption, σ_{abs}	Activation, σ_{act}	Coherent, σ_{coh}(sign)	$\sigma_{fa}\left(\frac{A+1}{A}\right)^2$ (*)	Average scattering, $\overline{\sigma_s}$
$_3$Li		71.0±1.0 b		0.40±0.03(-)	1.2±0.3	1.4±0.3
	Li^6(7.52)	(945b)	nγ 28±8 mb	6±3(+)		
	Li^7(92.48)		0.85 sec 33±5 mb	0.80±0.05(-)	1.4±0.2	
$_1$H		332±2 mb		1.79±0.02(-)	81.5±0.4	38±4(gas)
	H^1(~100)					
	H^2(0.015)	0.46±0.10 mb	12.4 yr 0.57±0.01 mb	5.4±0.3(+)	7.6±0.1	7±1

*σ_{fa} = free atom cross section, corrected

TABLE III-II. - RESONANCE PARAMETERS OF LITHIUM ISOTOPES (Ref. 181(U))

Isotope	Spin of target nucleus, I	Energy of resonance, E_0, kev	Compound nucleus level spin, J	Neutron angular momentum, l	Level width, Γ, kev
$_3Li^6$	1	284±4	5/2	1	Γ90±10 $*\Gamma_n$60±15 $*\Gamma_\alpha$30±10
$_3Li^7$	3/2	258±3	3	1	$*\Gamma_n$35±5

$*\Gamma_n$ = neutron elastic scattering level width.

$*\Gamma_\alpha$ = α-particle level width.

TABLE III-III. - NEUTRON TOTAL CROSS SECTIONS OF HYDROGEN (Ref. 197(U))

[Energies are given together with limits which specify width at half maximum of corresponding energy distributions.]

Energy, Mev	Cross section, mb	Energy, Mev	Cross section, mb
15.80±0.33	537.0±30	34.03±1.01	260.1±7.2
16.13±.33	634.0±34	35.08±1.07	245.8±6.7
16.46±.34	580.0±33	36.20±1.12	220.0±9.9
16.80±.35	597.0±32	37.32±1.15	226.8±6.3
17.15±.36	561.0±29	38.48±1.21	223.5±6.0
17.52±.37	544.0±28	39.75±1.29	219.1±5.7
17.90±.39	533.0±27	41.10±1.38	204.6±5.4
18.29±.40	517.0±27	42.53±1.45	196.0±5.2
18.69±.42	523.0±28	44.00±1.50	189.1±4.9
19.11±.43	498.0±40	45.50±1.60	187.3±4.6
19.55±.44	522.0±26	47.10±1.70	170.3±4.0
20.00±.46	479.0±24	48.80±1.80	166.8±4.1
20.46±.47	479.0±22	50.60±1.90	152.0±3.8
20.93±.48	449.0±21	52.50±2.0	152.0±3.6
21.41±.50	447.0±20	54.50±2.1	144.8±3.3
21.91±.51	420.0±20	56.60±2.2	141.5±3.1
22.43±.52	408.0±20	58.80±2.3	135.8±2.9
22.96±.54	433.0±19	61.10±2.4	124.3±2.8
23.51±.57	397.7±16	63.50±2.5	116.8±2.6
24.09±.59	393.0±15	66.10±2.7	114.9±2.5
24.69±.61	393.5±14	68.90±3.0	109.6±2.3
25.31±.63	362.9±13.5	72.00±3.2	100.1±2.3
25.94±.65	377.6±12.7	75.30±3.5	100.3±3.6
26.60±.68	345.7±12.3	78.90±3.8	94.7±2.1
27.29±.72	335.4±11.5	82.80±4.0	85.3±1.9
28.03±.76	321.5±11.0	86.90±4.3	82.1±1.9
28.80±.78	312.2±10.5	91.30±4.6	77.7±1.7
29.59±.80	309.1±9.5	96.00±4.8	72.1±1.6
30.40±.83	281.7±8.8	101.00±5.0	71.5±1.7
31.24±.86	286.4±8.3	106.00±5.3	69.4±1.6
32.12±.90	288.7±7.4	111.50±5.7	68.3±2.1
33.05±.96	276.0±6.8	117.50±6.0	

[redacted]

TABLE III-IV. - SOME NUCLEAR PROPERTIES OF LITHIUM HYDRIDE AND ITS CONSTITUENTS

Type of cross section	Material	Density	Value	Energy	Reference
Removal	LiH	---------------	0.1143 cm^{-1}	----------------	115(CRD)
Fast neutron removal	Li^{N}H Li in LiH Li^N	--------------- --------------- 5.64x10^{22} $\frac{atoms}{cu\ cm}$	2.01 b 0.057 cm^{-1} 0.07 cm^{-1}	---------------- ---------------- ----------------	113d(SRD) 183(CRD) 198(SRD)
Macroscopic removal	LiH	0.92	0.14 cm^{-1}	----------------	199(U)
Macroscopic fast neutron removal	LiH	---------------	0.118 cm^{-1}	----------------	113d(SRD)
Microscopic removal	Li^N	5.64x10^{22} $\frac{atoms}{cu\ cm}$	1.24 b	----------------	198(SRD)
Thermal absorption	H_2 Li	--------------- ---------------	0.335±0.005 b 70.4±0.4 b	Thermal neutrons Thermal neutrons	200(U) 200(U)
Differential, $\sigma(n,\alpha)$	Li^6(n,α) Li^6(n,α)	--------------- ---------------	See fig. III-10 0.215±0.030 b	2.15-Mev neutrons 2.15-Mev neutrons	201(U) 201(U)
Total, σ_T	H_2 H_2 Li^6 Li^7 Li^6 Li^7 Li^N	--------------- --------------- --------------- --------------- --------------- --------------- ---------------	See table III-VI 0.494±0.0025 b 1.40±0.14 b 2.0±0.3 b 1.37±0.06 b 1.35±0.08 b 1.24±0.06 b	16-111-Mev neutrons 19.665±0.026-Mev neutrons 2.5-Mev neutrons 2.5-Mev neutrons 14-Mev neutrons 14-Mev neutrons 14.8-Mev neutrons	197(U) 202(U) 203(U) 203(U) 203(U) 203(U) 204(U)

Other quantities	Material	Density	Value	Energy	Reference
Angular distribution of neutron dose rate	LiH, Li^{6}H 50 cm thick	---------	See fig. III-11	-----------	184(SRD)
Angular distributions	Li^6(n,t)He^4 reaction	---------	See figs. III-2 to III-8	150-565 kev	205(U) and 206(U)
Differential, scattering, $\sigma_s(\theta)$	Li^N and Li^6	---------	See figs. III-12 and III-13	50-2300-Mev neutrons	207(U)
Mass-absorption coefficient, μ	LiH pressed powder	0.70 g/cu cm 0.70 g/cu cm 0.70 g/cu cm	0.0444 cm^{-1} 0.0239 cm^{-1} 0.0172 cm^{-1}	1 Mev 3 Mev 6 Mev	199(U) 199(U) 199(U)
Mass attenuation coefficient	LiH	---------	0.152 cm^2/g	-----	113d(SRD)
Shielding effect	LiH	---------	See fig. III-14	-----	183(CRD)
Fast-neutron penetration	LiH LiH	--------- ---------	See section III.A. See section III.A.	2, 4, 6, 8, 10, and 14 Mev (See section III.A.)	184(SRD) 185(SRD)
Correlation functions	Li^7	---------	See section III.A. and fig. III-9	7.46 Mev (see section III.A.)	189(U)
Lifetime	Li^7	---------	1.09x10^{-13} sec	1/2 - (0.477 Mev) state (See section III.A.)	189(U)
Cluster nature	Li^7	---------	See section III.A.	Half energy (0.477 Mev) state (See section III.A.)	190(U)

[redacted]

TABLE III-V. - RESULTS OF NEUTRON IRRADIATION OF LITHIUM HYDRIDE (Ref. 19b(CRD))

Time:	100 hr	
Flux: Thermal neutrons Fast neutrons	0.3×10^{13} n/cm^2/sec $.3 \times 10^{13}$ n/cm^2/sec	
Dosage: Thermal neutrons Fast neutrons	9×10^{18} nvt 1×10^{18} nvt	
	Cast	Powder-pressed
Average temperature, center	970° F	972° F
Average temperature, periphery	818° F	738° F
Average temperature gradient	152° F	234° F
Average helium flow rate	3.5 scfm	3.8 scfm
Pressure change of LiH	<10 mm	<10 mm
Change in dimensions of container	None	None

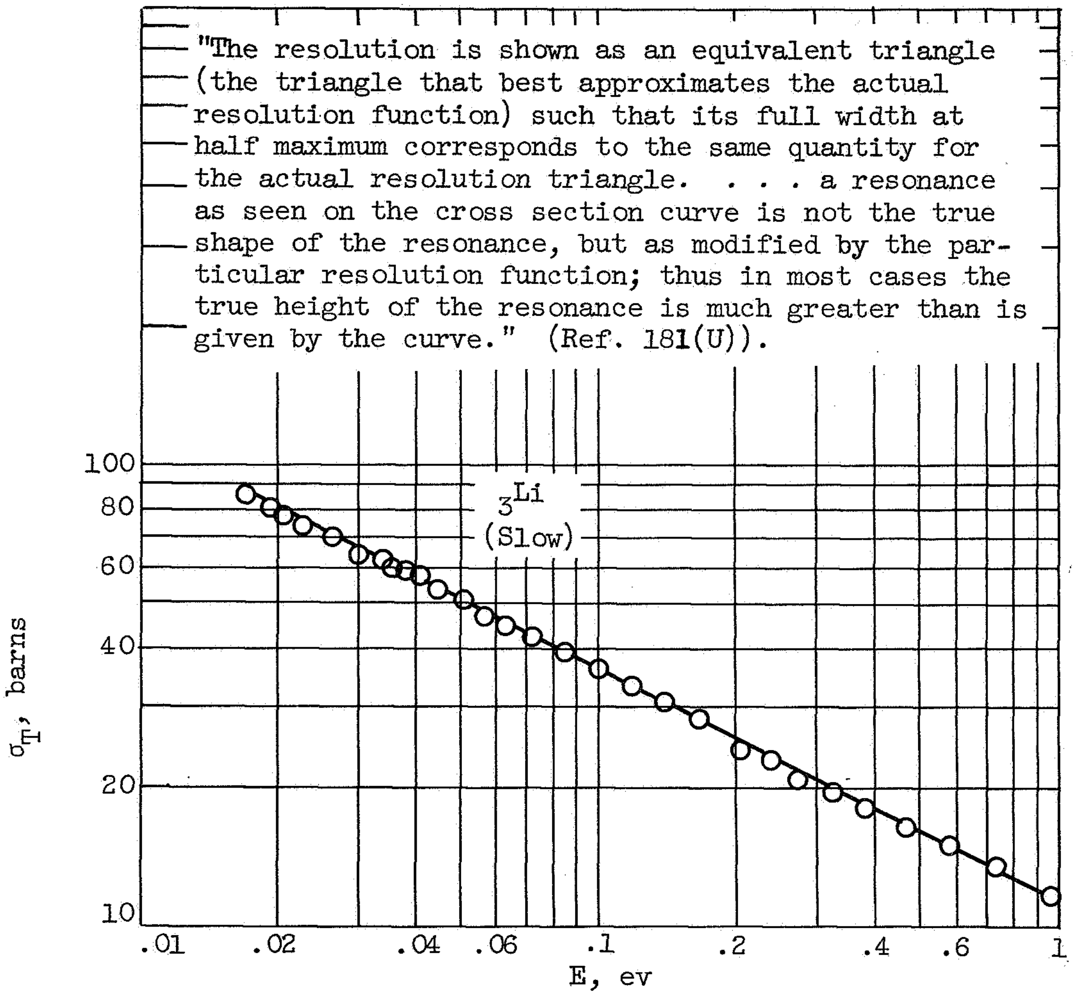
TABLE III-VI. - LITHIUM HYDRIDE RADIATION STABILITY TESTS ON COMPACTED POWDER

(Refs. 19b(CRD) and 192(SRD))

Run	Integrated dosage, nvt	Irradiation temperature, °F	Maximum pressure, $\frac{\text{lb}}{\text{sq in. abs}}$	Notes
1	[a]2×10^{16}	400	10	Sample blackened and expanded to fill capsule
2	[a]2×10^{16}	400	7.8	Sample blackened and expanded to fill capsule
3	[b]1.05×10^{18}	400 to 610	11.9	Sample blackened, outside surfaces appear to have flaked off, leaving coarse powder and cylinder of smaller diameter than original
4	[b]1.12×10^{18}	400 to 610	15	Sample blackened and completely disintegrated to a coarse crystalline powder
5	[b]1.55×10^{15}	100	10	Capsule contained 6 lb/sq in. abs He overpressure; samples slightly blackened but no evidence of growth or disintegration
6	0	400 to 600	25	After 2 days, no sign of blackening or growth
7	0	400 to 600	25	After 7 days, no sign of blackening or growth

[a] Based on unperturbed thermal neutron flux as reported by Brookhaven National Laboratory for sample location, with allowance for flux depression by sample.

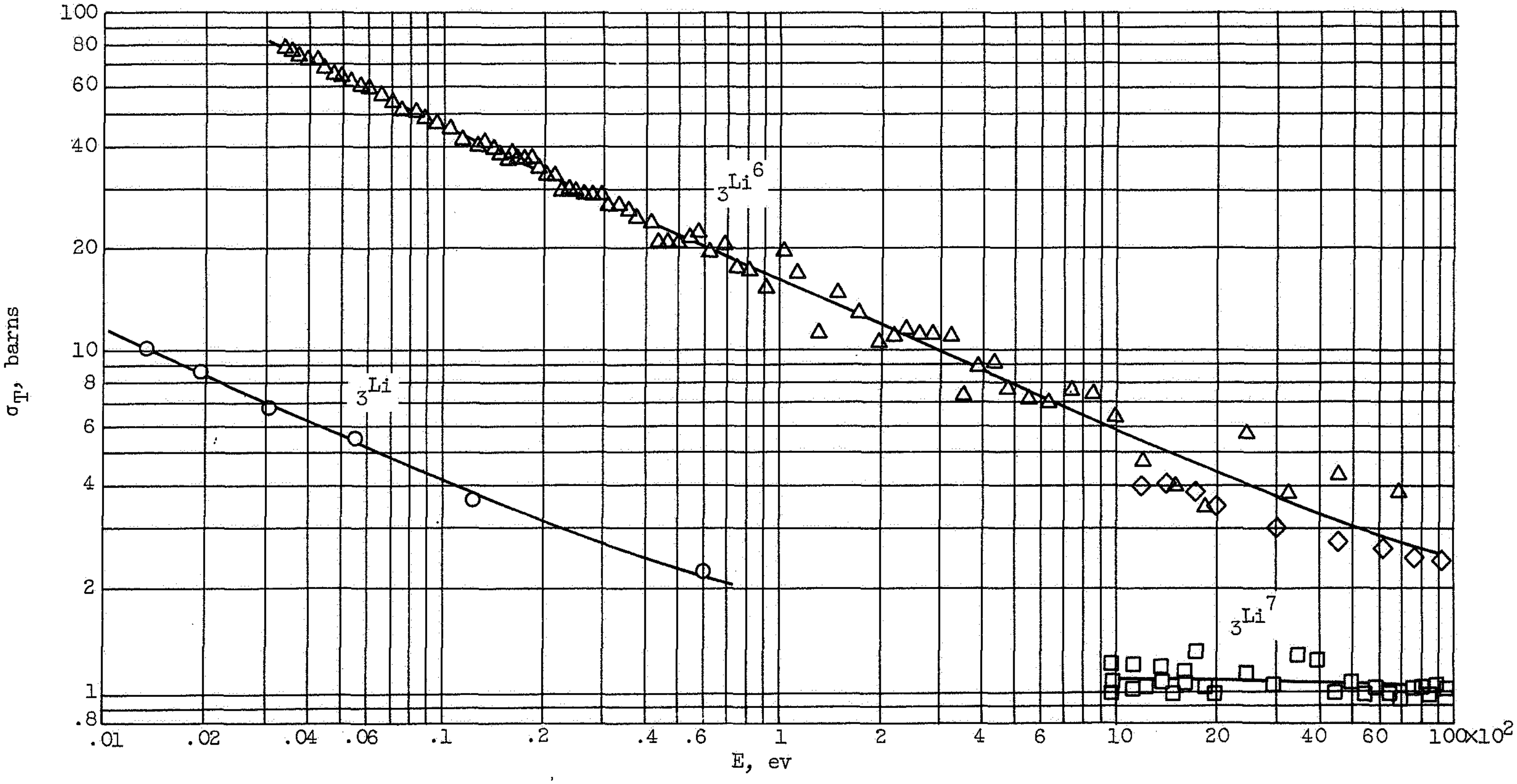
[b] Based on count of cobalt monitor wires wrapped around samples.



"The resolution is shown as an equivalent triangle (the triangle that best approximates the actual resolution function) such that its full width at half maximum corresponds to the same quantity for the actual resolution triangle. . . . a resonance as seen on the cross section curve is not the true shape of the resonance, but as modified by the particular resolution function; thus in most cases the true height of the resonance is much greater than is given by the curve." (Ref. 181(U)).

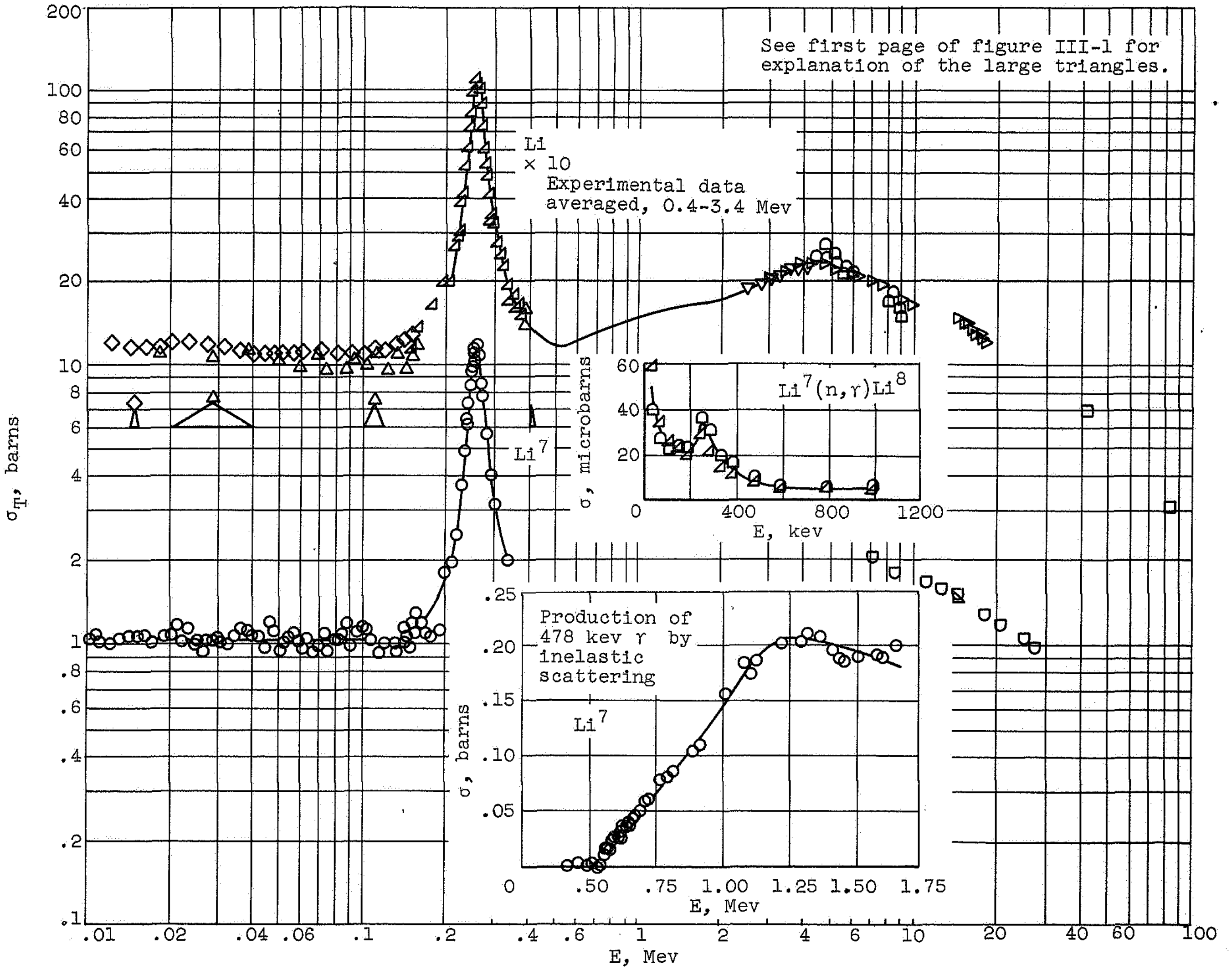
(a) Reference 181(U).

Figure III-1. - Neutron cross sections.



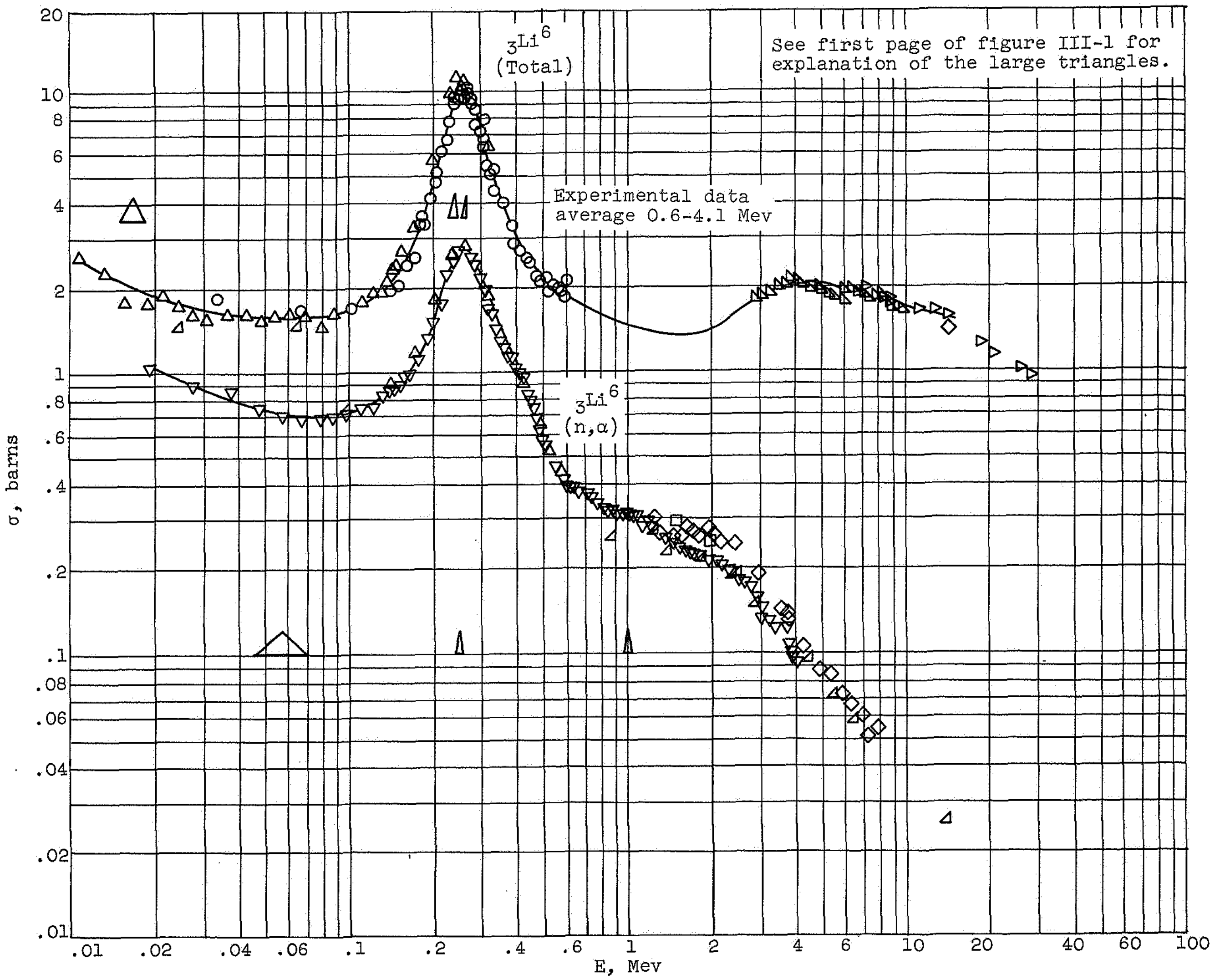
(a) Concluded. Reference 181(U).

Figure III-1. Continued. - Neutron cross sections.



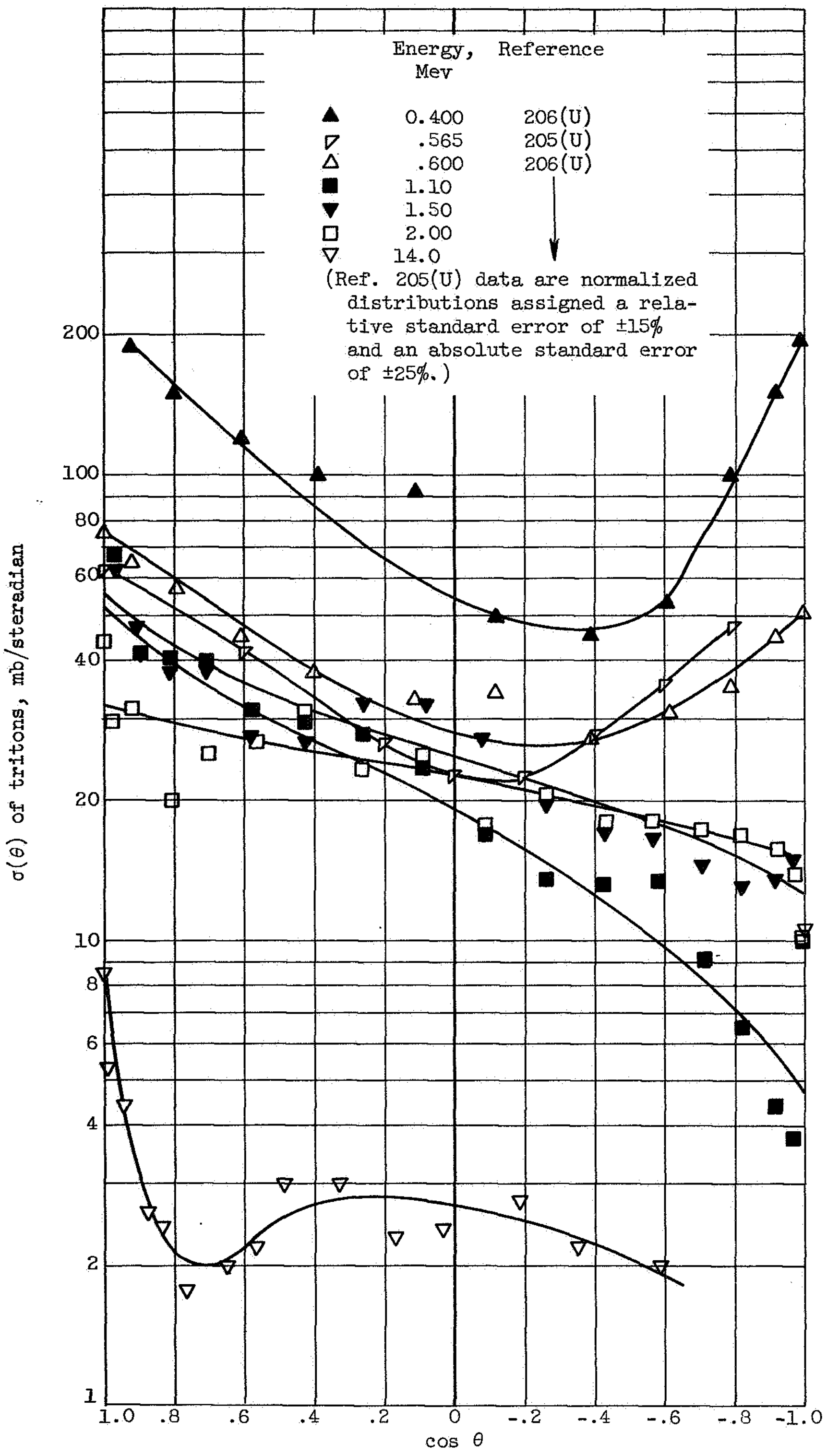
(b) Reference 182(U).

Figure III-1. - Continued. Neutron cross sections.



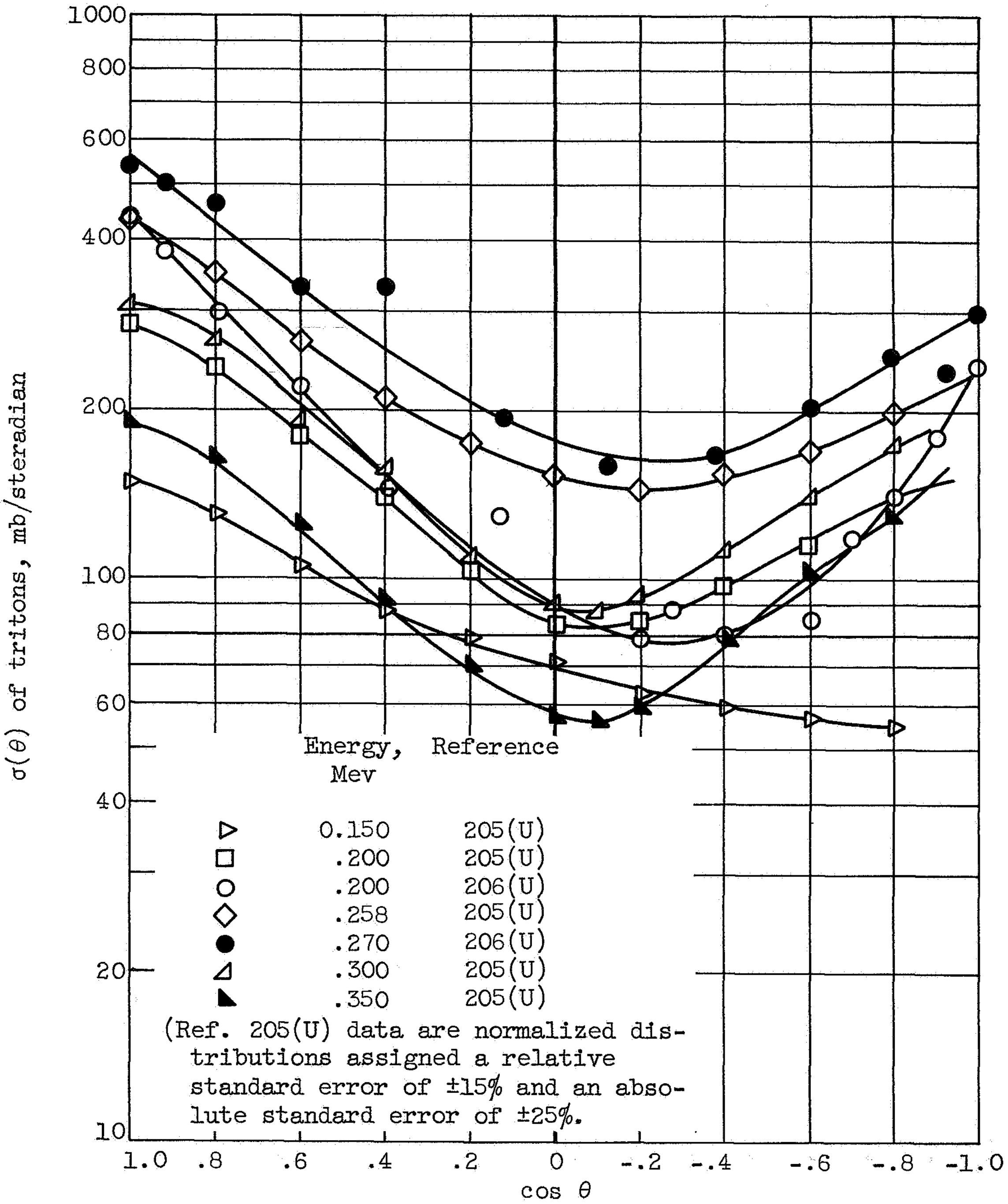
(b) Concluded. Reference 182(U).

Figure III-1. - Concluded. Neutron cross sections.



(Ref. 205(U) data are normalized distributions assigned a relative standard error of ±15% and an absolute standard error of ±25%.)

Figure III-2. - Angular distribution of tritons from $Li^6(n,t)He^4$ reaction from 0.400 to 14.0 Mev.



(Ref. 205(U) data are normalized distributions assigned a relative standard error of ±15% and an absolute standard error of ±25%.

Figure III-3. - Angular distribution of tritons from $Li^6(n,t)He^4$ reaction for 0.150 to 0.350 Mev.

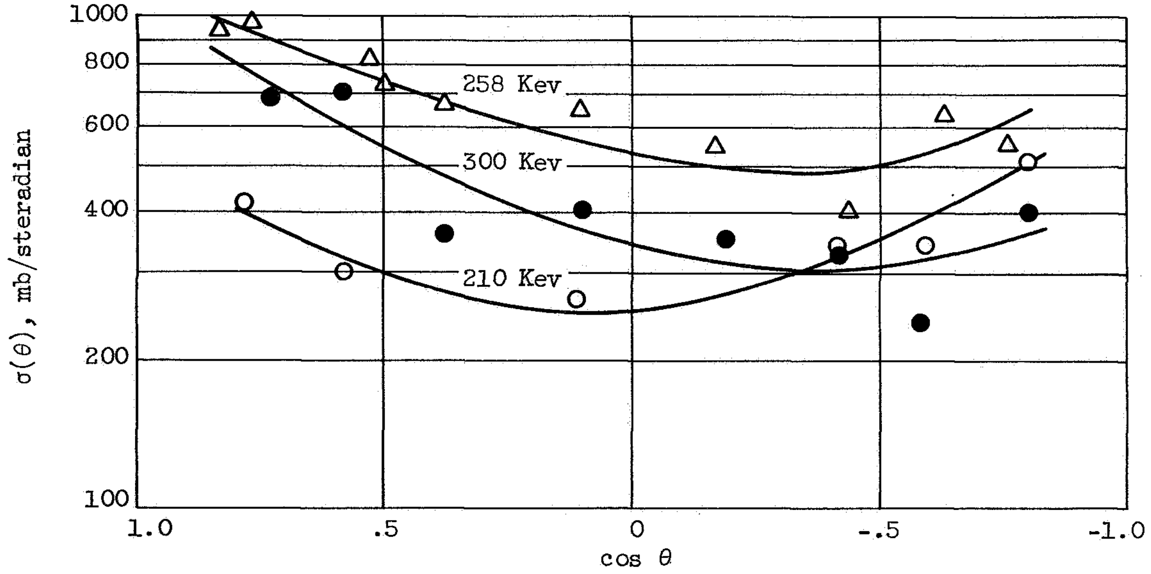


Figure III-4. - Absolute differential cross sections of $_3Li^6$ as function of scattering angle for elastic scattering (ref. 206(U)).

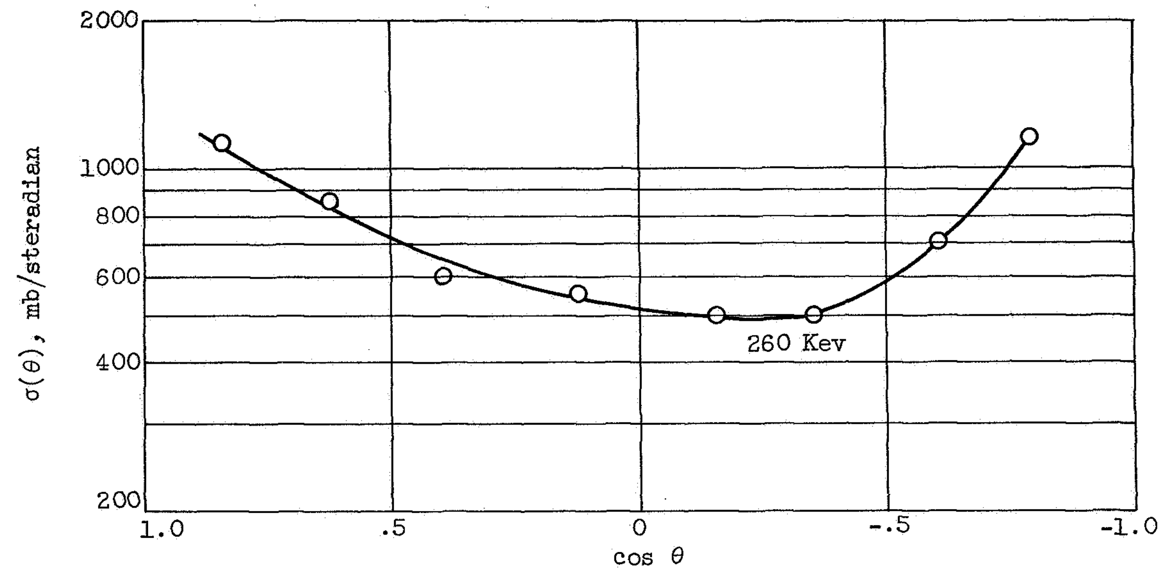


Figure III-5. - Absolute differential cross sections of $_3$Li as function of scattering angle for elastic scattering (ref. 206(U)).

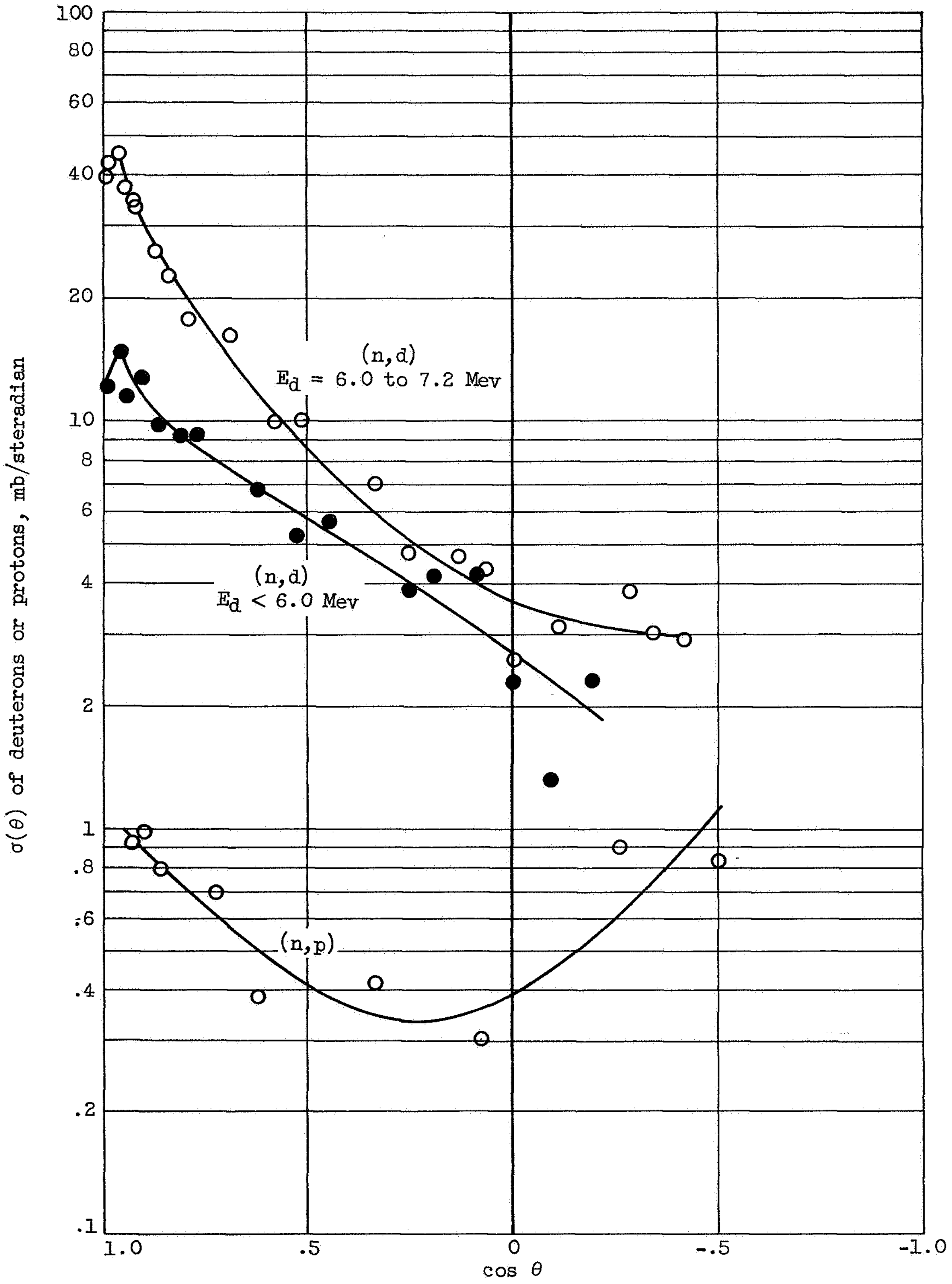


Figure III-6. - Absolute differential cross sections of $_3Li^6$ as function of scattering angle for (n,d) and (n,p) reactions (ref. 206(U)).

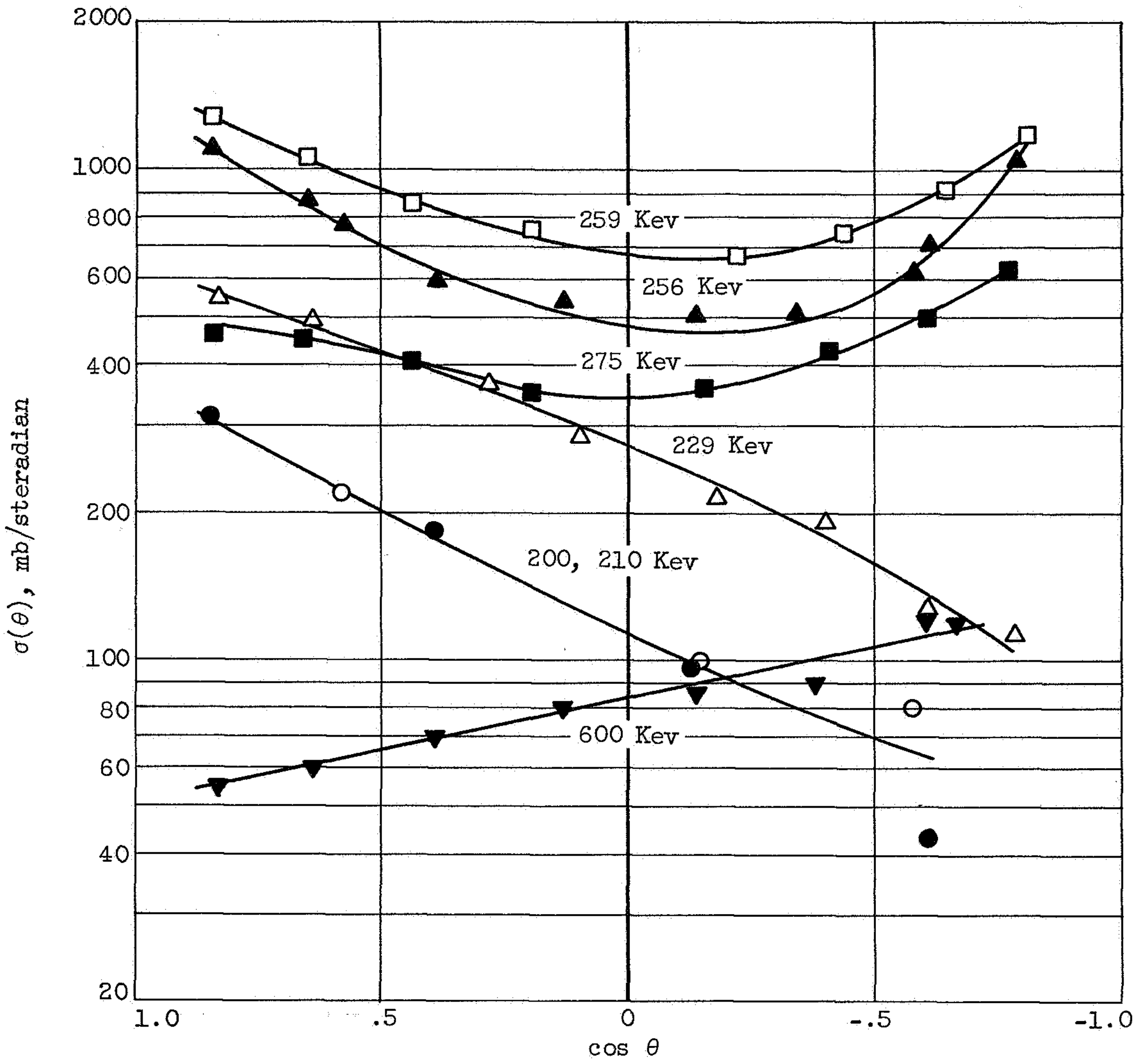


Figure III-7. - Absolute differential cross sections of $_3Li^7$ as function of scattering angle for elastic scattering (ref. 206(U)).

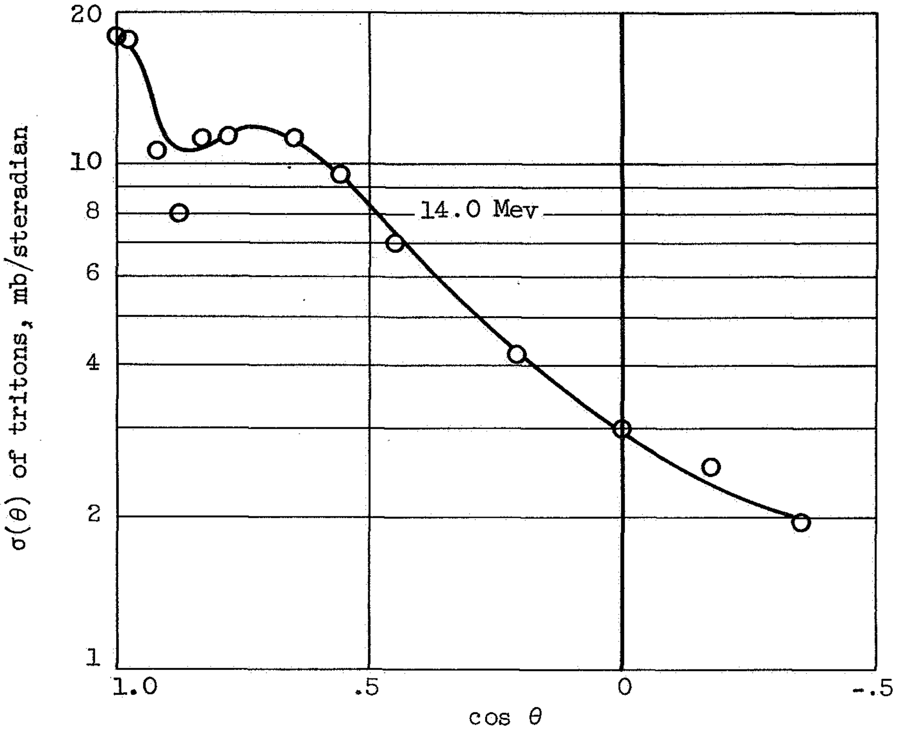


Figure III-8. - Absolute differential cross sections of $_3Li^7$ as function of scattering angle for (n,t) reaction (ref. 206(U)).

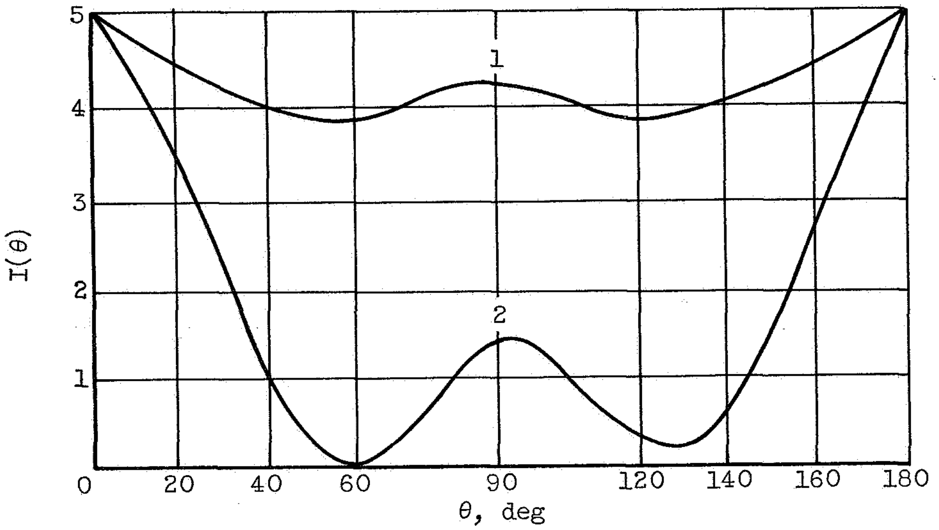


Figure III-9. - Correlation functions for Li^7 (ref. 189(U)). (Resonance scattering of quanta with excitation of the 5/2-level (7.46 Mev). Curve 1 corresponds to single-particle excitation; curve 2 to collective, i.e., rotational excitation.)

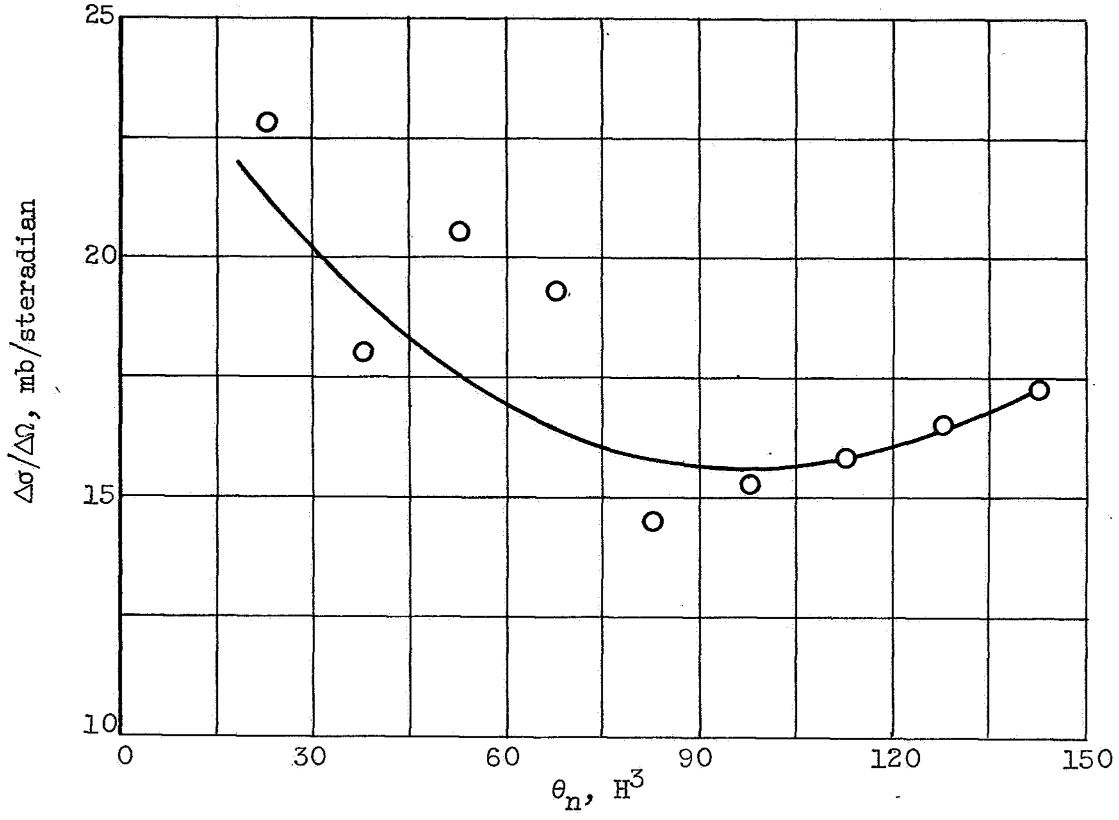


Figure III-10. - Differential cross section for $Li^6(n,\alpha)$ for 2.15-Mev neutrons (ref. 201(U)).

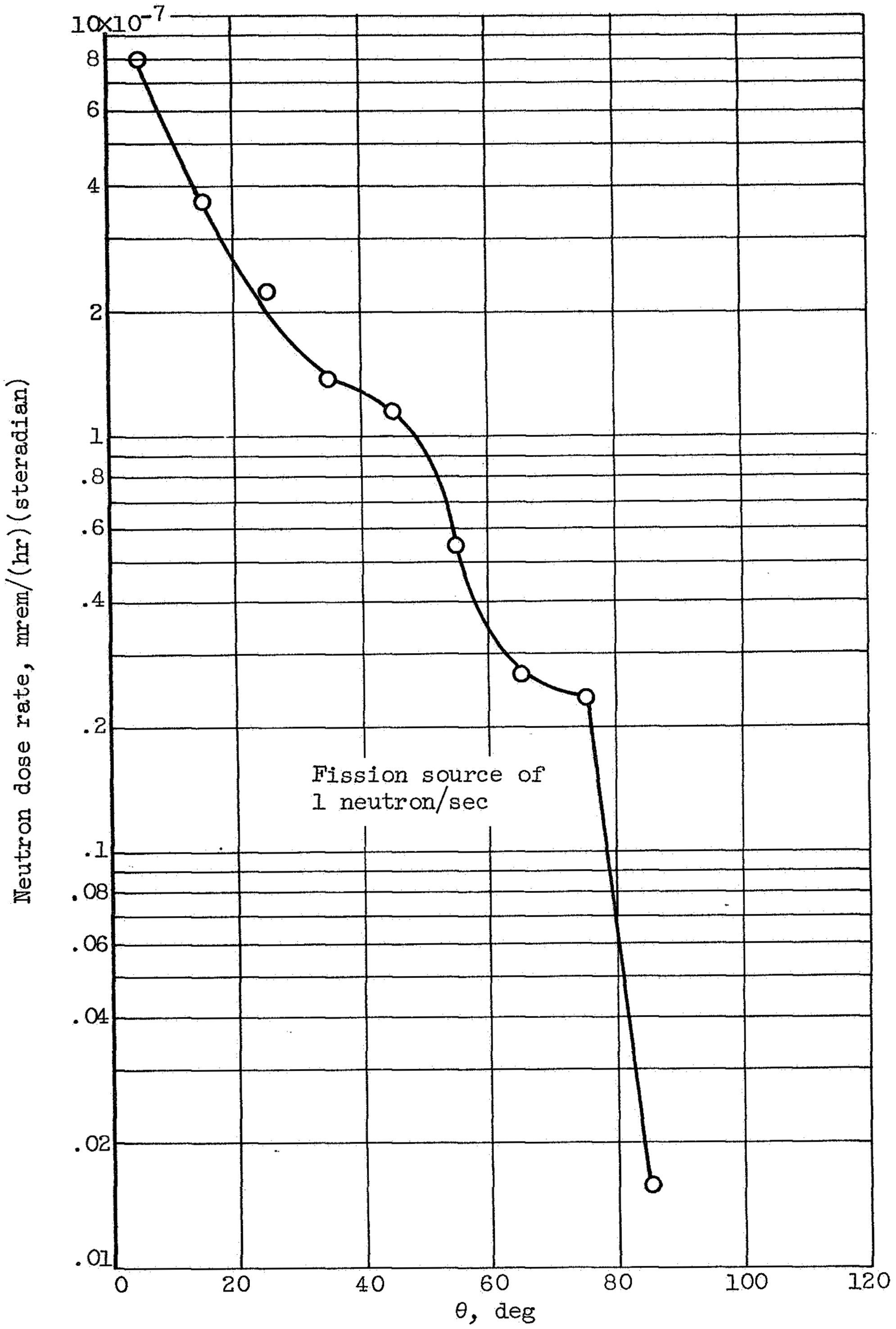


Figure III-11. - Angular distribution of neutron dose rate for 50 centimeters of LiH (calculated by Monte Carlo technique, ref. 184(SRD)).

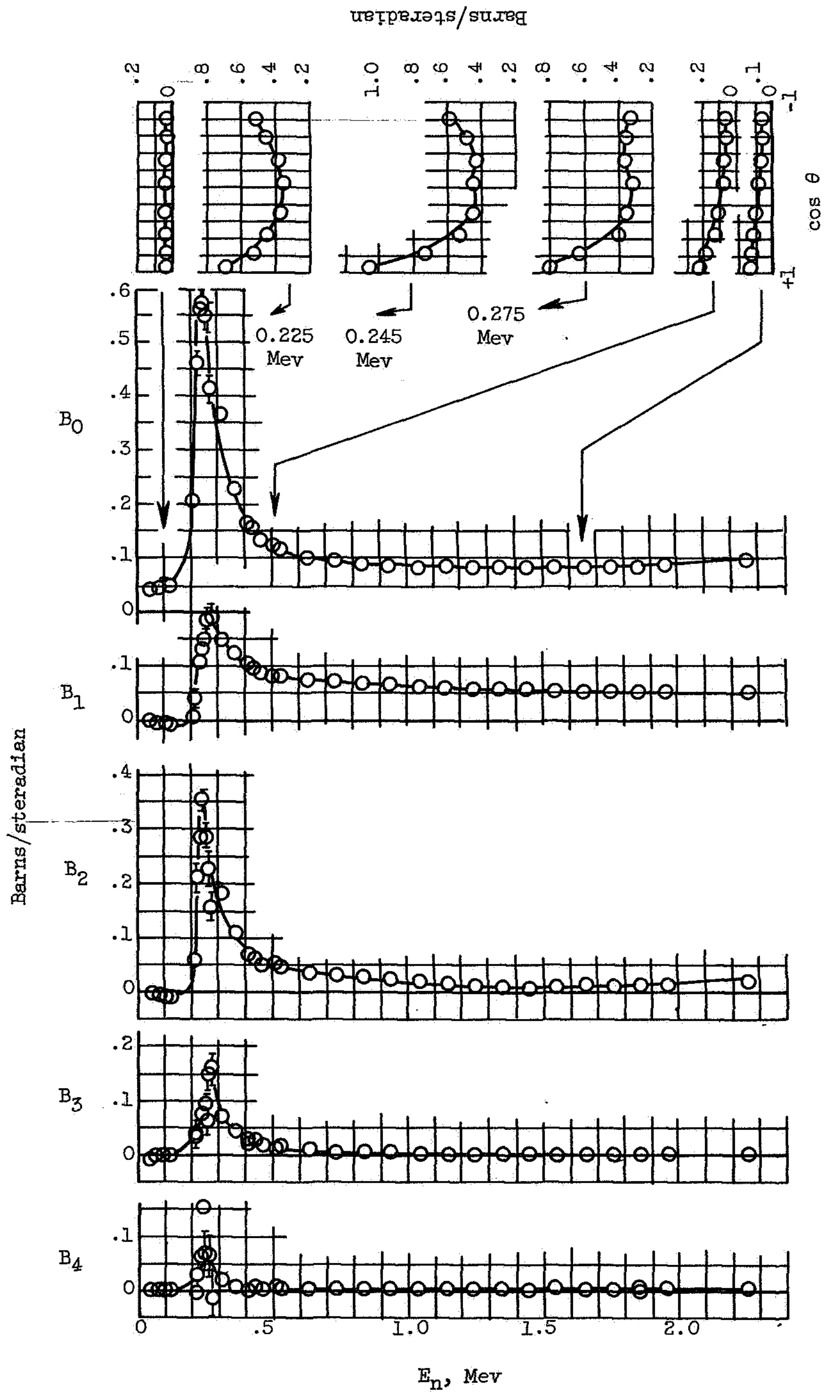


Figure III-12. - Differential cross section for scattering of neutrons by Li^6 (ref. 207(U)). (B_0, etc. are the "significantly nonzero" coefficients in the Legendre series expansion of the form

$$\sigma_s(\mu) = \sum_{l=0}^{N-1} B_l P_l(\mu)$$

where μ is the cosine of the angle of scatter.)

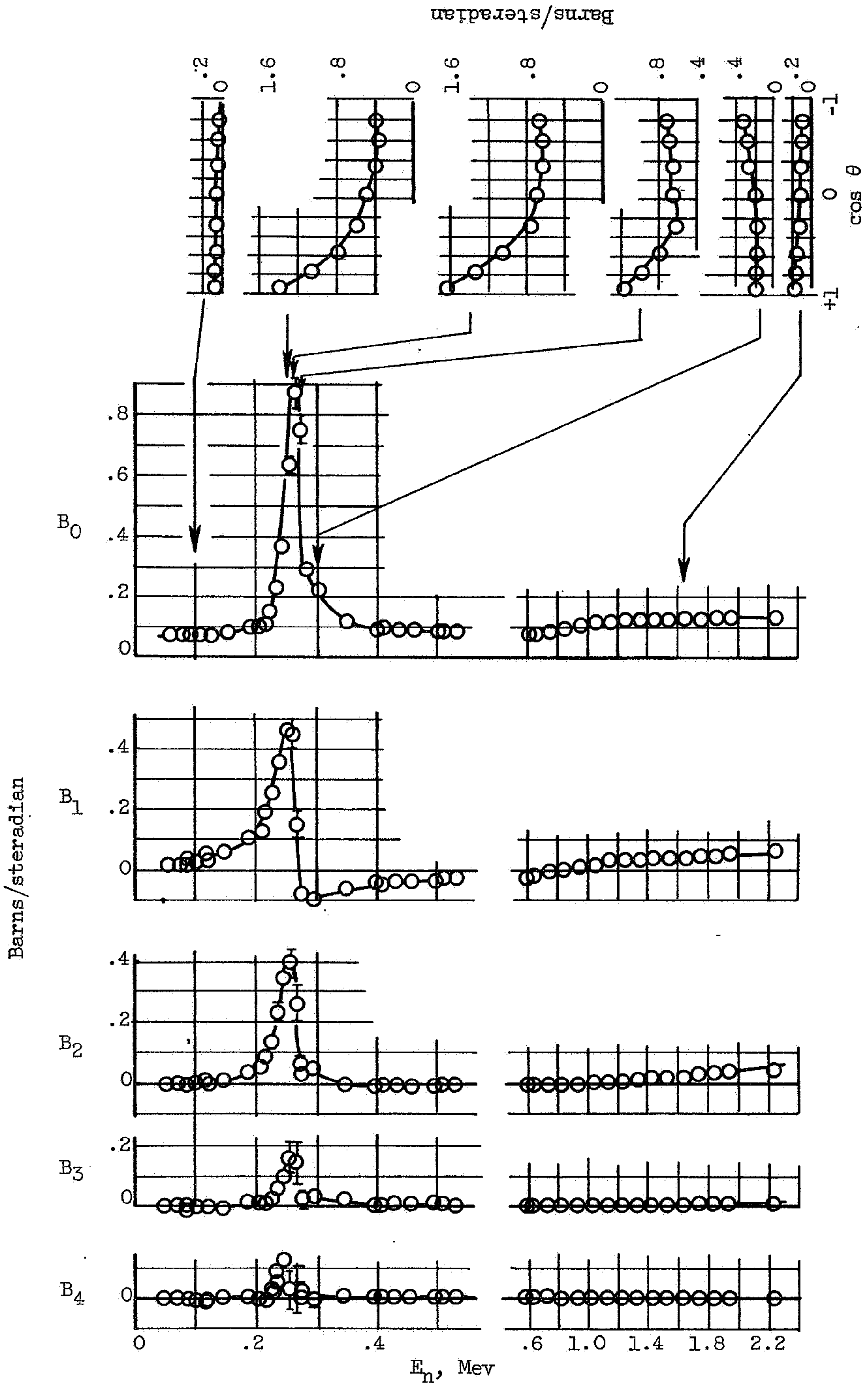


Figure III-13. - Differential cross section for scattering of neutrons by Li^7 (ref. 207(U)). (See note, fig. III-12.)

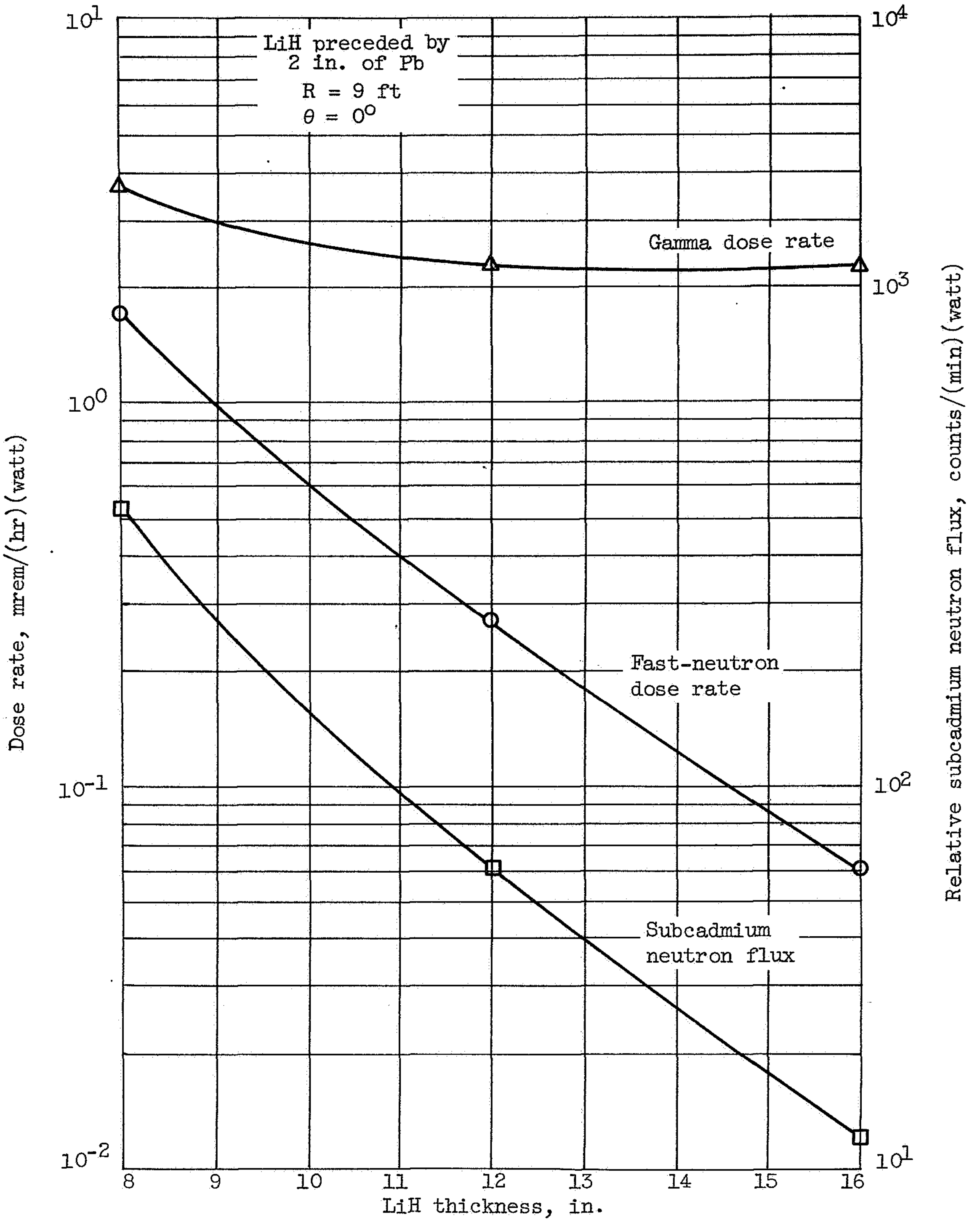


Figure III-14. - Radiation leakage plotted against thickness of LiH (ref. 183(CRD)).

IV. <u>THERMODYNAMIC PROPERTIES</u>

IV. THERMODYNAMIC PROPERTIES

A. HEAT CAPACITY

Figure IV-1 presents the best heat capacity data the authors could find. Some of the data shown in this figure is also presented in table IV-I.

Lang's data were obtained using a drop calorimeter and measuring the enthalpy. By differentiation of the equation for enthalpy, which fits Lang's data from 80° to 1050° F, the following equation for specific heat is obtained (ref. 133(U)):

$$c_p = 0.8475 + 0.7978 \times 10^{-3}\ T$$

where c_p is in Btu/(lb)(°F) and T is in °F.

> "Lonsdale [ref. 208(U)] has calculated the root-mean-square amplitude of vibration of the atoms in solid lithium hydride at 20° C. from the X-ray scattering data and the Debye θ [of 815° K]. The value for lithium hydride is 0.24 A . . . ." (Ref. 2d(U))

Reference 93(U) gives a Debye characteristic temperature for LiD of 611° K.

B. ENTROPY

Table IV-II gives the calculated values of entropy of the dissociated gas from 300° to 5000° K at pressures from 10^{-6} to 1000 atmospheres.

Table IV-I gives the calculated values of entropy from 0° to 6000° K. Note that the values are for the ideal gas at atmospheric pressure.

Figure IV-2 is a plot of calculated specific enthalpy against calculated specific entropy for dissociated lithium hydride, with cross plots of temperature, pressure, and molecular weight.

Messer (ref. 2i(U)) gives 5.9±0.5 cal/(mole)(deg) (at 298.1° K) and 1 atmosphere for the solid, and 40.8±0.2 cal/(mole)(deg) for the gas as calculated by Kelley from the limited low-temperature heat capacity data available such as that from Guenther. The Foote Mineral Co. (ref. 174(U)) gives entropy values for the solid as shown in the following table. These data were originated by Greenlee and Johnston of Ohio State University in 1940.

Temperature, °K	Entropy, cal/(mole)(°C)
50	0.06
100	.50
150	1.48
200	2.79
250	4.21
273.1	4.89
298.1	5.63

C. FREE ENERGY FUNCTION

Table IV-III gives the free energy function of the ideal gas from 0° to 6000° K at a pressure of 1 atmosphere.

D. ENTHALPY

Tables IV-I to IV-V and figures IV-2 and IV-3 are self-explanatory, except for the following: The data of figure IV-3 and table IV-IV (from different references) were obtained with a copper-block drop calorimeter. These data have been compared with each other and with those of Fieldhouse, Hedge, and Lang (table IV-V), and it was found that they are almost the same.

The enthalpy of Fieldhouse, Hedge, and Lang (ref. 133(U)) is represented by the following formula from 80° to 1050° F:

$$H_T - H_{80} = -59.17 + 0.8475\ T + 0.3989 \times 10^{-3}\ T^2$$

where H is in Btu per pound and T is in °F. At higher temperatures, experimental values deviate positively from this formula.

See also table IV-III(b) for the theoretical calculated enthalpy of ideal lithium hydride gas at 298.15° K.

Reference 180(U) reports the enthalpy for several mixtures of lithium hydride and hydrogen. The enthalpy is described by the following equation:

$$H_T - H_{30^\circ C} = A + BT \text{ cal/g, where } T \text{ is in } ^\circ C$$

The following table (ref. 180(U)) gives the values of the constants A and B for the different nominal mole percentages of LiH:

LiH, mole percent	Solid		Liquid	
	A	B	A	B
100	-484	2.087	398	1.762
88.9	-400	1.977	327	1.792
80.1	-639	2.352	446	1.542
69.9	-518	2.173	282	2.173

The numbers obtained for the 100 mole percent LiH follow very closely the curve in figure IV-3.

E. HEAT OF FORMATION AND HEAT OF HYDROLYSIS

Table III-III gives the heat of formation of the ideal gas at atmospheric pressure from 0° to 6000° K. The following table gives the values as reported in the references indicated:

Material	Heat of formation, (solid and liquid) kcal/mole	Heat of hydrolysis, kcal/mole	Temperature, $^{\circ}$K	Comments	Reference
LiH	-21.6	-31.6	298	(a)	209(U)
	-21.6	-31.1	298	(a)	88(U)
	-21.34±0.15	-31.76±0.10	298	(b)	155(U)
	-21.666±0.026	-31.476±0.018	298	(b)	210(U)
	-22.9	-------------	298.1	(c)	123(U)
	+33.62 (gas)	-------------	298.15	(d)	See (d) below
LiD	-23.6	-------------	298.1	(c)	123(U)
	-21.784-0.021	-31.32±0.007	298	(b)	210(U)

[a] Measured by means of the difference between the heats of hydrolysis of the hydride and of lithium metal, both measured in the same calorimeter with the same technique. The calorimeter was an open Dewar flask, the hydrogen gas being allowed to escape.

[b] Same as in (a), except that a closed bomb calorimeter was used, the hydrogen evolved thus being confined.

[c] Based on experimental data, method unknown.

[d] For lithium hydride gas, calculated by Bonnie McBride of NASA's Lewis Research Center. This was done using the dissociation energy of LiH from Velasco (ref. 125(U)), the dissociation energy of H_2 from Herzberg and Monfils (ref. 211(U)), the heat of sublimation of Li from Evans, et al. (ref. 212(U)), and the thermal properties from reference 213(U) (see table IV-III(b)). If the latest NASA thermal properties are used instead of those from reference 213(U), a value of 33.635 kcal/mole is obtained.

F. FREE ENERGY OF FORMATION

The free energy of formation at 298° K is about -17 kcal/mole for the crystalline form and +25.2 kcal/mole for the gaseous form. Comments on the data from each reference are as follows:

Reference 174(U) gives the value at 298° K as -16.45±0.3 kcal/mole for the crystalline form, and +25.2 kcal/mole for the gaseous form.

Reference 214(U) gives $\Delta F^o_{298.1}$ = -17.9 kcal/mole for the equation $Li(s) + \frac{1}{2} H_2(g) = LiH(s)$, the above value calculated using the following:

$$S^o_{298.1} = 6.70 \text{ cal/(mole)(deg) for Li(s)}$$

$$S^o_{298.1} = 15.60 \text{ cal/(mole)(deg) for } \frac{1}{2} H_2(g)$$

$$S^o_{298.1} = 5.63 \text{ cal/(mole)(deg) for LiH(s)}$$

Reference 117(U) gives $\Delta F^o_{298.1}$ = +25.2 kcal/mole for the ideal gas.

> "Kelley [ref. 215(U)] has calculated the entropy of lithium hydride at 298°K, from Guenther's [ref. 216(U)] heat capacity data. The free energy of formation [of the solid] may then be calculated, using this figure and the heat of formation of Gunn and Green [ref. 210(U)]. The resulting values are:
>
> $$S^o_{298^oK.} = 5.9 \pm 0.5 \text{ cal/mole-deg.}$$
>
> $$\Delta F^o_{f\ 298^oK.} = -16{,}780 \pm 300 \text{ cal/mole."}$$
>
> (Ref. 2i(U))

Table IV-III gives the free energy of formation of the ideal gas at temperatures from 0° to 6000° K at a pressure of 1 atmosphere.

G. EQUILIBRIUM CONSTANT OF FORMATION

Table IV-III gives the equilibrium constants of formation for the ideal gas at atmospheric pressure from 0° to 6000° K.

Klemperer (ref. 121(U)) has deduced from the intensity of the infrared emission spectrum that a considerable pressure of LiH must exist at 1400° K - of the order of 10 millimeters of mercury. This would give $K = 0.005$ atmosphere for the equilibrium constant of the reaction:

$$2\ \mathrm{Li(g)} + \mathrm{H_2(g)} = 2\ \mathrm{LiH(g)}$$

See also section I.A.10. for the equilibrium constants of various isotope exchange reactions.

H. ZERO-POINT ENERGY

THEORETICAL ZERO-POINT ENERGY OF LITHIUM HYDRIDE AND LITHIUM DEUTERIDE

(Ref. 2n(U))

Material	Zero-point energy, kcal/mole	Author of theory	Original reference
LiH	1.820	Debye	93(U)
	1.12	London	217(U)
	5.49	Born	217(U)
LiD	1.368	Debye	93(U)
	.59	London	217(U)
	4.30	Born	217(U)

[redacted]

[redacted]

I. HEAT OF DISSOCIATION (see also section IV.P. MOLECULAR PROPERTIES)

Reference 117(U) quotes from Velasco, giving $\Delta H_0 = 56.01 \pm 10$ kcal/mole. Reference 174(U) gives 59 kcal/mole for dissociation energy of lithium hydride. Reference 152(U) gives $\Delta H_0 = 2.56$ ev/mole.

J. HEAT OF FUSION

cal/mole	cal/g	Btu/lb
4700±600[a]	591±75.5	1062±136.0
4900±700[b]	617±88.1	1110±158.5
5510	694.4[c]	1249
5240±40	659±5[d]	1186±9

[a] Ref. 117(U) gives the value as a penciled correction on a publication received at NASA, Aug. 24, 1960.

[b] Ref. 21(U) states: "Messer [ref. 218(U)] has calculated a cryogenic value from the lowering of the freezing point of LiH by the solutes lithium oxide and calcium hydride, which show identical behavior within experimental error up to 9 mole % solute."

[c] Ref. 85(U) - Waldrop gave the heat of fusion with the qualification that Inconel containers were used and the lithium hydride was about 98 percent pure.

[d] Ref. 180(U) - Also given are the heats of fusion for 88.9 mole % LiH, 600±5 cal/g; for 80.1 mole % LiH, 528±3 cal/g; and for 69.9 mole % LiH, 461±5 cal/g.

[redacted]

[redacted]

K. SUBLIMATION EFFECTS

1. Latent Heat of Sublimation

Two numbers were found: 51.6 kcal/mole (ref. 2i(U)) and 52.0 kcal/mole (refs. 174(U) and 219(U)) at 298° K.

The value of 51.6 kcal/mole was calculated from thermochemical and spectroscopic data (ref. 220(U)).

Messer also mentions that

> "the heat of sublimation should not be very different at high temperatures, but the free energy of sublimation will undoubtedly be less positive at higher temperatures, corresponding to increasing relative stability of LiH (g). It is improbable, however, that the actual concentration of LiH gas will be significant at temperatures at which solid LiH is stable at 1 atm. pressure. This concentration should be susceptible to calculation by statistical mechanical methods from the spectral data." (Ref. 2p(U))

2. Free Energy of Sublimation

A value of 41.7 kcal/mole is given by references 174(U) and 2p(U). See also Messer's note in the preceding section.

L. ELECTRONIC ENERGY

This characteristic seems to be called by several other names including lattice, cohesive, and binding energy. The value that seems to be most accepted at the present time is -218 kcal/mole. The values reported range from -217.04 to -219.2 kcal/mole, and from 1.16 to 3.68 ev. The references that report these values are 122, 129, 131, 145, 149, 150, 154 to 161, and 171 to 173, all unclassified.

The Born repulsion coefficient n has been calculated and is reported in references 109(U), 110(U), and 221(U). The values of n were reported as 4.1 (ref. 109(U) and 4.4 (ref. 110(U)). Reference 221(U) shows that the form Be^{-gr}, with $g = 2.12\ A^{-1}$ for LiH, was superior to the Born expression as a means of representing repulsion energies in ionic crystals. Here, B is the repulsion energy coefficient; e is the base of natural logarithms; g is the repulsion constant; r is the anion-cation distance A; and Be^{-gr} is the repulsive energy term in an equation for the energy of formation. See reference 106b(U) for a description of the Born-Haber cycle.

M. LITHIUM - LITHIUM HYDRIDE SOLID-LIQUID EQUILIBRIUM

Messer, et al., report their results as follows:

> "The entire freezing point-composition curve is shown in Fig. IV-4, ... The measurements made by removing hydrogen agree closely with those made on adding hydrogen.
>
> "The freezing point of near-stoichiometric lithium hydride is found to be $688 \pm 1^{\circ}$. The removal of hydrogen lowers the freezing point to a monotectic at 685°, the temperature remaining constant at this value for all mixtures between about 26 and 98 mole % lithium hydride. Further removal of hydrogen lowers the freezing point again, down to 624° at 13 mole % lithium hydride, the lowest hydrogen content at which there was a detectable break in the freezing curves.
>
> . . . .
>
> "The curve below the lower plateau limit of 26 mole % LiH may be considered as the curve of solubility of lithium hydride in lithium as a function of temperature. It may be represented with an average deviation of ± 0.8% in composition by the equation
>
> $$\log n/(1 + n) = -3381/T + 2.835$$
>
> This corresponds to $\Delta H = + 15.74$ kcal./mole for the heat of solution of lithium hydride in liquid lithium saturated with hydrogen at the plateau dissociation pressure of the lithium-hydrogen system.
>
> "The equilibrium dissociation pressure was checked at 700° at 37.0 and 64.8 mole % lithium hydride on [one] sample . . . . One hour was sufficient to establish equilibrium. The values found were 28.8 and 27.8 mm., respectively, agreeing well with Heumann and Salmon's plateau pressure of 28 ± 1 mm." (Ref. 177(U))

Other information on the solid-liquid equilibrium of lithium and lithium hydride is reported by Messer as follows:

> "The full phase diagram of the lithium-lithium hydride system, incorporating the data and hypotheses discussed in this . . . section, is shown in Figure [IV-5]. The α phase is a solution of hydrogen in lithium; the β phase is a hydrogen-deficient lithium hydride. The solubility of hydrogen in lithium at the lower temperatures, and the extent of freezing point depression of lithium metal by dissolved hydrogen (both not determined experimentally) are exaggerated for the sake of clarity." (Ref. 21(U))

[redacted]

N. SOLID-LIQUID EQUILIBRIUM IN SYSTEMS OF LITHIUM HYDRIDE WITH LITHIUM OXIDE, CALCIUM HYDRIDE, TITANIUM HYDRIDE, AND ZIRCONIUM HYDRIDE

These systems are reported in reference 222(U). For Li_2O, a eutectic was found at 654.3°±0.3° C and 9.3 mole percent Li_2O. The system with 18.3 mole percent CaH_2 gave a eutectic at 614.0°±0.3° C.

The following table gives the effect of titanium and zirconium hydrides on the liquidus temperature of lithium hydride (ref. 222(U)):

Added substance	Liquidus difference, °C	8.0±0.3 Mole % MH_n at 688° C			
		Pressure range, atm	Composition range, $\frac{\text{atoms H}}{\text{atom M}}$	Phase(s) predicted M-H system	
				From composition	From pressure
$TiH_{1.97}$	+0.5±0.5	0.77-0.81	0.89-0.94	β	β
$TiH_{1.97}$	+2.4±0.5	0.75-1.04	1.0-1.6	β + γ, γ	β
$TiH_{1.97}$	+1.5±1.5[a]	0.30-0.35	1.5-1.7	γ	β
$TiH_{1.96}$	+0.1±0.3	10.0±0.2	1.75[b]	γ	γ
$ZrH_{1.99}$	+1.0±1.1	0.84-0.90	1.80-1.90	δ	δ

[a] Difference was increased above experimental value by 1°±1° C, because at hydrogen pressures as low as 0.3 atm the melting point of LiH could be lowered by this amount because of loss of hydrogen (ref. 177(U)).

[b] Predicted from reference 223(U) at 688° and 10 atm.

[redacted]

O. DISSOCIATION AND ITS EFFECTS

Messer covers dissociation pressure as follows:

> "The equilibrium hydrogen pressure over lithium hydride, pure and partially decomposed, varies with both temperature and composition in the manner characteristic of most metal-hydrogen systems. The variation with temperature at constant composition of the phase (s) present normally follows the van't Hoff isochore, as represented by the approximately linear plot of the logarithm of the 'Plateau' pressure vs. the reciprocal of the absolute temperature shown in Figure [IV-6]. The variation of dissociation pressure with composition at constant temperature is shown in Figure [IV-7].
>
> "The explanation of the shapes of the isotherms in Figure [IV-7] is as follows: The dissociation pressure of the hypothetical completely pure stoichiometric lithium hydride is very high--theoretically the limiting value could be infinity. The removal of small amounts of hydrogen produces a large decrease in pressure, with the formation of hydrogen vacancies in the lithium hydride lattice, which remains as a single phase. When enough hydrogen has been removed, the lattice becomes saturated with hydrogen vacancies. Further withdrawal produces a constant pressure, independent of composition, and a second phase, a saturated solution of hydrogen in lithium, begins to separate out. The constant pressure in this two-phase region of varying composition is the so-called 'plateau' pressure. When enough further hydrogen has been withdrawn, the hydride phase disappears, and further hydrogen loss produces a decrease in pressure due to loss of hydrogen from solution in the remaining single phase. The pressure finally diminishes to zero at zero hydrogen content (pure lithium).
>
> "Some of the earlier measurements of the equilibrium dissociation pressure of lithium hydride gave variable and inconsistent results because the variation of pressure with composition was not realized or not controlled by the workers making the measurements. Hence, the compositions for which the reported pressures were valid are unknown. The dissociation pressure measurements of Ephraim and Michel [ref. 71(U)], Tronstad and Wergeland [ref. 224(U)], and Peters [ref. 24(U)] all gave results higher than the thermodynamically estimated pressures, which are essentially the plateau pressures. This, of course, was because nearly stoichiometric lithium hydride has a pressure much higher than the plateau pressure, and a pressure highly sensitive to small changes in composition.
>
> "The dissociation pressure of the lithium hydride-lithium system as a function of temperature and composition

has been investigated more carefully by Hill [ref. 225(U)], Perlow [ref. 14(U)], Gibb [ref. 226(U)], and, most recently and carefully, by Heumann and Salmon [ref. 227(U)]. The isotherms of Hill, Perlow, and Heumann and Salmon are shown in Figure [IV-7]. . . . The sample of Hill was 99.7% LiH by hydrogen evolution analysis; the sample of Perlow was 96.5% . . .. These have been corrected to 100% purity by assuming the impurities to be inert, even though it is recognized that '100%' represents maximum hydrogen absorption under the circumstances rather than completely pure LiH.

"The curve of Hill is seen to be higher than that of Neumann [sic] and Salmon at 700°. It is also seen in Figure [IV-7] that the dissociation pressures of Hill and Perlow at the three temperatures seem to approach each other at a hydrogen content definitely above zero. Hence, these isotherms are less reliable at the lower hydrogen contents.

"The plateau pressure of Heumann and Salmon of 28 mm. at 700°C. was confirmed by Messer et al [ref. 177(U)].

. . . .

"The Plateau pressures at a series of temperatures have been measured by Hurd and Moore [ref. 228(U)], Hill [ref. 225(U)], Gibb [ref. 226(U)], Heumann and Salmon [ref. 227(U)], and Welch [ref. 111(U)]. The results of these measurements, and the best composite straight line for the log p vs. 1/T graph, are shown in Figure [IV-6]. Also, Table [IV-VI] gives the constants of the equations for the best straight lines for each set of measurements, the plateau pressures at certain selected temperatures, the temperatures at which the plateau pressures are calculated to reach 1 atm., and the plateau composition limits at certain temperatures. All values in parentheses are extrapolated beyond the temperature range of the experimental data.

. . . .

"The plateau regions are seen to become shorter with increasing temperature, indicating increasing mutual solubility of hydride and metal. The dissymmetry would indicate that hydrogen is more soluble in lithium metal than metal in molten hydride. The shortening plateaus also indicate the existence of a critical solution temperature above which metal and hydride would mix in all proportions to give a homogeneous melt. Such a point is predicted by Heumann and Salmon's calculations as 997°C." (Ref. 2j,k(U))

Some of the details of the experiments, the results of which are given in the preceding paragraphs by Messer, have been reported by Anderson (ref. 13c(U)).

Reference 174(U) gives these values of dissociation pressure of lithium hydride as reported by different research workers (no refs. given):

509-655° C	$\log p_{mm} = -9600/T + 11.227$
578-728° C	$\log p_{mm} = -10{,}320/T + 12.165$
700-825° C	$\log p_{mm} = -9337/T + 11.131$
900-1000° C	$\log p_{mm} = -8800/T + 10.20$

where T is in °K.

The pertinent data from Heumann and Salmon's work (ref. 227(U)) are contained in the information already presented from reference 2.

> "Measurements on the decomposition pressure of lithium hydride were made, using a quartz system which was designed to maintain a constant hydrogen pressure inside and outside of the system. The results with this system have given a decomposition temperature of 650° to 658°C, which appears to be independent of the hydrogen over-pressure. The experiment was repeated with a pure lithium sample which was converted to the hydride <u>in situ</u> by allowing it to react with hydrogen at elevated temperatures. This sample gave a decomposition temperature of 670° to 676°C, independent of the hydrogen overpressure. A differential thermal analysis study showed a thermal effect occurring in pure lithium hydride at 642°C and another at 670°C. A very low decomposition temperature between 250° and 300°C was obtained when a mixture of 15 mole per cent lithium fluoride - 85 mole per cent lithium hydride was used in the apparatus." (Ref. 229(SRD))

The following table gives the vapor pressure of lithium hydride as measured by the transpiration method and reported by Welch (ref. 111(U)).

Temperature, °C	Vapor pressure, mm Hg
615.0	0.0670
639.7	.265
664.6	.522
671.3	.605
691.3	.662
727.9	1.01
766.0	2.20
787.5	3.58

See figure IV-6 for the dissociation pressures reported by Welch.

The molecular weight from 300° to 5000° K, at pressures from 10^{-6} to 1000 atm is given in table IV-II.

Reference 230(U) gives the theoretical maximum exhaust velocities obtainable with lithium hydride and nitrogen oxidants. The following table gives the values reported:

Oxidant	Velocity, m/sec
NO_3F	5310
$NClO_5$	5050
$NClO_6$	5120
$FClO_4$	5280
O_2	5740
H_2O_2	4660

P. MOLECULAR PROPERTIES

PROPERTIES OF LITHIUM HYDRIDE AND LITHIUM DEUTERIDE GAS MOLECULES

	State	LiH	LiD	Original reference
Equilibrium internuclear distance, A	Ground	1.5953	1.5949	231(U)
	Ground	1.6 (Experimental)		152(U)
	1st excited	2.596	2.586	231(U)
	2nd excited	2.378	2.376	125(U)
Dissociation energy, ev/mole (normal atoms)	Ground	2.5154	2.5160	125(U)
	Ground	2.56		152(U)
	1st excited	1.0765	1.0770	125(U)
	2nd excited	.035	.036	125(U)
Vibration frequency, Li^7 molecule, cm^{-1}		1405.65	1055.12	94(U)
Vibration frequency, Li^6 molecule, cm^{-1}		1420.32	1074.59	94(U)
Vibration frequency, Li^N molecule, cm^{-1}		1380 1359.78		152(U) 232(U)
Moment of inertia, g/cm^2		6×10^{-24}		233(U)

TABLE IV-I. - THERMODYNAMIC FUNCTIONS OF LITHIUM HYDRIDE (FOR 1 MOLE OF THE IDEAL GAS AT 1 ATM, DIMENSIONLESS, Ref. 232(U))

Temperature, °K	Heat capacity, C_p^o/R	Enthalpy per °K, $(H^o - E_0^o)/RT$	Gibbs free energy per °K, $-(F^o - E_0^o)/RT$	Entropy, S^o/R
50	3.50220	3.42843	10.84307	14.27151
60	3.50206	3.44072	11.46930	14.91002
70	3.50207	3.44948	12.00038	15.44987
80	3.50215	3.45606	12.46145	15.91751
90	3.50227	3.46118	12.86882	16.33001
100	3.50243	3.46530	13.23371	16.69902
110	3.50260	3.46868	13.56416	17.03284
120	3.50280	3.47152	13.86610	17.33762
130	3.50303	3.47393	14.14406	17.61800
140	3.50332	3.47602	14.40159	17.87761
150	3.50373	3.47785	14.64147	18.11933
160	3.50431	3.47949	14.86598	18.34548
170	3.50513	3.48097	15.07697	18.55795
180	3.50628	3.48234	15.27598	18.75832
190	3.50785	3.48364	15.46429	18.94794
200	3.50994	3.48490	15.64301	19.12792
210	3.51263	3.48616	15.81307	19.29923
220	3.51599	3.48743	15.97528	19.46272
230	3.52009	3.48876	16.13033	19.61910
240	3.52496	3.49017	16.27884	19.76901
250	3.53065	3.49167	16.42135	19.91302
260	3.53715	3.49329	16.55832	20.05162
270	3.54448	3.49505	16.69019	20.18525
280	3.55262	3.49696	16.81733	20.31429
290	3.56154	3.49903	16.94008	20.43911
300	3.57120	3.50127	17.05874	20.56002
310	3.58156	3.50369	17.17359	20.67728
320	3.59257	3.50629	17.28487	20.79117
330	3.60417	3.50908	17.39280	20.90189
340	3.61631	3.51206	17.49760	21.00967
350	3.62894	3.51522	17.59945	21.11467
360	3.64198	3.51855	17.69853	21.21709
370	3.65539	3.52207	17.79498	21.31706
380	3.66911	3.52576	17.88896	21.41472
390	3.68309	3.52961	17.98059	21.51021
400	3.69728	3.53363	18.07000	21.60363
450	3.76976	3.55582	18.48745	22.04328
500	3.84167	3.58083	18.86337	22.44421
550	3.91012	3.60769	19.20591	22.81360
600	3.97364	3.63558	19.52101	23.15659
650	4.03169	3.66385	19.81313	23.47698
700	4.08426	3.69203	20.08568	23.77772
750	4.13164	3.71979	20.34136	24.06115
800	4.17428	3.74688	20.58229	24.32918
850	4.21266	3.77317	20.81024	24.58342
900	4.24724	3.79857	21.02663	24.82520
950	4.27849	3.82302	21.23267	25.05569
1000	4.30680	3.84651	21.42937	25.27588
1050	4.33253	3.86905	21.61759	25.48664
1100	4.35601	3.89066	21.79808	25.68874
1150	4.37751	3.91137	21.97149	25.88286

TABLE IV-I. - Concluded. THERMODYNAMIC FUNCTIONS OF LITHIUM HYDRIDE (FOR 1 MOLE OF THE IDEAL GAS AT 1 ATM, DIMENSIONLESS, Ref. 232(U))

Temperature, °K	Heat capacity, C_p^o/R	Enthalpy per °K, $(H^o - E_0^o)/RT$	Gibbs free energy per °K, $-(F^o - E_0^o)/RT$	Entropy, S^o/R
1200	4.39728	3.93121	22.13838	26.06959
1300	4.43239	3.96844	22.45453	26.42298
1400	4.46270	4.00269	22.74990	26.75259
1500	4.48925	4.03426	23.02714	27.06141
1600	4.51279	4.06345	23.28845	27.35191
1700	4.53392	4.09051	23.53562	27.62614
1800	4.55309	4.11569	23.77015	27.88584
1900	4.57066	4.13918	23.99331	28.13249
2000	4.58690	4.16116	24.20619	28.36735
2100	4.60204	4.18180	24.40972	28.59152
2200	4.61624	4.20123	24.60471	28.80594
2300	4.62966	4.21956	24.79187	29.01144
2400	4.64240	4.23692	24.97182	29.20874
2500	4.65457	4.25338	25.14512	29.39851
2600	4.66623	4.26904	25.31224	29.58129
2700	4.67746	4.28396	25.47364	29.75761
2800	4.68830	4.29821	25.62970	29.92791
2900	4.69881	4.31184	25.78077	30.09262
3000	4.70902	4.32491	25.92717	30.25209
3200	4.72867	4.34954	26.20709	30.55663
3400	4.74746	4.37240	26.47147	30.84387
3600	4.76554	4.39374	26.72200	31.11575
3800	4.78303	4.41377	26.96010	31.37388
4000	4.80002	4.43266	27.18698	31.61965
4200	4.81657	4.45055	27.40369	31.85424
4400	4.83276	4.46755	27.61113	32.07869
4600	4.84860	4.48378	27.81008	32.29386
4800	4.86415	4.49930	28.00124	32.50055
5000	4.87943	4.51420	28.18521	32.69942

The constants used in calculating this table are as follows:

Fundamental vibrational frequency, ν, 1359.78 wave numbers

First-order anharmonic constant, $\frac{\omega_e x_e}{\nu}$, 0.0171 cm^{-1}

Rotation-vibration interaction constant, δ, 0.0288 (dimensionless)

Rotational constant, B_0, 7.4067 wave numbers

Stretching constant, $\rho \times 10^5$, 1.08 $°K^{-1}$

Zero-point vibrational energy, $G(0)$, 697.05 cm^{-1}

TABLE IV-II. - COMPUTED VALUES OF SPECIFIC ENTROPY, ENTHALPY, AND MOLECULAR WEIGHT FOR DISSOCIATED LITHIUM HYDRIDE AT VARIOUS TEMPERATURES AND PRESSURES (Ref. 234(U))

Pressure, P, atm	Entropy, s, cal/(deg)(g)	Enthalpy, H, cal/g	Molecular weight, M, g/mole	Pressure, P, atm	Entropy, s, cal/(deg)(g)	Enthalpy, H, cal/g	Molecular weight, M, g/mole
Temperature, T, 300° K				Temperature, T, 1500° K			
1.0×10^{-6}	8.7477	0.34476×10^{4}	7.9479	1.0×10^{-6}	13.358	0.64991×10^{4}	5.2731
1.0×10^{-5}	8.1720	$.34476\times10^{4}$	7.9479	1.0×10^{-5}	12.444	$.64268\times10^{4}$	5.2936
1.0×10^{-4}	7.5962	$.34476\times10^{4}$	7.9479	1.0×10^{-4}	11.561	$.63973\times10^{4}$	5.3069
1.0×10^{-3}	7.0205	$.34476\times10^{4}$	7.9479	1.0×10^{-3}	10.681	$.63675\times10^{4}$	5.3324
1.0×10^{-2}	6.4448	$.34476\times10^{4}$	7.9479	1.0×10^{-2}	9.7799	$.62948\times10^{4}$	5.4077
1.0×10^{-1}	5.8692	$.34476\times10^{4}$	7.9479	1.0×10^{-1}	8.8108	$.60887\times10^{4}$	5.6379
1.0	5.2934	$.34476\times10^{4}$	7.9479	1.0	7.7276	$.56288\times10^{4}$	6.2300
1.0×10	4.7178	$.34476\times10^{4}$	7.9480	1.0×10	6.6926	$.51092\times10^{4}$	7.0680
1.0×10^{2}	4.1420	$.34476\times10^{4}$	7.9480	1.0×10^{2}	5.8875	$.48327\times10^{4}$	7.6126
1.0×10^{3}	3.5663	$.34476\times10^{4}$	7.9480	1.0×10^{3}	5.2285	$.47308\times10^{4}$	7.8351
Temperature, T, 500° K				Temperature, T, 2000° K			
1.0×10^{-6}	9.6121	0.38138×10^{4}	7.5541	1.0×10^{-6}	16.499	0.12204×10^{5}	4.2189
1.0×10^{-5}	8.7837	$.36965\times10^{4}$	7.8138	1.0×10^{-5}	14.126	$.95137\times10^{4}$	4.7118
1.0×10^{-4}	8.1238	$.36574\times10^{4}$	7.9045	1.0×10^{-4}	12.374	$.78715\times10^{4}$	5.0752
1.0×10^{-3}	7.5211	$.36448\times10^{4}$	7.9341	1.0×10^{-3}	11.177	$.72486\times10^{4}$	5.2330
1.0×10^{-2}	6.9368	$.36408\times10^{4}$	7.9436	1.0×10^{-2}	10.195	$.70217\times10^{4}$	5.3086
1.0×10^{-1}	6.3584	$.36396\times10^{4}$	7.9466	1.0×10^{-1}	9.2719	$.68847\times10^{4}$	5.4007
1.0	5.7818	$.36392\times10^{4}$	7.9475	1.0	8.3253	$.66559\times10^{4}$	5.6374
1.0×10	5.2058	$.36391\times10^{4}$	7.9478	1.0×10	7.3138	$.61858\times10^{4}$	6.2320
1.0×10^{2}	4.6301	$.36390\times10^{4}$	7.9479	1.0×10^{2}	6.3631	$.56613\times10^{4}$	7.0693
1.0×10^{3}	4.0543	$.36390\times10^{4}$	7.9479	1.0×10^{3}	5.6031	$.53826\times10^{4}$	7.6131
Temperature, T, 1000° K				Temperature, T, 2500° K			
1.0×10^{-6}	12.835	0.58403×10^{4}	5.3093	1.0×10^{-6}	17.910	0.15293×10^{5}	3.8748
1.0×10^{-5}	11.952	$.58181\times10^{4}$	5.3325	1.0×10^{-5}	16.461	$.14589\times10^{5}$	3.9564
1.0×10^{-4}	11.030	$.57494\times10^{4}$	5.4056	1.0×10^{-4}	14.939	$.13636\times10^{5}$	4.0920
1.0×10^{-3}	9.9980	$.55489\times10^{4}$	5.6309	1.0×10^{-3}	12.858	$.11109\times10^{5}$	4.5192
1.0×10^{-2}	8.7680	$.50970\times10^{4}$	6.2150	1.0×10^{-2}	11.014	$.89025\times10^{4}$	4.9802
1.0×10^{-1}	7.5598	$.45789\times10^{4}$	7.0540	1.0×10^{-1}	9.7368	$.79416\times10^{4}$	5.2350
1.0	6.6597	$.43005\times10^{4}$	7.6060	1.0	8.7074	$.75148\times10^{4}$	5.4319
1.0×10	5.9653	$.41975\times10^{4}$	7.8327	1.0×10	7.7268	$.71092\times10^{4}$	5.8076
1.0×10^{2}	5.3505	$.41634\times10^{4}$	7.9108	1.0×10^{2}	6.7534	$.65359\times10^{4}$	6.5638
1.0×10^{3}	4.7622	$.41525\times10^{4}$	7.9361	1.0×10^{3}	5.9169	$.60867\times10^{4}$	7.3320

TABLE IV-II. - Concluded. COMPUTED VALUES OF SPECIFIC ENTROPY, ENTHALPY, AND MOLECULAR WEIGHT FOR DISSOCIATED LITHIUM HYDRIDE AT VARIOUS TEMPERATURES AND PRESSURES (Ref. 234(U))

Pressure, P, atm	Entropy, s, cal/(deg)(g)	Enthalpy, H, cal/g	Molecular weight, M, g/mole
Temperature, T, 3000° K			
1.0×10^{-6}	20.572	0.22743×10^{5}	3.2577
1.0×10^{-5}	17.627	$.17847\times10^{5}$	3.6796
1.0×10^{-4}	15.783	$.15935\times10^{5}$	3.8762
1.0×10^{-3}	14.331	$.15075\times10^{5}$	3.9786
1.0×10^{-2}	12.660	$.13428\times10^{5}$	4.2261
1.0×10^{-1}	10.702	$.10625\times10^{5}$	4.7467
1.0	9.2100	$.89075\times10^{4}$	5.1769
1.0×10	8.0815	$.80854\times10^{4}$	5.5531
1.0×10^{2}	7.0743	$.74163\times10^{4}$	6.1941
1.0×10^{3}	6.1856	$.68252\times10^{4}$	7.0520
Temperature, T, 3500° K			
1.0×10^{-6}	23.526	0.32204×10^{5}	2.7147
1.0×10^{-5}	20.398	$.26903\times10^{5}$	3.0221
1.0×10^{-4}	17.122	$.20336\times10^{5}$	3.5154
1.0×10^{-3}	14.995	$.17241\times10^{5}$	3.8089
1.0×10^{-2}	13.462	$.16002\times10^{5}$	3.9461
1.0×10^{-1}	11.887	$.14457\times10^{5}$	4.1687
1.0	10.022	$.11562\times10^{5}$	4.7022
1.0×10	8.5205	$.95187\times10^{4}$	5.2600
1.0×10^{2}	7.3774	$.84024\times10^{4}$	5.8984
1.0×10^{3}	6.4270	$.76083\times10^{4}$	6.7890
Temperature, T, 4000° K			
1.0×10^{-6}	24.097	0.34311×10^{5}	2.6550
1.0×10^{-5}	22.154	$.33391\times10^{5}$	2.7009
1.0×10^{-4}	19.319	$.28577\times10^{5}$	2.9698
1.0×10^{-3}	16.161	$.21642\times10^{5}$	3.4670
1.0×10^{-2}	14.029	$.18134\times10^{5}$	3.7886
1.0×10^{-1}	12.481	$.16668\times10^{5}$	3.9499
1.0	10.879	$.14762\times10^{5}$	4.2340
1.0×10	9.1029	$.11707\times10^{5}$	4.8563
1.0×10^{2}	7.7118	$.96578\times10^{4}$	5.5959
1.0×10^{3}	6.6590	$.84776\times10^{4}$	6.5341
Temperature, T, 4500° K			
1.0×10^{-6}	24.342	0.35346×10^{5}	2.6502
1.0×10^{-5}	22.586	$.35211\times10^{5}$	2.6568
1.0×10^{-4}	20.611	$.34014\times10^{5}$	2.7161
1.0×10^{-3}	17.776	$.28507\times10^{5}$	3.0270
1.0×10^{-2}	14.881	$.21770\times10^{5}$	3.5200
1.0×10^{-1}	12.930	$.18577\times10^{5}$	3.8166
1.0	11.407	$.16994\times10^{5}$	4.0028
1.0×10	9.7290	$.14366\times10^{5}$	4.4437
1.0×10^{2}	8.0988	$.11305\times10^{5}$	5.2512
1.0×10^{3}	6.8955	$.94839\times10^{4}$	6.2697
Temperature, T, 5000° K			
1.0×10^{-6}	24.542	0.36296×10^{5}	2.6496
1.0×10^{-5}	22.810	$.36269\times10^{5}$	2.6509
1.0×10^{-4}	21.034	$.36006\times10^{5}$	2.6635
1.0×10^{-3}	18.920	$.33910\times10^{5}$	2.7683
1.0×10^{-2}	16.003	$.27108\times10^{5}$	3.1736
1.0×10^{-1}	13.495	$.21268\times10^{5}$	3.6303
1.0	11.772	$.18730\times10^{5}$	3.8795
1.0×10	10.226	$.16721\times10^{5}$	4.1598
1.0×10^{2}	8.5216	$.13316\times10^{5}$	4.8871
1.0×10^{3}	7.1441	$.10666\times10^{5}$	5.9853

TABLE IV-III. - THERMODYNAMIC PROPERTIES OF LITHIUM HYDRIDE

GAS AT ATMOSPHERIC PRESSURE (Ref. 213(U))

(a) Calculated values

[See Table IV-III(b) for constants.]

Temperature, T, °K	ΔH_f^o	ΔF_f^o	Log K_p	Temperature, T, °K	ΔH_f^o	ΔF_f^o	Log K_p
	kcal/mole				kcal/mole		
0	32.141	32.141	∞	3100	-6.539	20.233	-1.426
100	32.331	30.408	-66.452	3200	-6.559	21.096	-1.441
200	32.287	28.483	-31.124	3300	-6.584	21.961	-1.454
298	32.100	26.654	-19.537	3400	-6.615	22.827	-1.467
				3500	-6.650	23.693	-1.479
300	32.096	26.619	-19.391				
400	31.851	24.831	-13.566	3600	-6.693	24.561	-1.491
500	30.834	23.182	-10.132	3700	-6.742	25.428	-1.502
				3800	-6.797	26.302	-1.513
600	30.551	21.679	-7.896	3900	-6.860	27.174	-1.523
700	30.303	20.220	-6.313	4000	-6.930	28.046	-1.532
800	30.083	18.795	-5.134				
900	29.876	17.396	-4.224	4100	-7.008	28.922	-1.542
1000	29.682	16.020	-3.501	4200	-7.095	29.799	-1.551
				4300	-7.190	30.678	-1.559
1100	29.496	14.663	-2.913	4400	-7.294	31.561	-1.568
1200	29.316	13.322	-2.426	4500	-7.409	32.444	-1.576
1300	29.141	11.996	-2.017				
1400	28.968	10.684	-1.668	4600	-7.533	33.332	-1.584
1500	28.798	9.385	-1.367	4700	-7.668	34.220	-1.591
				4800	-7.815	35.112	-1.599
1600	28.630	8.096	-1.106	4900	-7.975	36.008	-1.606
1700	-6.585	8.146	-1.047	5000	-8.146	36.908	-1.613
1800	-6.569	9.013	-1.094				
1900	-6.554	9.879	-1.136	5100	-8.332	37.814	-1.620
2000	-6.541	10.743	-1.174	5200	-8.532	38.719	-1.627
				5300	-8.748	39.629	-1.634
2100	-6.530	11.606	-1.208	5400	-8.980	40.543	-1.641
2200	-6.520	12.470	-1.239	5500	-9.230	41.464	-1.648
2300	-6.510	13.332	-1.267				
2400	-6.504	14.195	-1.293	5600	-9.498	42.384	-1.654
2500	-6.501	15.057	-1.316	5700	-9.785	43.316	-1.661
				5800	-10.094	44.250	-1.667
2600	-6.498	15.919	-1.338	5900	-10.424	45.190	-1.674
2700	-6.500	16.781	-1.358	6000	-10.776	46.136	-1.680
2800	-6.503	17.643	-1.377				
2900	-6.512	18.506	-1.395				
3000	-6.523	19.369	-1.411				

TABLE IV-III. - Concluded. THERMODYNAMIC PROPERTIES OF LITHIUM HYDRIDE GAS AT ATMOSPHERIC PRESSURE (Ref. 213(U))

(b) Constants used in the calculations (LiH, ideal gas. Excited states were not considered.)

Molecular weight	7.948
$\Delta H^{o}_{f,298.15}$, kcal/mole	32.1
$S^{o}_{298.15}$, cal/(deg)(mole)	40.825
Ground state configuration	$^{1}\Sigma$
ω_e, vibrational wave number, equilibrium position, cm^{-1}	1405.649
$\omega_e x_e$, cm^{-1} (where x_e is the quadratic vibrational amplitude, equilibrium position)	23.200
B_e, rotational constant, equilibrium position, cm^{-1}	7.5131
α_e, vibrational-rotational constant, cm^{-1}	0.2132
σ, symmetry number	1

ΔH^{o}_{f} 298.15 from J. S. Gordon, Thiokol Chem. Corp., Reaction Motors Div., Denville, N. J., Thermodynamic Data for Combustion Products, Jan. 1960. Other data from G. Herzberg, Spectra of Diatomic Molecules, Second ed., D. Van Nostrand Co., Inc., 1950.

TABLE IV-IV. - ENTHALPY OF LITHIUM HYDRIDE (Ref. 235(U))

Temperature, °C	Enthalpy, $H_T - H_{25}$, cal/g
89.6	63.0
125.4	103.6
189.5	185.3
235.0	247.3
329.0	375.4
372.0	435.0
425.3	495.3
470.5	556.1
491.0	595.7
496.0	608.0
521.3	632.4
562.6	673.7
585.0	740.3
588.3	753.2
617.5	835.6
643.0	863.7
652.0	858.0
662.5	[a]997.2
669.0	938.6
690.0	1110.3
714.5	1592.2
727.6	1620.4
753.5	1669.6

[a] This is the number which appears in ref. 235, although it may be incorrect.

TABLE IV-V. - ENTHALPY VALUES FOR SOLID LITHIUM HYDRIDE

(OBTAINED EXPERIMENTALLY, Ref. 133(U))

[Corrected to a datum of 80° F.]

Temperature			ΔH_c	
°F	°C	°K	Btu/lb	cal/g
284	140	413	212.1	118
397	202.5	475.5	345.0	192
561	294	567	541.2	301
648	342.5	615.5	653.6	363
735	390.5	663.5	779.2	433
807	431	704	886.7	493
895	479	752	1021	567
936	502	775	1080	600
989	532	805	1166	647
1029	555	828	1234	686
1053	578	851	1280	711
1105	597	870	1365	759
1117	603	876	1411	785
1158	625	898	1526	847
1186	642	915	1653	920

The numbers with English units are those from ref. 133(U); the conversion to metric units was made by the authors.

TABLE IV-VI. - PLATEAU PRESSURES AND COMPOSITIONS IN THE LITHIUM - LITHIUM HYDRIDE SYSTEM (Ref. 2k(U))

		Hurd and Moore (a)	Hill (a)	Perlow (a)	Gibb (a)	Heumann and Salmon (a)
Coefficients:[b]	A	9600	10320	9337	8800	[c][8244]
	B	11.227	12.165	11.131	10.20	9.9258
p_{H_2} mm at	600° C	1.7	2.2	-------	-----	(3.2)
	700° C	(22.9)	36.2	(34.2)	-----	28
	800° C	-------	(352)	268	100	180
	900° C	-------	-------	-------	500	(830)
T, °C for p = 1 atm		(879)	(841)	(859)	(929)	(897)
Plateau composition ranges, mole percent LiH	700° C	-------	18-98	-------	-----	26-96
	800° C	-------	-------	45-95	??-94	(35-92)
	900° C	-------	-------	-------	??-88	(47-86)

[a] Values in parentheses are extrapolated beyond the temperature range of the experimental data.

[b] The equation for plateau pressure p is: $\log p = -A/T(°K) + B$.

[c] Ref. 227(U).

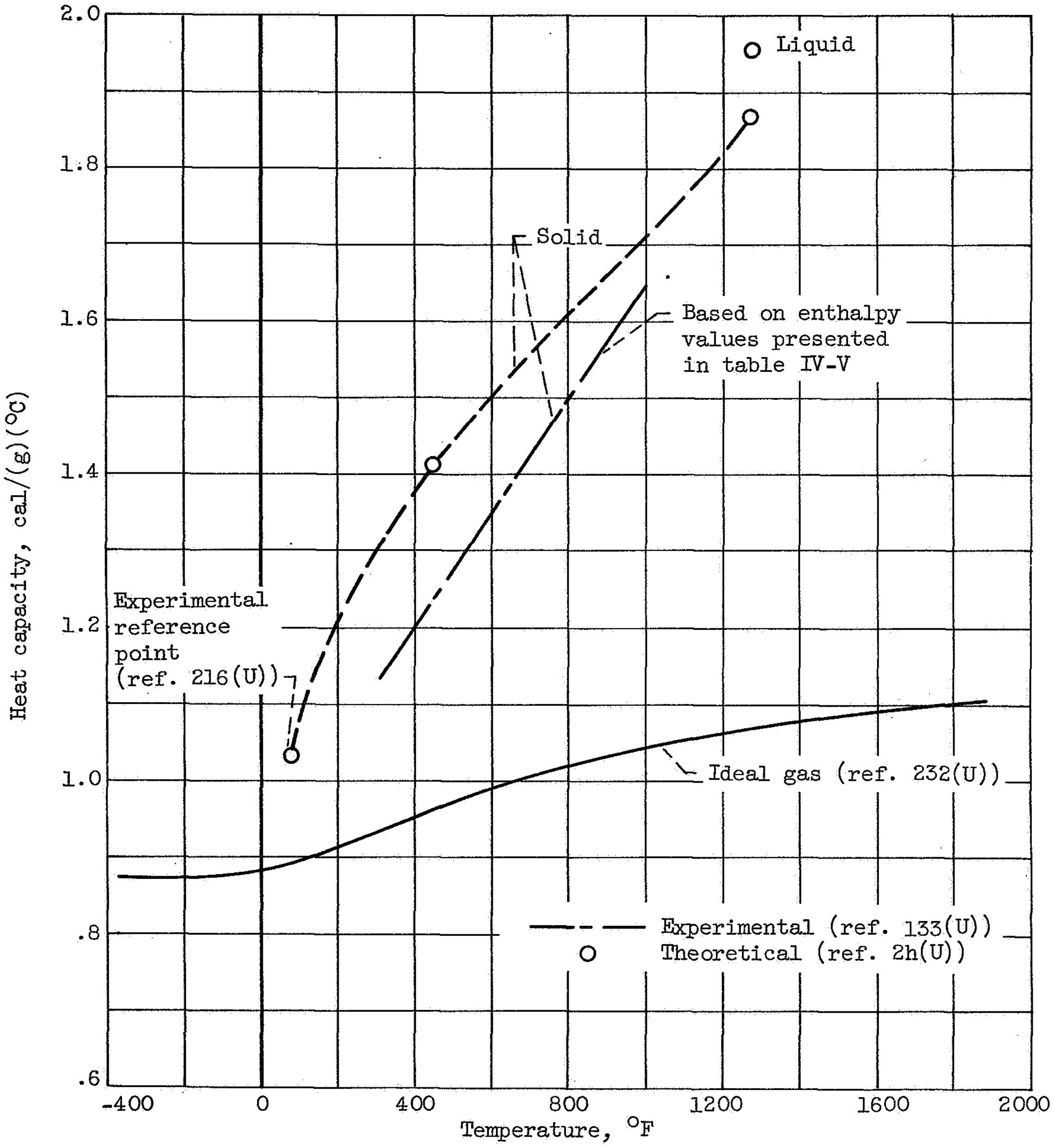


Figure IV-1. - Heat capacity of lithium hydride.

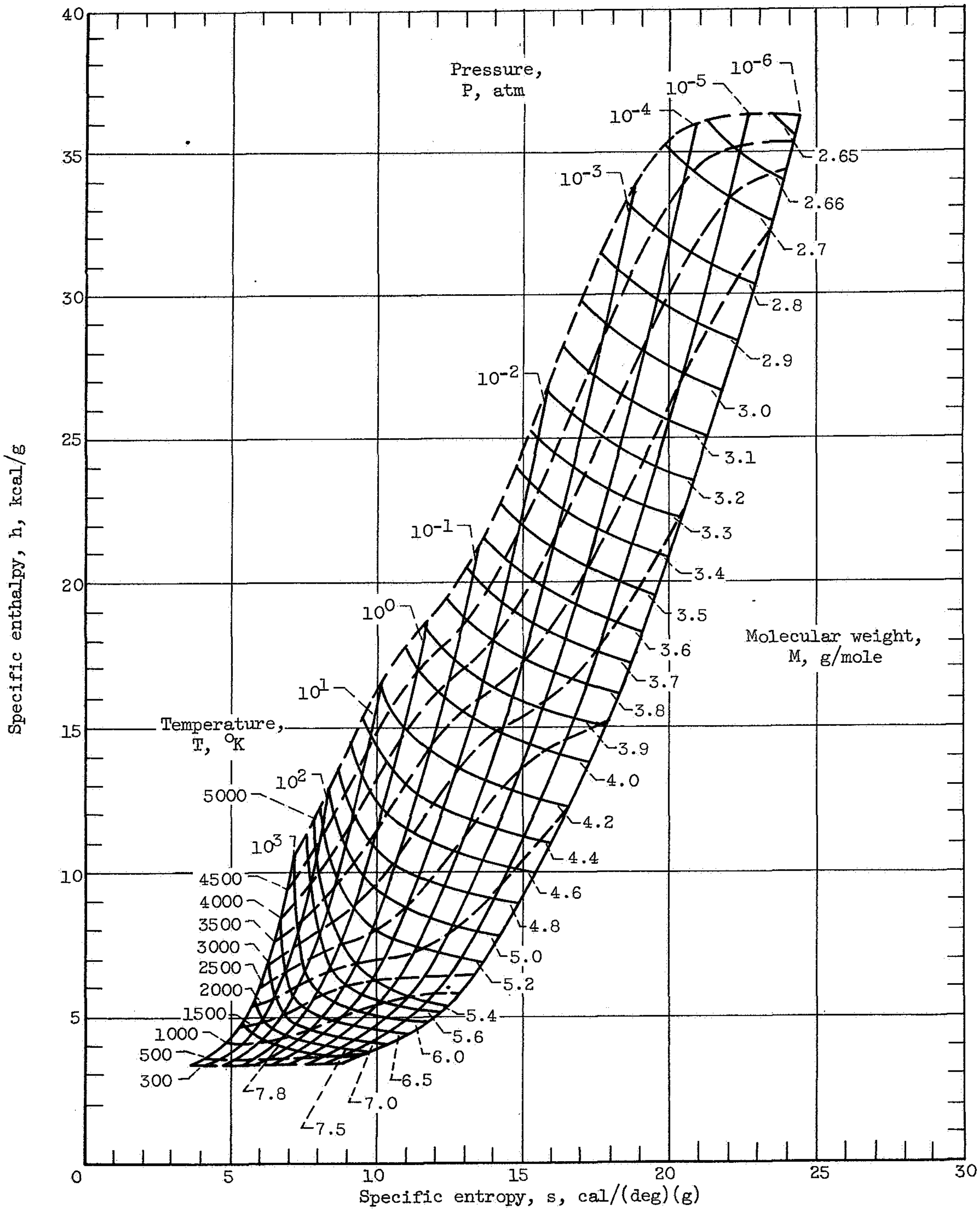


Figure IV-2. - Specific enthalpy plotted against specific entropy for lithium hydride, with cross plots of temperature, pressure, and molecular weight (ref. 234(U)).

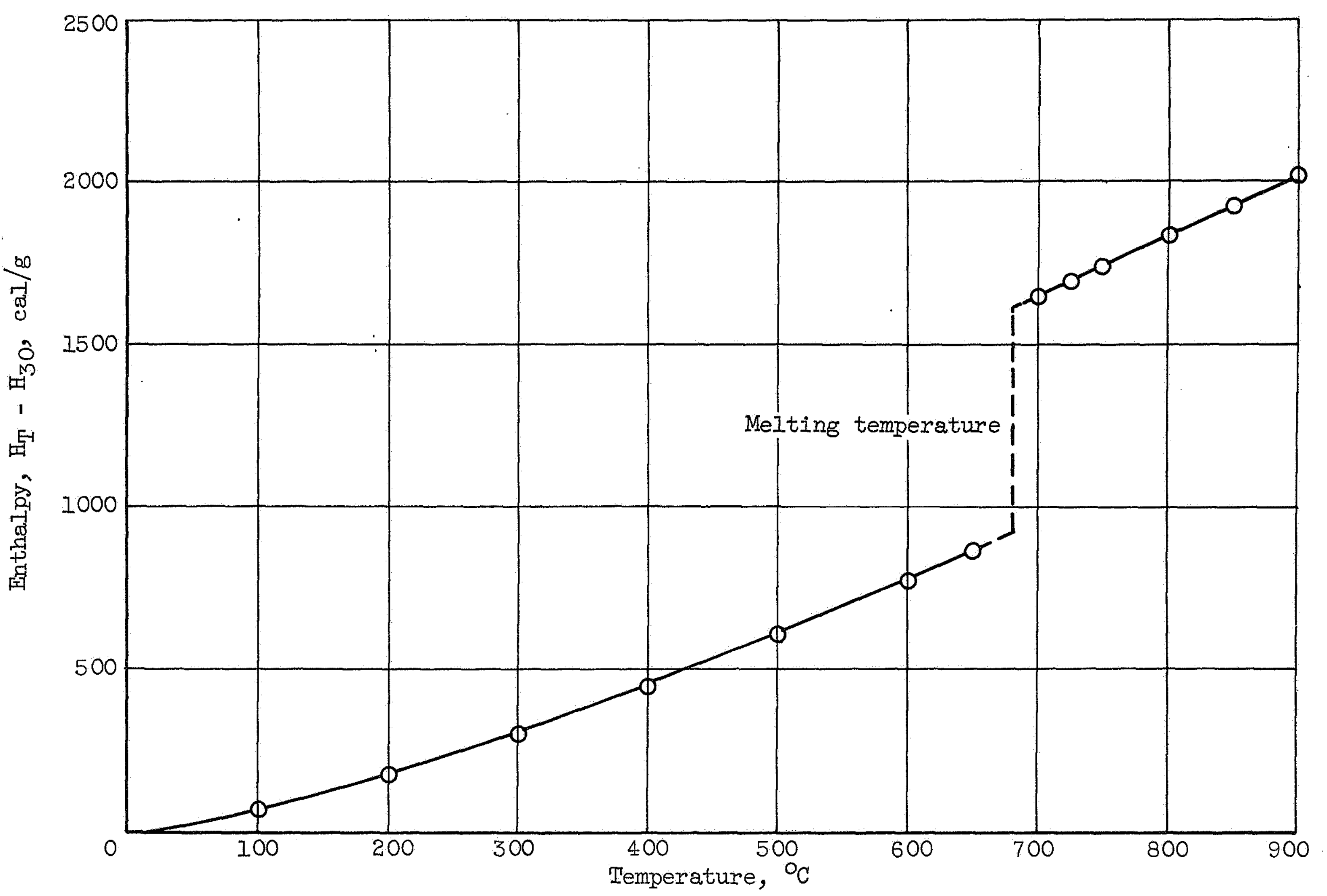


Figure IV-3. - Enthalpy of lithium hydride (ref. 236(SRD)). (This figure Unclassified.)

[redacted]

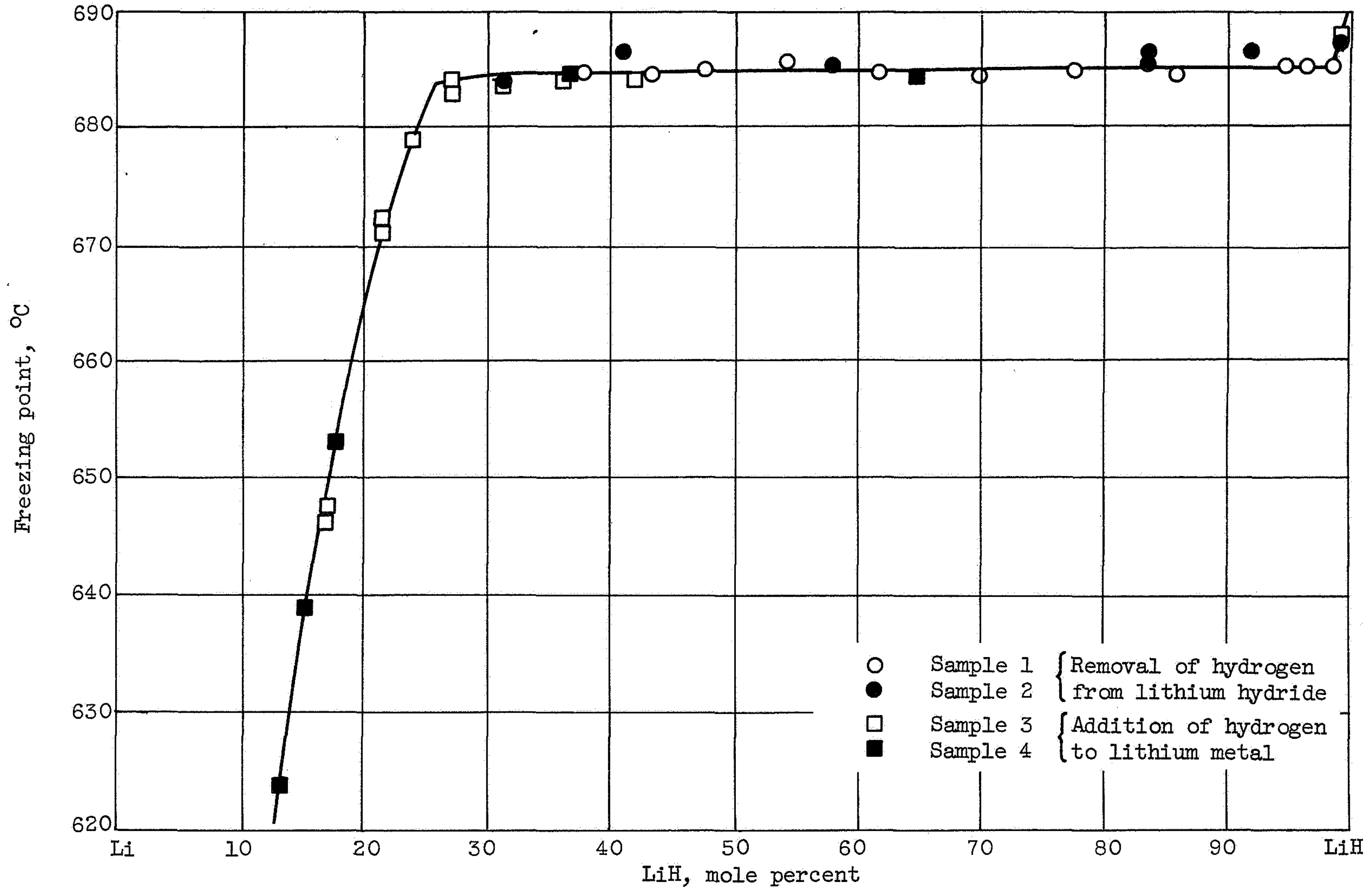


Figure IV-4. - Freezing-point composition, lithium - lithium hydride system (ref. 177(U)).

[redacted]

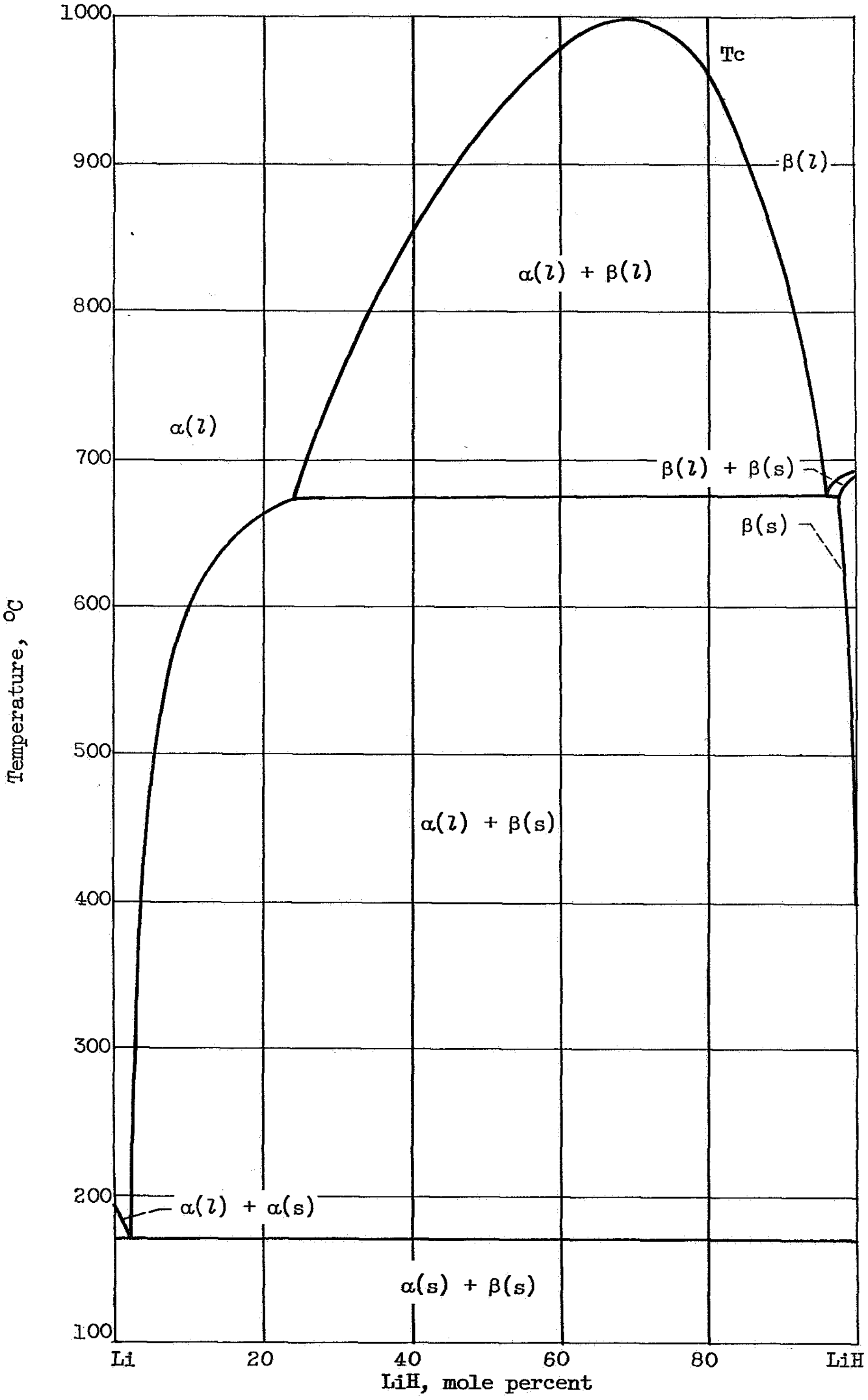


Figure IV-5. - Phase diagram of lithium - lithium hydride system. (Based on both experimental data and hypotheses, ref. 21(U).)

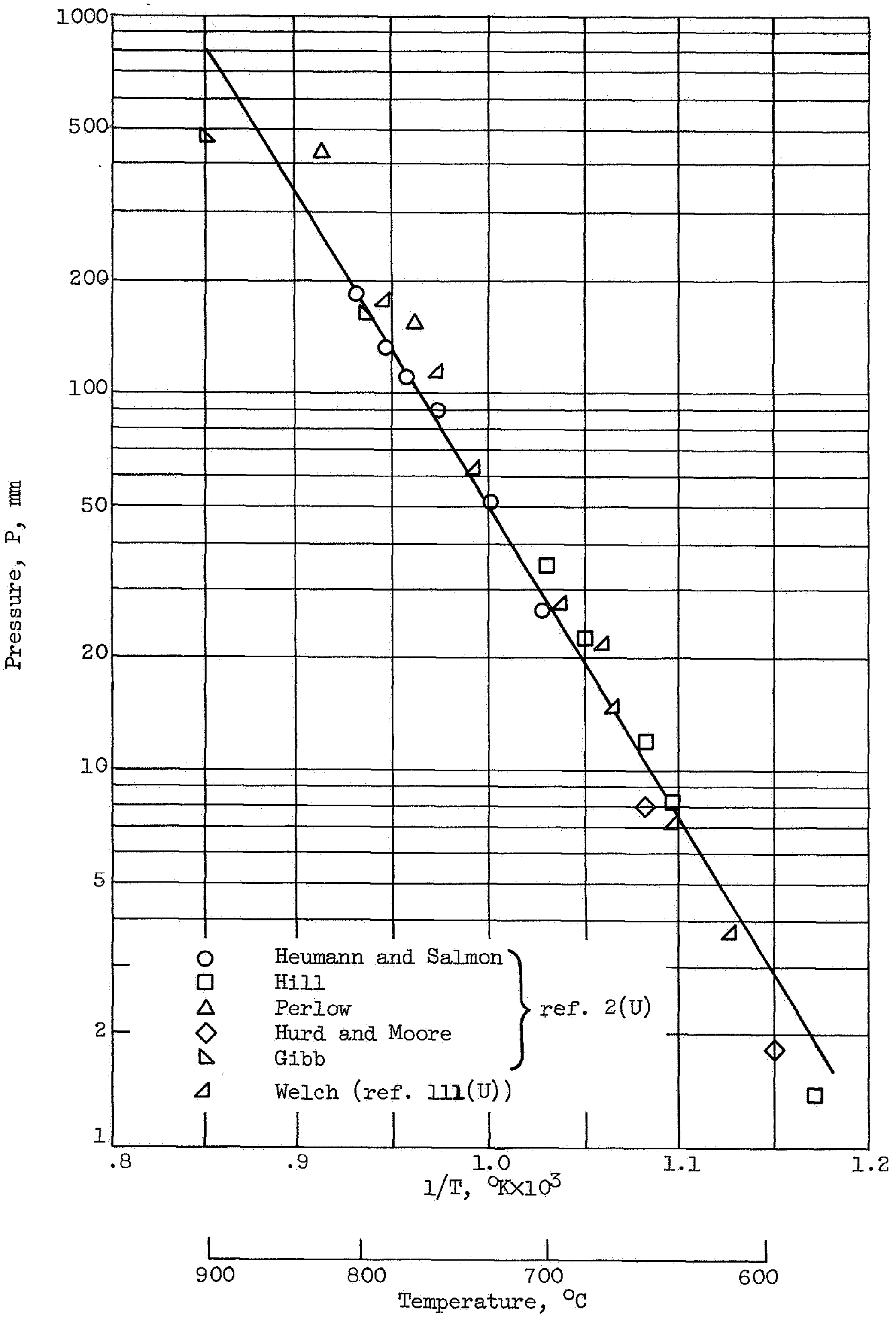


Figure IV-6. - Plateau dissociation pressures for lithium - lithium hydride system.

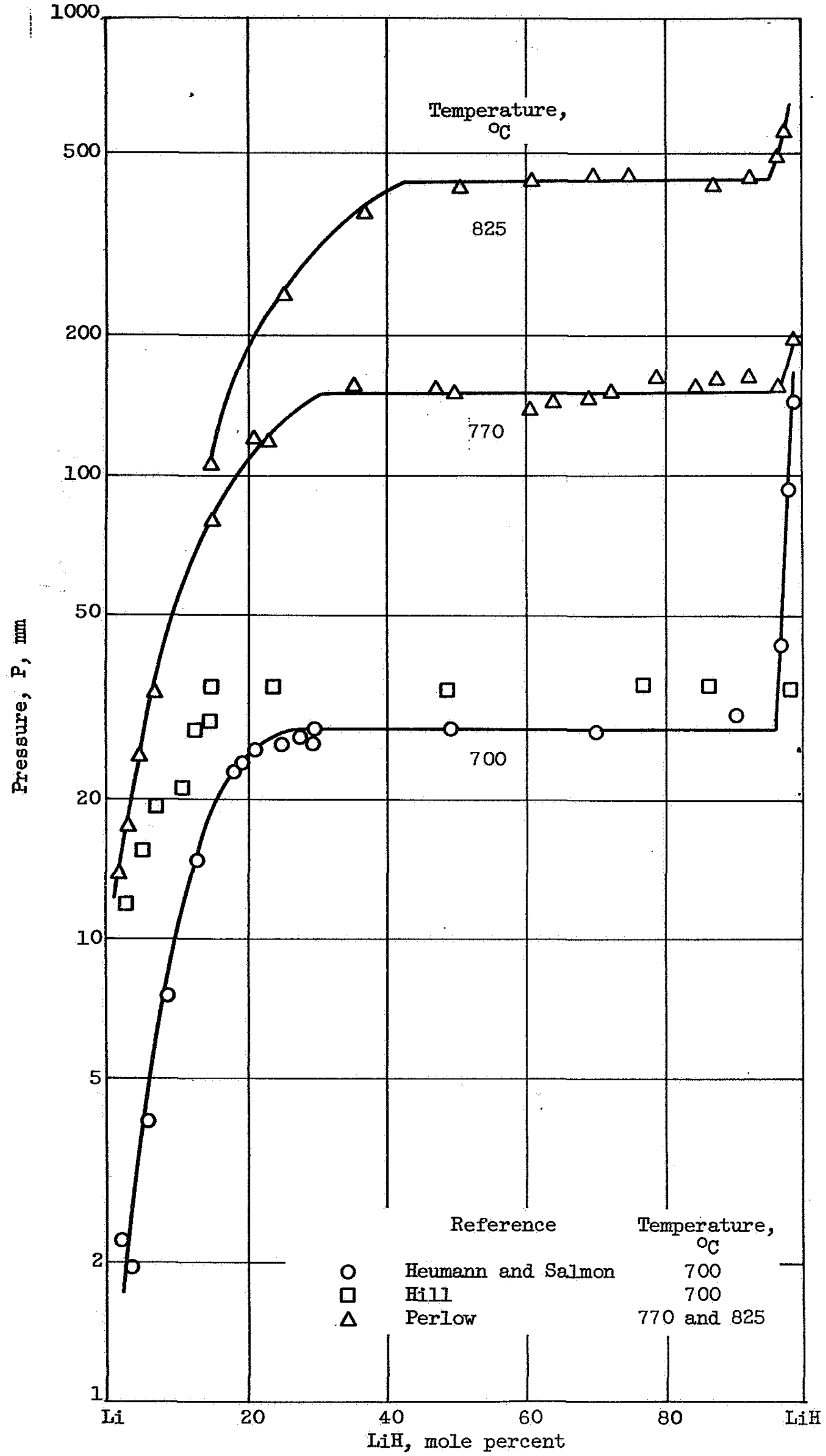


Figure IV-7. - Dissociation pressure isotherms for lithium - lithium hydride system (ref. 2(U)).

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V. MANUFACTURE AND RELATED SUBJECTS

[redacted]

V. MANUFACTURE AND RELATED SUBJECTS

A. PREPARATION

1. Laboratory Methods

a. Methods described by Messer

"Standard Preparation of Lithium Hydride

"The best available method is direct synthesis from the elements. No other way has been reported as a preparative method. The following directions may be used to obtain white crystalline material of 99% purity or better depending on the purity of the lithium metal used.

"Clean a crucible or boat of 316 or 347 alloy stainless steel, bake dry and partially fill with lithium cut in an argon atmosphere. (Carbon dioxide may be used with less satisfactory results but definitely not nitrogen, which reacts rapidly with lithium at room temperature to form the dark nitride.) Place the boat anaerobically in a stainless steel retort. Evacuate, heat to 100°C. in vacuum. Admit hydrogen (purified by passage over uranium turnings, or other getter) at atmospheric pressure. Raise the temperature to 750°C. for two hours. Cool in hydrogen, purge with dry argon, open retort and drop boat in a large volume of pure methyl alcohol. This procedure effectively removes lithium-soluble impurities. When boat is clean, rinse in methyl alcohol and ether. Dry and place in argon-filled drybox. Refill with purest available lithium previously washed with petroleum ether, etched in hexane containing a few drops of methyl alcohol, and rinsed in dry hexane.

"Transfer anaerobically to the retort, evacuate to 0.01 mu and heat to about 200°C. After one-half hour admit purified hydrogen and heat to 720°C. The cycle, if properly performed, results in removal of sodium and potassium which condense as fluffy pyrophoric hydrides on the cool portions of the retort. When hydrogen absorption ceases, lower the temperature to 690°C. and then allow the retort to cool very slowly between 690 and 680°C. Hold the temperature at 680°C. until no further hydrogen absorption occurs, then cool slowly to 500°C. and again hold the temperature until no further absorption occurs (over night, if necessary). Cool in steps of 100°C., noting any small absorption of hydrogen. When at room temperature, purge with <u>dry</u> argon or nitrogen and remove quickly, preferably anaerobically to a nitrogen or argon-filled drybox. The mass of large crystals may be iridescent on

> the upper surface which is scraped clean. The balance of the material is readily detached by tapping the boat and should be white clear crystals of lithium hydride. The purity and appearance will vary with the purity of the starting materials. Larger crystals may be obtained by using a similarly cleaned pure iron crucible of the Stockbarger type (conical bottom) and a thermal gradient technique. The iron becomes very brittle after cleaning and must be handled with great care. (Iron is used because of its permeability to hydrogen).
>
> "The manufacture of lithium hydride is briefly described in a literature reference to the lithium operations of Lithium Corporation of America [ref. 117(U)].
>
> "Recent attempts to improve the low-temperature oil-dispersion synthesis of lithium hydride have been reported by the Olin-Mathieson Chemical Corporation [refs. 237(U), 238(U), and 239(U)] . . . . Here, the liquid lithium is reacted with hydrogen dispersed in oil in the presence of a catalyst.
>
> "Ponomarenko and Mironov [ref. 54(U)] have described a modification of the standard preparation, based on an improvement of the earlier work of Albert and Mahé [ref. 15(U)].
>
> "Large single crystals have been grown by the Stockbarger technique by Pretzel [et al., ref. 101(U). See also section V.A.3.].
>
> "Suspensions of lithium hydride in hydrocarbons have been prepared in concentrations up to 15% by Ziegler [ref. 55(U)] . . . ." (Ref. 2c,d(U))

Reference 230(U) describes an attempt to prepare lithium hydride from lithium wire in trimethylene glycol dimethyl ether with hydrogen gas bubbled through it. Preliminary results only indicated that the ether was not inert to the reaction.

Other reactions leading to the formation of lithium hydride, but not at the moment of interest as methods of preparation, are:

$$LiAlH_4 \xrightarrow{200^oC} LiH + Al + 3/2\ H_2 \quad \text{(Ref. 2d(U))}$$

$$LiBH_4 \xrightarrow{300^oC} LiH + (?) \quad \text{(Ref. 2d(U))}$$

$$CH_3 \cdot CH_2 \cdot CH_2 \cdot CH_2 \cdot Li \xrightarrow{150^oC} LiH + CH_3 \cdot CH_2 \cdot CH = CH_2$$

(Ref. 240(U))

Ethyl lithium $\xrightarrow{120^{\circ}C}$ LiH + ? (Ref. 241(U))

LiR + pyridine $\longrightarrow$ alkylpyridine + LiH (Ref. 242(U))

C_6H_5Li + dihydrodibenzothiophene $\longrightarrow$ C_6H_6
+ dibenzothiophene + LiH (Ref. 243(U))

C_6H_5Li + 1,4-dihydronaphthalene $\longrightarrow$ naphthalene
+ C_6H_6 + LiH (Ref. 244(U))

1,4-dihydrodibenzofuran + C_6H_5Li $\longrightarrow$ dibenzofuran
+ C_6H_6 + LiH (Ref. 244(U))

$Li_3N + 2H_2 \longrightarrow 2LiH + LiNH_2$ (Ref. 32(U))

$Li_3N + 2H_2 \longrightarrow Li_3NH_4$ (Ref. 33(U))

$LiCH_3 + (C_6H_5)_3\ SnH \longrightarrow LiH + (C_6H_5)_3\ SnCH_3$
$+ (C_6H_5)_4\ Sn$ (Ref. 245(U))

$LiAlH_4 + LiCH_3 = LiH + ?$ (Ref. 246(U))

$2\ LiCH_3 = Li_2C_2 + LiH + ?$, 420° C (Ref. 247(U))

$4\ Li + C_2H_2 = Li_2C_2 + 2\ LiH$ (Ref. 23(U))

$6\ Li + C_2H_4 = C_2Li_2 + 4\ LiH$ (Ref. 23(U))

b. Direct synthesis

> ". . . In producing the hydride, pure metallic lithium is melted under high vacuum and is allowed to react with hydrogen. The initial evacuation, to a pressure of 1 mm. of mercury or less, is necessary to prevent reaction between the hot metal and oxygen, nitrogen, or moisture from the atmosphere. About 70 pounds of lithium are melted in each charge to the electrically heated hydride furnace. Hydrogen is admitted slowly until a positive pressure of about 5 pounds per square inch is built up. The reaction is highly exothermic, and once begun requires no further external heat to maintain the required temperature. The walls of the hydride furnace bowl are tapered toward the bottom, so that the hydride can be conveniently removed as a single mass when reaction is complete." (Ref. 248(U))

"Metallic lithium is placed in a relatively large iron boat and set inside of a tube furnace rigged for passage of dry, nitrogen-free hydrogen diluted with an inert gas. The hydrogen should also be passed over platinized asbestos and then through a liquid nitrogen trap if best results are desired.

"Lithium metal was placed in an iron boat [and] . . . a mixture of purified hydrogen and argon (50% by volume hydrogen) [was passed] through the tube containing the boat. At this dilution ratio the reaction is sufficiently slow to prevent overheating and spattering of the product.' (Ref. 13a(U))

c. Reduction of other lithium compounds

"A charge of finely divided Li compd., such as Li_2O, and a Si-bearing reducing material is confined in a reaction zone. Vacuum is applied to reduce air and moisture, and then heat applied to reduce the Li compd. and distil the metal to a cooler part of the reaction zone removed from the charge. Gaseous H is then admitted to form LiH. Cf. C. A. <u>40</u>, 5212(9)" (Ref. 249(U))

d. Reduction of LiOH

"LiH is produced by the action of a reducing metal on LiOH. Anhyd. LiOH is mixed with the finely divided metal and briquetted. The pellets are charged into a furnace and heated, in the absence of air, to about 300° to start the reaction, which is exothermic. The temp. must not be permitted to rise above the dissoc. temp. of LiOH. Cf. C. A. <u>41</u>, 840h." (Ref. 250(U))

2. Commercial Methods

a. Oil dispersion method, batch process

Olin Mathieson gives this method, with the ultimate use of the lithium hydride to be as an intermediate in the manufacture of high-energy fuels.

(1) Description

"The two methods of synthesis investigated during the batch program were the direct hydrogenation of lithium metal and the oil dispersion method.

"The direct hydrogenation program was carried out in a batch reactor with and without agitation. Several

[redacted]

[redacted]

experiments were performed and conversions as high as 80 percent were obtained. However, the temperatures required (750-930°F), the equipment problems involved, and the development time necessary made this method less desirable.

"The major effort was concentrated on the oil dispersion method. The investigation included studies of reactor geometry, agitation, catalysis, raw materials, temperature, pressure, reaction kinetics, particle size, dispersion agents and corrosion.

"Over 1300 experiments were performed using statistical methods to correlate data where possible and a reaction mechanism was developed and postulated from the bench scale data.

. . . .

"The batch hydrogenation program was undertaken to study the initial steps which would lead to a feasible continuous process for the production of lithium hydride . . ..

"The program demonstrated that high-quality hydride could be produced by the dispersion method and sufficient data were realized to provide the design basis of the pilot plant and a basis for many decisions in the design of the 5 ton per day production facility." (Ref. 238a(U))

(2) Conclusions

"1. High quality lithium hydride can be prepared by the oil dispersion method. Conversions of 98 per cent were obtained with a 2-hour residence time.

"2. Carbon in amounts ranging from 0.001 to 0.003 percent of total lithium charge increased the degree of conversion. The amount of carbon necessary depends upon the amount present in the lithium metal and in the materials of construction of the reactors.

"3. High temperature and pressure favor more complete conversion. This is partially a function of the increasing solubility of hydrogen in oil with increasing temperatures and pressures.

"4. A reaction mechanism has been hypothesized for the process. The reaction rate depends upon the diffusion of hydrogen through liquid lithium hydride and gaseous oil films. For reactor slurry temperatures of 430°F, individual particle temperatures are estimated to be 1500°F.

[redacted]

[redacted]

"5. Predispersion of lithium in oil decreases conversions. Reductions of 30 per cent in conversion were noted for a predispersed hold time of 1/2 hour.

"6. Dispersion agents, such as lithium stearate have no apparent effect on the hydrogenation reaction.

"7. Several dispersion oils were tested for interaction with molten lithium metal, and then used in reaction experiments. Dispersion oils found suitable for use are Standard Oil Corporation's 'Primol D'; Texas Corporation's 'Canopus D' and Socony Mobil 'Stock 318.'

"8. Increasing the peripheral velocity of the dispersator impeller up to 48 ft./sec. results in decreased particle size. Velocities above 48 ft./sec. result in relatively small further decreases in particle size.

"9. Lithium hydride is readily filtered and washed. Wash tests were conducted with _n_-heptane, _n_-hexane, petroleum ether and ethyl ether. [The ethers were found to be the best wash solvents with respect to the amount of oil removed per volume of wash liquid.]

"10. Static corrosion tests were performed on cast iron, Types 304, 316, 316 LC, 321, 347, 416, 446 stainless steels, phosphorbronze, monel, mild steel, and ingot iron. These tests were inconclusive. Practical experience indicates that the 300 series of stainless steels are [_sic_] most satisfactory for use in molten lithium service. The extra low carbon stainless steels (304, 316) or type 347 should be used in field work because of the danger of carbide precipitation at grain boundaries when welding stainless steels with normal carbon content." (Ref. 238a(U))

b. Oil dispersion method, continuous process

Olin Mathieson gives another method for the synthesis of lithium hydride as a step in the synthesis of diborane as follows:

(1) Summary

"An operable prepilot-scale apparatus for the continuous hydrogenation of lithium was assembled. Ten systems were investigated before the major problems encountered were solved and results were reproducible. The development of the process, and a description of the systems investigated and the problems encountered are given in this report.

"A micro bellows pump was found suitable to deliver the liquid-lithium at the feed rate desired (5 cc/min.).

Bellows-sealed valves, Flexitallic gaskets made from stainless steel and asbestos, and low-carbon Types 304 and 316 stainless steels solved the corrosion problems presented by the lithium and the reaction products. These materials were incorporated into the final apparatus. Satisfactory cleaning, waste disposal, and material handling procedures for use in this system were developed.

"A total of 93 experiments were performed in the final apparatus. It was found that agitation speed had an appreciable effect on both particle size and conversion. The per cent conversion was found to increase as temperature and reaction time were increased. Carbon, at a concentration of 0.002 per cent based on the weight of lithium, was effective as a catalyst; a 0.01 per cent concentration of lithium stearate in the dispersion oil also seemed to increase the conversion. The per cent conversion decreased as slurry concentration, particle size, and the amount of inert materials present in hydrogen stream increased. Lithium reacted extensively with Penn Drake No. 4451 oil above $464^{\circ}F$; Primol 'D' oil was found to be satisfactory as the dispersion medium at temperatures as high as $694^{\circ}C$.

"Lithium supplied by both Lithium Corporation of America, and Olin Mathieson Chemical Corporation (electrolysis of lithium chloride) resulted in comparable conversions and yields provided [the lithium was] filtered before use.

"The data from these experiments were correlated and are presented. An empirical relationship which relates conversion to slurry concentration, temperature, agitation speed, and reaction time was derived." (Ref. 237(U))

(2) Introduction

"A step in the synthesis of diborane is the hydrogenation of metallic lithium to form lithium hydride. A continuous prepilot scale lithium hydrogenation unit was built to provide data for pilot plant design, to gain experience with continuous hydride operation before pilot plant start up, and to verify batch hydrogenation data. It was necessary to develop specialized equipment, to determine suitable materials of construction and to develop material handling procedures. Since lithium metal has a melting point of $180^{\circ}C$ ($356^{\circ}F$) and it was desired to pump liquid lithium in the apparatus, various heating methods were also investigated.

[redacted]

"The principal obstacle was to pump lithium at a controlled rate into the reactor; ten different methods were tested before a micro bellows pump was found with which the low desired flow rate, 5 cc. per minute could be obtained. Neither magnetic flowmeters nor electromagnetic pumps could be purchased which could measure this low flow rate.

"The experimental unit which was finally developed was operated continuously for 9 days. An overflowing, stirred, tank reactor was used. It was necessary to develop procedures to control the operation so as to measure lithium flow rates and reactor slurry concentrations quickly.

"The variables which were investigated in this program were agitation, metal filtration, carbon content, lithium stearate content, slurry concentration, temperature, and the presence of inert materials in the hydrogen feed stream. Pressure was not investigated because the mechanical problems associated with its use could not be overcome at the time." (Ref. 237(U))

(3) General process description

"The process consisted of melting the lithium and removing the impurities by filtration through a sintered stainless-steel filter having 60-70 micron openings. Since most impurities either float or sink in the molten metal, the filter was placed a few inches off the bottom of the melt tank, thus assuring that a minimum quantity of impurities would have to be removed by the filter. This filter placement considerably increased the useful life of a filter before it had to be cleaned. After being filtered, the molten lithium was fed into the reactor at a level slightly below the agitator.

"The hydrogen was purified to remove oxygen and moisture before it was introduced into the reactor. This was done by passing the hydrogen through a lithium hydride scrubber placed between the hydrogen feed tank and the reactor. The hydrogen was fed into the bottom of the reactor so that it could be dispersed by the dispersator as thoroughly as possible in the reaction mixture.

"The oil received from the supplier was placed in a vessel where catalyst and a dispersing agent were added, if they were required for the experiment. From this vessel, the dispersion oil was pumped through a preheater and into the top of the reactor.

[redacted]

"The reactor contents were agitated by means of a Duplex Dispersator purchased from the Premier Mill Corporation, Auburn, New York. From the reactor, the slurry was directed to either another reactor or into a reactor receiver. (In prepilot work, a second reactor was not used.) The excess hydrogen was separated from the reacted material in the reactor receiver. This was accomplished by venting the hydrogen through a bubbler to the atmosphere outside the building.

"The oil was then separated from the hydride cake. Both centrifugation and filtration were tried, but filtration was found to be the better method.

"The filtered oil can be stored, recycled directly, or chemically treated to remove unsaturated compounds or color, before recycling. To date, the study of the effect of recycling the oil has been inconclusive.

"The oil-wet hydride cake was then washed free of oil with a solvent (ethyl ether) and the filtrate was distilled; the ether was reused. The waste oil from the still was disposed of by burning because of the possibility that it might contain residual ether. The hydride was then dried in an argon atmosphere and packaged in static-resistant polyethylene bags." (Ref. 237a(U))

(4) Conclusions

Olin Mathieson gives the following conclusions on the continuous process after its exhaustive development program to produce lithium hydride:

"1. A correlation exists between product particle size and product conversion showing an increase in conversion with a decrease in particle size. . . .

"2. A correlation exists between product particle size and power input (at the impeller) showing a decrease in particle size with an increase in power input. . . .

"3. In the range investigated, the effects of pressure and catalyst concentration were not evident. This could be due to inherent system variables or to the narrow range of pressure and catalyst concentration used in the experiments.

"4. The increase of hydrogen solubility in oil due to increased temperature and pressure alone was insufficient to maintain the required reaction rate. It was necessary to sparge hydrogen into the reaction slurry in sufficient quantity to assure an equitable distribution and an adequate rate of solution.

[redacted]

"5. The results of the metallurgical examinations and operating experience indicated that carbon steel is not adequate material for fabrication of the hydrogenation reactors.

"6. The Lapp diaphragm pump was unsatisfactory [in this application because of poor flow control at the low flow rates used (5 cc/min)].

"7. The electromagnetic pump proved to be satisfactory for pumping liquid metal. Good control, even at low flow rates, was obtained with this pump.

"8. The magnetic flowmeter was satisfactory for measurement of liquid metal flow rates. The accuracy of this instrument, however, was impaired by the low flow rates used in the pilot plant, (2 g.p.h.).

"9. Counter-pressured double mechanical seals were successfully used for sealing pumps and agitators handling lithium hydride-ether and lithium hydride-oil slurries.

"10. A liquid metal level indicator utilizing a $1\frac{1}{2}$ volt potential to ground was used successfully. It is accurate to ±1/16 inch.

"11. Prior filling of the electromagnetic flow tube and transfer lines with metal reduced frequency of plugging after shutdown.

"12. The presence of free lithium in a slurry presented a difficult pumping problem. The free metal tended to coalesce and "plate" out on the pump impeller. Metal also plugs the pump discharge port, flow control valves and other restrictions in the line.

"13. Hydrogen starvation in the first reactor resulted in some reaction between the metal and dispersion oil. As a result overall conversions to hydride were reduced.

"14. Hydride-oil slurries are readily filtered. The filtration rate is directly proportional to differential pressure.

"15. Complete removal of oil from the filter cake proved difficult. Cake washing efficiencies averaged 77 per cent based on six experiments. A wash ratio of 12 lb. ether/1 lb. hydride was used.

"16. Use of heating and vacuum systems insures essentially complete removal of ethyl ether from filtered dispersion oil, (less than 1 per cent ether).

"17. Dryer and still capacities can be increased by providing individual heat-transfer oil systems.

"18. The use of a LaBour 5 DZT, close coupled centrifugal pump, equipped with a John Cranes single,

[redacted]

[redacted]

outside, balanced mechanical seal, eliminated the pumping problems encountered in the distillation section.

"19. A filtered metal hold tank was necessary to provide a reservoir of filtered metal and a positive suction head for the liquid metal pump.

"20. Good results were obtained when a reaction was conducted at 536°F and 25 p.s.i.g. with 0.002 per cent catalyst. Increasing reaction temperatures and pressures should increase the reaction rate."
(Ref. 239(U))

c. Production of lithium hydride powder

"A method for the production of LiH powder is described. Li_2Cl_3 was converted into the chloride. From a saturated solution of the chloride lithium amalgam was produced by electrolysis. The lithium amalgam was then converted into the hydride by heating the amalgam in a hydrogen atm. The app. used for the reaction of the lithium amalgam is described, and the conditions are given. At a reaction temp. of 600°C, a yield of 87% is obtained. The product was light gray and crumbled to powder at a light pressure." (Ref. 251(U))

d. Direct synthesis

"The usual commercial preparation [see ref. 249(U)] of lithium hydride is by direct union of the elements. A charge of a finely ground lithium compound, such as Li_2O, is confined in a reaction vessel with a reducing agent such as aluminum powder. The vessel is evacuated to remove air and moisture, and it is then heated to reduce the lithium compound. The metallic lithium thus produced is distilled to a cooler part of the apparatus to separate it from the charge, and gaseous hydrogen is admitted producing the LiH. The hydrogen is used diluted by an inert gas to control the usually rapid reaction and excess heat . . . .

"Another commercial process . . . is based on the use of an organ-metallic catalyst. In this process molten lithium metal is mixed with finely-divided NaH and about 0.1 - 1% by weight of a fatty acid of more than 8 carbon atoms, e.g. caprylic, stearic, oleic, palmitic, linoleic, and their metal salts. Hydrogen is then introduced, and the temperature maintained at 200 - 450°C. Under these conditions the rate of absorption is reported to be rapid and the reaction complete." (Ref. 13a(U))

[redacted]

3. Preparation and Purity of Single Crystals

> "It is possible to prepare single crystals of LiH from the melt by a method which is, in many ways, similar to those previously used for different materials by Bridgman, by Obriemov and Schubnikov, and by Stockbarger . . . . The dissociation pressure of LiH is not exceedingly high at its melting point, but the literature . . . indicates that the dissociation pressure over the molten salts of the other alkali hydrides is probably too great to allow the preparation of crystals of the higher alkali hydrides from the melt.
>
> "The procedure used to prepare a single crystal of LiH includes the following: (1) cleaning a pure iron crucible; (2) distilling lithium metal into the crucible; (3) sealing the crucible by welding under vacuum; (4) heating and outgassing the crucible in the apparatus . . .; (5) introducing purified hydrogen under pressure to synthesize molten lithium hydride inside the crucible; (6) establishing a proper thermal gradient through the melt by increasing the heat losses down the crucible support; (7) crystallizing the melt by slowly cooling the furnace; and (8) freeing the crystal by removing the crucible under an inert atmosphere. The steps are described in more detail in [ref. 101(U)]."
>
> . . . .
>
> ". . . The crystals are normally obtained in the form of cylinders which are about 16 mm in diameter and 50 to 80 mm long. The {100} cleavage planes of the crystal do not normally include the axis of the crucible. The single crystals are cleaved into plates for annealing under H_2 to remove strains and traces of colloidal Li, which render the crystal slightly blue. Annealing is carried out in a large crucible . . . and under conditions similar to those used for crystallization, except that the gradient is held at a minimum and the maximum temperature is about 550° C to keep the plates from sticking together. If annealing under H_2 has been continued for a sufficient length of time, the crystals obtained are clear, colorless, and also free from strains." (Ref. 101(U))

The Los Alamos work on single crystals produced these remarks on crystal purity:

> "The composition of the lithium hydride crystals which have been prepared at this laboratory by the above method, or by earlier variations of it, include [<u>sic</u>] the various isotopic

combinations[a] LiH, LiD, Li^6H, Li^6D, and either of the first two containing small quantities of LiT. The thirty-odd crystals prepared so far also include mixed crystals with LiF, and those doped with MgH_2 or with impurities such as MgO and Li_2O. Studies of the distribution of impurities indicate that most of them -- with the possible exception of MgH_2 -- are swept to the top of the crystal rendering this portion cloudy and more dense than the bulk of the crystal. During attempts to grow clear single crystals of LiH from the best available salt it was found that crystals of better and more uniform quality could be obtained by adding a small amount of magnesium metal to the melt, apparently because the MgO impurity is segregated into the top crust more efficiently than the Li_2O impurity normally present in the salts. Mg-doped LiH crystals, like LiF, possess better cleavage characteristics than undoped crystals.

"The purity of the LiH single crystals and of the starting materials have [sic] been tested by spectrographic analysis, and chemical analysis has also been done on Mg-doped crystals . . . .. From these measurements typical concentrations of metallic impurities in molar p.p.m. detected in 'pure' LiH crystals are: Na(20-200), Mg (0.5-6), Fe (0.5-2), Cu (0.5-2). Other metallic impurities are evidently present at concentrations <1 molar p.p.m., although the spectroscopic sensitivity may not be this high in some cases.

"The most important non-metallic impurities, oxygen and nitrogen, cannot be detected with comparable sensitivity by spectrographic or chemical means. Sensitive density measurements are used to estimate that non-metallic impurity concentrations, estimated as oxide, are in the 10-1000 molar p.p.m. concentration range. The isotopic purity of the D_2 gas was checked by mass spectrometry before and after preparation of the salt. The isotopic purity was found to be greater than 99 per cent and nearer to 99.9 per cent for some crystals of LiD. These results indicate that some of the single crystals of LiH or LiD prepared are of comparable purity to single crystals of the alkali halides which are commercially available." (Ref. 101(U))

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[a] The symbol Li alone indicates lithium of normal 92.5 per cent abundance in Li^7 whereas the Li^6 was approximately 95 per cent enriched in that isotope.

[redacted]

B. HANDLING

1. General

"If the hydride is to be heated or stored in an hermetical, full container, it is imperative that no gas shall be evolved irreversibly. This is readily ensured by 100% anaerobic handling of the hydride from the time it is prepared. For large scale operations, where a mass of large crystals is obtained from the preparation, these may be transferred in dry air to a nitrogen drybox without serious contamination. All crushing and grinding operations should be carried out in dry nitrogen, argon, or helium and the crushed or powdered product not thereafter exposed to air. [See discussions of precautions during casting, section V.C.5.]

"Lithium hydride dust is apt to explode in humid air [presumably because of local heating of particles by reaction with moisture] or, even in dry air, due to static electricity. Moreover, it is extremely irritating to the mucous membranes and skin, causing an allergic skin reaction in some individuals. Lithium salts are sometimes toxic. Drybox handling eliminates these hazards.

"The hydride must not be moistened or exposed to an open flame. Burning hydride is best extinguished by smothering with an inverted can or pail, or by covering with bone dry graphite powder or dolomite. Carbon dioxide, carbon tetrachloride, or aqueous fire extinguishers, must <u>not</u> be used since an explosion may result. Sand may react explosively with burning hydride, especially if sand is not completely dry, or contains hygroscopic sea salt." (Ref. 2u(U))

"A method for handling pure lithium hydride was not available at the start of this [development] program. Commercially available lithium hydride, could be handled in the atmosphere because it was coated with lithium hydroxide. It was found, however, that the material produced in this program would spontaneously ignite and burn in the atmosphere if the protective oil coating was disturbed. On several occasions, when oil was removed, the hydride was ignited because of reaction with atmospheric moisture.

"It was evident that removal of the oil film while washing the product left the hydride very reactive. To counteract the reactivity, an inert gas purge was kept over the hydride after removal of the protective film. Nitrogen has been used successfully for this purpose.

[redacted]

[redacted]

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"Nitrogen was found to be detrimental in the presence of liquid lithium. Lithium reacts preferentially with the nitrogen in air, and the reaction is greatly increased at elevated temperatures. Therefore, argon, helium, or other inert gases must be used as a purge for liquid lithium.

"No special precautions were found to be necessary for handling oil-wet hydride. The oil coating was found to be sufficient to prevent reaction with the moisture in the air.

. . . .

"Several wash solvents, n-hexane, n-heptane, petroleum ether, and diethyl ether were tested. Tests proved that the ethers were superior with respect to the amount of oil removed per volume of wash liquid. . . ." (Ref. 237b(U))

"Spills of hydride should be wetted with oil and wiped or swept up with a rag or broom dampened with oil. The residual hydride can then be reacted with water, methanol or some other solvent suitable for use in the area where the spillage occurs.

. . . .

"Drums, liners, gloves, vessels and all equipment that will come in contact with hydride must be absolutely dry and well purged with inert gas if possible." (Ref. 238b(U))

2. Containment of Lithium Hydride

"The reactivity of the hydride with air makes necessary an air-tight moisture-proof container. If the container is to be heated, it must also be impermeable to the hydrogen produced by thermal dissociation of the hydride. Several types of plastic containers (aluminum foil-lined) and ceramics are generally suitable for room temperature use but metals are preferred at higher temperatures. Stainless steels 316 and 347 have a combination of hydrogen impermeability and hot strength which suggests their use for containers.

"Owing to the fact that stoichiometric LiH exhibits a higher thermal dissociation pressure than compositions containing slightly less hydrogen, and since the decrease in hydrogen content may be offset by the attendant decrease in necessary container thickness; it follows, that there will be an optimum composition containing somewhat less than the stoichiometric amount of hydrogen which, when loaded into a minimum weight shell, will give the maximum over-all N_H at a given temperature. This composition and shell-thickness may be evaluated for cylindrical containers using the A.S.M.E. code formula for maximum safe working

[redacted]

pressures and the data on density and dissociation pressure for lithium hydride cited [in sections II.D.2 and IV.O.]. The rapid decline in tensile strength with temperature of container materials must be taken into account. This calculation yields an optimum design of container and system which may be scaled up in size until the walls become thick enough to prevent diffusion loss at operating temperatures, as well as to safeguard against superficial flaws in the metal.

"The relatively great thermal stability of [solid] lithium hydride implies that the container may be very light and that the N_H of hydride-plus-container should be only slightly less than for unclad hydride up to approximately 800°C. Hydrogen diffusion loss through stainless steels according to Gibb . . . [ref. 226(U)] is given below.

Alloy	Temperature	Pressure	$cc/cm^2/mm/hr.$
316	650°C	1 atm.	0.009 to 0.03
347	650°C	1 atm.	0.01 to 0.04
304	650°C	1 atm.	0.003 to 0.04

For molybdenum, the diffusion loss at 650°C. and 1 atm. is calculated to be of the order of 4×10^{-12} $cc/cm^2/mm/hr.$ although it is doubtful if the commercial sintered metal would show the theoretical permeability." (Ref. 2v(U))

"Large crystals of lithium hydride can be stored in ordinary closed containers for short periods without serious decomposition occurring. Special care is required when the lithium hydride is finely divided since the large surface area increases the decomposition rate. Therefore, to insure maximum stability, the containers should be completely sealed. When lithium hydride is powdered in a dry box, an electrostatic charge builds up on the surface of the box which may produce spontaneous combustion of the material if nitrogen and oxygen are present." (Ref. 19c(CRD))

"The slow increase in hydrogen pressure observed in systems housing lithium hydride is due to the reactions described by the equations in [section I.A.2. from ref. 19(CRD)] stage (2). The pressure increase can be eliminated by heating the material to drive the second reaction to completion, evacuating to remove the hydrogen gas produced, and then sealing the container." (Ref. 19a(CRD))

Reference 252(U) gives the Interstate Commerce Commission regulations which include specifications for shipping containers for lithium hydride. In general, steel drums are required.

3. Fire and Safety

a. Personal safety equipment

> "Operators handling quantities of lithium hydride should wear the following protective clothing: goggles, face shields, or both, rubberized gloves, flameproof outer clothing, and a respirator." (Ref. 117a(U))

b. Fire extinguishers

> "If lithium hydride does ignite, the fire is not violent and can be extinguished by exclusion of air and moisture. Dry nitrogen, graphite, or lithium chloride may be used to extinguish lithium hydride fires. The use of dry nitrogen for extinguishing fires in finely ground lithium hydride should be done with caution since a stream of nitrogen, if played on the finely ground material, may cause dusting of lithium hydride and a consequent spread of the fire. A very effective means of extinguishing lithium hydride fires which are confined to drums is to exclude the air by placing the cover on the drum. The cover should, of course, be free to move in case there is a pressure build-up in the drum.
>
> "To extinguish fires, never use water, carbon tetrachloride, chlorinated hydrocarbons, soda acid, chemical or carbon dioxide fire extinguishers." (Ref. 117a(U))

c. Treatment and antidotes

> "In case of accidental contact with the skin, the particles should be brushed off at once, and the affected area flooded with sufficient water to remove adhering particles of hydride. After washing, treat the exposed area as a caustic burn. In case of inhalation of lithium hydride dust to the point of discomfort, call a physician because some individuals may be highly sensitive to lithium." (Ref. 117a(U))

C. FABRICABILITY

1. General

"Powdered lithium hydride is easily formed into pellets of theoretical density by powder-metallurgy techniques. No binder is used. The dry powder is loaded anaerobically into the clean die and compressed at 20 [tons/sq in.] or more. Most of [Messer's] experience was with a 1/2" carbolloy cylindrical die, but no difficulty was encountered with a 1.8" tool steel die (Airkool Steel Rockwell 65), or with a home-made steel press-plate which made 1/2" square bars. The pressings are conveniently carried out by loading the die in a drybox and extruding the pellet into a jar containing argon and held by a rubber flange on to the base of the die. The pressing characteristics resemble those of dry sodium chloride and very hard pellets may be obtained. In the event that the die is not highly polished, it may be necessary to wipe the walls with a suspension of colloidal graphite (DAG 154) in acetone, and then dry thoroughly. Very little graphite wipes off on the pellets if this is done properly. The pressed shapes may be sawed (in the drybox) and [have a hard glossy white appearance].

. . . .

"Lithium hydride may also be cast into shapes. This operation is rendered difficult by the unusually large shrinkage when the melt solidifies. This has been estimated as 16% [ref. 253(U)]. The solidifying melt shrinks from the center leaving usually a funnel-shaped indentation. The edges of the top meniscus are drawn down sharply from the walls towards the center and some cracking may occur. The lower parts of the casting are usually sound, at least in small molds where the thermal gradients are not large. In any event, the thermal gradient should be such that the bottom cools first. The cooled polycrystalline product adheres tenaciously to the mold but the material is sufficiently brittle so that tapping the mold often frees the casting. [Messer has] no data on the bulk density of castings, but it is likely that fissures, voids and lattice imperfections will reduce this to a small extent." (Ref. 2u(U))

2. Hot Powder Pressing

A condensation of some work reported by Los Alamos Scientific Laboratory on hot-pressing follows (ref. 254(U)):

(a) Die material. - Graphite was used, mainly because of its good machinability and high-temperature characteristics. The disadvantages of low tensile strength and nonuniformity of properties from piece to piece were not sufficient to prevent use in these tests.

(b) Powder lubricant. - Several lubricants were mixed with the lithium hydride powder to aid the pressing process. Paraffin, or other lubricants such as cetyl alcohol dissolved in an organic solvent such as ether, were tried and abandoned because of poor results. A density of 0.81 gram per cubic centimeter was obtained, indicating that the hydride acquired many impurities such as lithium hydroxide and lithium carbide.

(c) Particle-size distribution of powder. - Charges were hot-pressed in a 1-inch-diameter graphite die at 1500 pounds per square inch and 615° C die-case temperature. Oil dag was used on the die as lubricant. To protect the compact from oxidation, hydrogen was run into the die cavity during the heating cycle and argon was used during the cooling cycle. Results are given in table V-I.

(d) Die coating. - It was found that some sort of coating on the graphite dies was desirable to prevent the lithium hydride from sticking to them and to prevent absorption of the hydride into the pores of the dies. The following methods were tried:

(1) Lining the die with sheet metal

(2) Covering the graphite surfaces with a sprayed layer of metal

(3) Impregnating the pores of the graphite with some inert material

(4) Electroplating the die surfaces

Stainless steel and nickel sheet were tried and worked satisfactorily for very small sizes, but it was felt that differential expansion would rule out their use for larger pieces. Also, nickel is chemically attacked by the hydride.

Sprayed-metal surfaces also suffer from differential expansion. Sprayed stainless steel was found to be satisfactory, but it took a great deal of work to obtain a good surface.

Lithium fluoride was tried as a filler for the pores in the graphite but seemed to be of little value. Also, it appeared to weaken the dies.

A graphite die having a thin layer of electroplated iron on the working surfaces showed promise. The deposited iron was quite pure and therefore was not readily attacked by the lithium hydride. The thickness of the iron plate was usually between 1 and 3 mils. Because the iron was so thin, it did not seem to be affected by the differences in the coefficients of thermal expansion between the iron and the graphite. The loss of material (lithium hydride) was reduced to 1 percent or less (during pressing) when an iron plate was used, and the average loss on large pressed pieces was about 0.5 percent.

The surfaces of the die to be plated were first painted with a layer of conductive silver paint. The die cases were used to hold the plating operation. To eliminate the necessity of immersing the entire die into the plating bath, an anode was placed in the die cavity when the inner surfaces of the die were plated. A hot chloride bath, 85° to 87° C, was used, and all dies and die parts were preheated. In use, the dies were coated with a layer of Aquadag to prevent the lithium hydride from sticking to the iron.

Graphite dies electroplated with different metals were also tried. The powder used showed a hydride content of 94.36±0.24 percent. This powder was used in the runs cited in table V-II, all of which were made at 600° C and 1000 pounds per square inch. Fabricated pieces have shown a purity of 95 percent LiH with a density of 98 percent of theoretical.

This series of runs indicated that the iron plate was about as good a plate as any. Prefiring the plated die at 600° C before pressing hydride in it seemed to help a little. Very often the plate would peel off during prefiring, however.

(e) Pressure, temperature, and density relations. - A number of small, cylindrical hot-pressings were made to determine these effects. The results are presented in graphical form in figure V-1. All pressings were in 2-inch-diameter dies and the compacts were about 2 inches long. The regular -20-mesh 95-percent-pure lithium hydride powder was used. The 300° and 400° C runs were made in steel dies because of the high pressures involved.

Runs were also made to determine the effect of pressing time on the density of a compact pressed at a certain pressure and temperature. These runs were similar to the previous runs made in steel dies. The results are presented in graphical form in figure V-2.

(f) Powder preparation. - The lithium hydride was a brittle, friable material that could be easily ground into powder. It could be ground by hand with a mortar and pestle or crushed with a jaw crusher, hammer mill, ball mill, etc. Most grinding methods produced a large amount of very fine powder, which increased the difficulties of handling and hot-pressing and tended to reduce the powder purity.

Ball milling and jaw crushing both produced a great deal of very fine powder - about 20 percent was -80 mesh. A method was devised of removing some of the finer material as it was being crushed in a ball mill. This reduced the -80 mesh to 12 percent.

Some of the hydride received was very hard and would not mill. The purity of this hard material was the same as the friable material. The only obvious difference was that it had a very small grain size, and the friable material had a large one.

(g) Miscellaneous. - The hot-powder-pressing experience of the General Electric Company is related as follows:

> "Work on this method of fabrication has indicated that some of the disadvantages of cold powder pressing are not serious in this case. The theoretical density is easily obtained and the reaction of lithium hydride and lithium hydroxide is driven to completion at the temperatures and pressures resulting in no pressure build-up in the container. Common hot pressing conditions are 1100°F and 1000-1500 psi in graphite dies. The disadvantages of this method are the special precautions required to prevent cracking of the lithium hydride from shrinkage on cooling, and the long heating and cooling times that are necessary for the hot pressing operation. The fabrication techniques described above are summarized in [the following table]:

	% Theoretical Density	Gas Build-up	Machinability	Adherence to Container Wall
Casting	99	Nil	Poor	Good
Cold pressing	95	Some	Good	Poor
Hot pressing	99	Nil	Good	Poor

(Ref. 19c(CRD))

3. Cold Pressing

The General Electric Company's cold-pressing techniques are reviewed as follows:

> "The hydrostatic pressing technique was adopted several years ago as a method for fabricating . . . lithium hydride. Granular hydride is vibration-packed into a thick-walled flexible rubber or plastic bag, sealed and evacuated. The bag is loaded into an oil-filled chamber and pressure applied. About 95 percent of theoretical density is obtained with a pressure of 30,000 psi. The bag is stripped off leaving a dense compact which can be machined, drilled, sawed, etc. into a variety of shapes. . . .
>
> "The cold pressing technique was used to press a lithium hydride-filled metallic honeycomb . . . . The pressing was sectioned and the honeycomb (0.005 inch Type 321 stainless steel) was found to have been slightly distorted during compression of the hydride.
>
> "The cold-pressed (22,500 psi) hydride outside the honeycomb had a density of about 94 percent of theoretical, while the hydride in the honeycomb had a density of only 91 percent.
>
> "The technique of cold pressing into a heavy wall honeycomb, although possible, did not appear to be suitable [for the use intended]. Stainless steel honeycomb made with 0.002 inch thick foil was obtained and additional hydrostatic pressings were made. The compacts had only slightly increased . . . density (~92 percent of theoretical) . . . .
>
> "A . . . honeycomb [was formed] of 0.001 inch thick stainless steel foil (one inch square cell) containing perforations, based upon the belief that the thin perforated foil would give less resistance to the compacting forces, [would give] higher lithium hydride density, and in addition, [would] reduce the bulk density of the compact. Development pieces of this honeycomb (Figure [II-11]) were obtained with two different size perforations (1/2 and 11/16 inch) and compacts prepared with a resultant lithium hydride density about 0.73 g/cc (94 percent of theoretical). Additional details on the . . . characteristics of the perforated honeycomb compacts are included in [section II.D.3.]." (Ref. 141c(CRD))

Other cold-pressing data of Waldrop follows:

> ". . . The size of compact which one can prepare by [the isostatic cold-pressing] technique is limited only

> by the size pressure vessel which [can be built] . . . . Our experience has shown that compacts of a range of sizes from two inches in diameter by three inches long to about 25 inches diameter by 36 inches long will have a very uniform density distribution. . . . " (Ref. 113e(SRD))

See also sections II.D.2. on density and V.C.6. on machinability.

4. Sintering

The General Electric Company notes that vibrational sintering with and without applied pressure was not a successful method of fabricating dense lithium hydride shapes (ref. 141c(CRD)).

> "To determine the effect of temperature upon the sintering of the powdered hydride, a 10 inch diameter can was loaded with lithium hydride powder and held at 1100-1150 F for four days. A dense, crystalline cylindrical body only nine inches in diameter was obtained. Very little flow occurred at the bottom of the cylinder. The specimen was reheated at 1180F for 63 hours to see if flow would occur. Again very little flow was noted.
>
> "It is believed that, even at temperatures near the melting point, time and temperatures alone are not sufficient to cause lithium hydride to flow and conform to the container, and apparently some externally applied pressure must be present." (Ref. 141d(CRD))

5. Casting

a. General

Because of the large amount of shrinkage (approx. 19%, see section II.D.2.) that occurs during solidification, special measures must be taken to prevent formation of a "shrink pipe" or unwanted void when the liquid cools and solidifies.

> ". . . the hydride generally wets the mold in which it is cast and adheres very tenaciously so that . . . the casting must be made in the container in which it is to be used." (Ref. 113b(SRD))

Welch reports on General Electric's casting experience as follows:

> ". . . All of the methods used were necessarily complicated by the extreme reactivity of the hot lithium

hydride with air, water vapor, etc. requiring an inert atmosphere over the casting.

. . . .

"In general, the casting development program established the 'zone cooling' method [see section V.C.5.b.] as a very versatile and useful method of producing relatively large and intricate shapes of lithium hydride limited only by the available equipment. The cast material is superior to the cold pressed material for several reasons:

1. Higher density: $\sim$99 versus 93-94 percent of theoretical density.
2. Higher purity: $\sim$98 versus 95-97 percent.
3. Higher thermal conductivity
4. Closer fit to container (no machining tolerances) and subsequently better heat transfer characteristics.
5. Versatile: easy to fabricate shapes directly into intricate containers.
6. Cheaper: requires no expensive machine shop equipment
7. Faster: large, intricate shapes can be made without time consuming machining, etc."

(Ref. 141e(CRD))

"Casting operations involving the molten hydride must have an atmosphere of hydrogen of at least 10-20 mm over the lithium hydride in order to prevent decomposition of the hydride to lithium metal. Since nitrogen and lithium metal react at elevated temperatures its use as an inert gas should be avoided." (Ref. 19c(CRD))

b. Methods of casting

Welch reports the three methods investigated by General Electric as follows:

(1) Zone cooling

"The technique of 'zone-cooling' was used to prevent 'shrink pipe' formation by keeping a top of molten hydride on the solidifying mass, thus filling up the pipe as the hydride freezes and shrinks."
(Ref. 141f(CRD))

(2) Transfer casting

> "The technique of 'transfer' casting was investigated as a method of producing 'shrink-pipe' free castings where the casting container is large, or irregularly shaped, not readily adapted to a furnace.
>
> "The method consists of transferring molten lithium hydride from a reservoir to an unheated casting container using gravity flow or inert gas pressure to move the molten material from reservoir to container. Considerable difficulty was encountered by freezing of the hydride in the piping or at the entrance to the casting container, even where the piping was externally heated." (Ref. 141g(CRD))

(3) Zone melting

> "An apparatus for casting by 'zone-melting' was set up, consisting of a ten-foot length of six-inch pipe flanged at the top and closed at the bottom. Inside the pipe was placed a mold of five-inch tubing nine feet long filled with powdered lithium hydride. A tube furnace with an 18 inch long heating zone was placed around the external pipe and the charge heated until molten. The furnace was moved in one inch increments up the pipe at the rate of six inches per hour.
>
> "X-ray examination of the first casting showed only 30 inches of sound casting. The hydride had sintered above the melt zone and failed to feed properly. . . .
>
> "A solids-feeding valve was added to the set-up to feed weighed charges of powder as the melt progressed. A sound casting about four and one-half feet long was produced in this manner. A second casting (five feet long) made by this method was sound, except for one small void near the bottom and some small gas bubbles which appear in all castings of this type.
>
> ". . . it was believed the method was successful on a small scale, and could be scaled-up to produce larger castings without difficulty." (Ref. 141h(CRD))

c. Casting experience with lithium hydride and deuteride

Since lithium hydride and deuteride are essentially the same in physical properties related to handling and manufacturing, they are treated together in this section.

> "Zone-cooled methods produce castings of lithium hydride of approximately theoretical density and

> approximately 95-97% purity with some free lithium present in the crystals. Cast lithium hydride usually contains small quantities of metals such as chromium, iron, and nickel which have been leached from the container when the salt was in the molten state. Soluble salts such as the lithium oxide and lithium chloride tend to concentrate on top of the casting on cooling. It has been [reported] that small amounts of lithium oxide influence the corrosion rate. [See section V.D.]
>
> "Large castings which are virtually crack-free may be obtained by casting the lithium hydride into a mold containing a honeycombed matrix of stainless steel. [See figs. II-10 and II-11.] The stainless steel provides paths for removal of heat in the molten salt resulting in considerable reduction in cracks from shrinkage . . . ." (Ref. 19d(CRD))

Welch comments on the zone-cooling method at General Electric as follows:

> "To reduce the problem of machining cast lithium hydride, a reservoir type container was designed and fabricated . . . . A casting was made into a T-shaped container using the reservoir as a container of molten hydride during the freezing of the material in the can. The riser tube and hydride were cut off and a patch type weld made over the hole. This method proved very successful and was used in the fabrication of later castings . . . .
>
> "A slight modification in technique was developed to increase the number of castings which could be made using one melting furnace. The procedure consisted of melting the hydride in one furnace and transferring the casting, full of molten hydride, to a second furnace which was heated only at the top to keep the hydride in the reservoir molten.
>
> "The implication of this technique is that a complicated, zone-controlled furnace is not a necessary requirement for successful casting. A casting set-up could consist of a simple, uniformly heated furnace for melting the lithium hydride, and a simple, insulated cabinet with a heater for the reservoir, permitting one casting to be melting while a second casting is cooling." (Ref. 141i(CRD))

> "A facility for casting lithium-hydride-filled test specimens (up to 6 feet long) was installed at GE-ANPD. . . . [Specimens] 6 feet in length and extended-life test specimens 3 feet in length were prepared for casting.

Three castings of specimens 12 inches in diameter and 54 inches long were made . . ., and a group of 6-foot-long test specimens was cast . . . for thermal and mechanical evaluation.

"The castings produced . . . were of two types: (1) a 29-inch-long type to evaluate the effects of temperature cycling on the mechanical characteristics of the container - cooling-tube - lithium hydride system, and (2) a 36-inch-long type to obtain heat transfer data on finned and unfinned cooling tubes incorporated into cast lithium hydride." (Ref. 255(SRD))

"Nine lithium hydride castings in various shapes and sizes have been produced . . . by the reservoir-zone cooling technique. The castings contain from 25 to 275 pounds of material. . . .

"Experience gained in producing these castings has led to the conclusion that the design of the auxiliary heater and heater leads is quite critical in castings where the filler tube is not at or very near the vertical centerline of the container. Failure to provide auxiliary heat to the filler tube during the final stages of freezing invariably results in the formation of a void (shrink pipe) at the top of the casting when the filler tube is off-center.

"[A casting] was made to determine some of the problems associated with the production of large-volume castings. The container-reservoir assembly was designed to provide the largest possible casting that could be handled within the retort bell, which has a 24-inch diameter and is 54 inches high on the inside (usable height). Roughly 275 pounds of high-purity, granulated LiH was used to fill the container. The complete casting cycle required about 48 hours including heating and cooling. However, the 30-hour cooling cycle might have been shortened by about 10 hours had the auxiliary heaters functioned. The additional time was required to control the freeze-out of the top of the casting by slow manipulation of the Globar furnace temperature and position.

"The casting was instrumented with . . . thermocouples . . . placed within the primary container, filler tube, and reservoir and on the outside of the assembly to monitor the heating and cooling characteristics of the large-volume casting.

"The freezing pattern of the casting was as expected on the basis of experience with smaller castings, and even though the auxiliary heaters were inoperable (grounded),

> the central filler tube permitted complete filling of the container. X-ray examination revealed that the casting was free of a shrink pipe void, although a fairly large shrink crack (which is believed to be characteristic of large-cross-section castings) parallel to the central tube was observed.
>
> "The large flat ends (20 inches in diameter and 0.125 inch thick, made of type 304 stainless steel) were bulged upward. It has been postulated that temperature differentials between the cylinder wall and ends (the wall is cooler than the ends) produced the bulging, sometimes referred to as 'oil-can' effect." (Ref. 256(SRD))

Summarizing the General Electric casting experience, castings with cylindrical or hexagonal shapes with diameters from 3 to 20 inches have been made. The lengths varied from 6 to 36 inches, with most of the castings in the 29- to 36-inch range. Various cooling-tube configurations were cast into these blocks, such as straight axial, unfinned tubes (see ref. 135(U)) or spiral, finned tubes. The containers were made of one of the following stainless steels: 301, 304, or 316, with thicknesses of either 0.030, 0.031, or 0.125 inch. As mentioned previously, most of the problems encountered when handling molten lithium hydride were overcome (refs. 255(SRD), 256(SRD), and 257(SRD).

The following is a condensation of the casting experience reported in reference 253(U):

This was a laboratory operation to obtain small castings of uniform density. The casting cell was $\frac{1}{2}$ by 1 by $5\frac{1}{2}$ inches long, of 0.010-inch-thick stainless steel. A helium atmosphere was used so that the lithium hydride or deuteride, about 90 percent pure, would not be further contaminated. The mold was filled with powder, and then several cycles of selective melting, freezing, and refilling were carefully carried out so that the shrink pipe was eliminated.

The problem of "crawling" was encountered to the extent of about 2 centimeters on a clean stainless steel surface. On an oxidized surface the crawling was found to be about five times as great.

The lithium deuteride sample obtained was a dense aggregation of blue-white crystals on the lower end (which had been melted and frozen just once) and a blue-green glassy-appearing crystal on the upper end (which had been reheated). The density of the sample was 0.84 gram per cubic centimeter.

d. Miscellaneous

Thompson Ramo Wooldridge has done some work that produced interesting photographs and X-rays of lithium hydride after cooling from the liquid state. Figure V-3 is a photograph of a $1\frac{1}{2}$-inch-diameter container that was cut open after the lithium hydride had solidified. As can be seen, the lithium hydride adheres to the walls. It was found quite difficult to break it away from the walls by mechanical means.

Figures V-4 and V-5 show X-rays of a slab about 12 inches long, 6 inches wide, and 1/2 inch thick that was used for heat-transfer tests. Figure V-4 shows the pellets of compacted powder with the spaces between filled with loose powder prior to melting. Figure V-5 shows the cracks, surface contour, and void after melting and freezing. After each of several cycles of melting and freezing almost exactly the same pattern was evident (personal communication from E. G. Rapp, Thompson Ramo Wooldridge, Inc.).

6. Machinability

There is some experience in the machining of both cold-pressed and cast lithium hydride. The cold-pressed compacts "are easily machinable using carbide tipped bits with standard machine tools and tolerances of ±0.004 inch are held without difficulty." (Ref. 113e(SRD))

Castings are very hard to machine, as shown by the following:

> "Figure [V-6] shows the result of machining cast lithium hydride with a fly cutter. By reducing the cut to 0.050 inch, the material was machined with reasonable success.
>
> "Milling out of the hydride below the edge of the container was done using a small milling head and considerable care, although some hydride along the inside surface of the container still cracked loose and was thrown out." (Ref. 141i(CRD))

D. COMPATIBILITY AND CORROSION

Compatibility and corrosion information of a general nature involving lithium hydride can be found in items numbered 1, 2, and 8 to follow. Specific materials which have been observed after contact with lithium hydride are listed in alphabetical order in table V-III, with short comments and references to the numbered items, tables, and figures where the detailed data may be found.

1. Messer has this to say about the corrosion character of molten lithium hydride:

> "This matter has not been adequately investigated and results of various workers seldom agree. Probably, the reason for this lack of agreement lies in the considerable effect of impurities present in the hydride and of the cleanliness of the containers whose resistance is being ascertained. Moreover, the effect of even brief exposure of the hydride or container to air has sometimes been neglected.
>
> "Molten lithium hydride of high purity does not appear to attack Armco iron, 312 or 347 stainless steels at temperatures below 720°C. Vessels made from these materials have been used repeatedly in this laboratory and elsewhere without evidence of corrosion. A 347-alloy bomb contained molten lithium hydride for 150 hours at temperatures up to 1050°C. and showed, on metallographic examination, a roughened surface layer only 0.01 mm thick. . . .
>
> "The hydride showed a spectrographic trace of nickel but no chromium or iron. The latter may have been overlooked, however, owing to the very small amounts present.
>
> "The presence of impurities, including those due to exposure of lithium hydride to air, may profoundly affect the corrosive character. Lithium oxide and lithium chloride are extremely corrosive to most metals and ceramics. The former is a common result of undue exposure to air; the latter is present in most commercial lithium in varying amounts. The corrosive properties of other likely impurities have not been ascertained.
>
> "Vessels in which the hydride is to be melted should be scrupulously cleaned and pretreated with molten hydride and hydrogen. . . . Anaerobic technique is recommended for handling the hydride and the <u>cleaned container</u>. If the container is welded, special attention should be given to ensuring absence of pockets of flux. Atomic hydrogen welds are recommended. Neither mild steel nor silica are recommended for containers, despite favorable experience of others reported for molten lithium. . . .

[redacted]

> "The tendency of lithium to alloy with various metals is discussed in the "Liquid Metals Handbook" [ref. 82(U)] and in the older literature [ref. 258(U)]." (Ref. 2t,u(U))

2. The Olin Mathieson Chemical Corporation undertook a pilot program to produce lithium hydride continuously in large quantities (5 tons/day). (See section V.A.2.a.) As a result, experience was obtained in handling both lithium and lithium hydride. The portions of their reports which bear directly on the compatibility and corrosion problem are presented here.

> "Throughout the pilot plant operations, a considerable number of metallurgical failures occurred in process vessels and lines. . . . Equipment involved contained lithium and/or lithium hydride-dispersion oil-hydrogen gas slurries, normally at temperatures of 400°-550° F and at pressures of 0-30 p.s.i.g." (Ref. 239a(U))

Several metallurgical failures occurred early in the program, most of which were attributed to things other than corrosion, such as poor welding or lack of stress-relief.

> "Following the termination of the pilot plant operations, [a carbon steel vessel] was removed to obtain metallurgical samples. A total of twelve samples were taken . . .. The metallurgical report indicated decarburization to a slight degree in both the liquid and gas spaces, and also corrosion, cracking and pitting of the [vessel] wall and cracking at welds and nozzles. The samples submitted for hydrogen analysis indicated concentrations of 18-25 p.p.m. in the gas-space of the vessel and a concentration of 3-7 p.p.m. below the liquid level, near the bottom of the vessel." (Ref. 239a(U))

Reference 239b(U) concludes from this corrosion experience as follows:

> "The results indicated above become even more significant when it is considered that this vessel operated for a period of only 340 hours. In view of the short time the vessel was used, and the extensive metallurgical attack, the obvious conclusion was that carbon steel or stress-relieved carbon steel is not satisfactory for this service."

Lithium hydride usually has varying amounts of free lithium in it, and therefore the problems encountered in handling pure lithium will probably also apply to the handling of lithium hydride. Lithium

[redacted]

has been quite intensely studied as a possible heat-exchanger fluid for nuclear reactors, and hence a large amount of data (some of it classified) is available. Since this report is not concerned with lithium as such, a minimum of this information is presented. That from the Olin Mathieson Chemical Corp. follows:

"a. Corrosion by Lithium

[An experimental] unit was fabricated from mild steel, since it was considered acceptable in the 'Liquid Metals Handbook', 2nd edition [ref. 82(U)] for the planned operating conditions. After only 2 to 3 hours contact with molten lithium excessive intergranular attack was observed and process pipe lines fractured around threaded joints and other points of stress.

"Later corrosion tests indicated that lithium had a tendency to diffuse through mild steel; it leached carbon and other ingredients from the steel, thus causing it to fail. This has also been observed by other investigators. Albert and [Mahé, ref. 15(U)] stated that lithium diffused into steel at the rate of 2 mm. in four hours. Low-carbon or extra-low-carbon 304 stainless steel is now used for lithium service [at about 430° F].

. . . .

"b. Corrosion of Gaskets

"Gaskets were also affected by lithium. Darcoid and asbestos were found to react with lithium; they hardened sufficiently at elevated temperature (200-300°C) to allow atmospheric leaks into the system, thus causing lines to foul.

"The most suitable gasket for lithium service was found to be a Flexitallic Gasket. This gasket is constructed with alternate circular rings of stainless steel and blue asbestos (for sealing) and is reinforced with an outer ring (approximately 1/2 inch) of mild steel. Garlock (a form of asbestos) was later found to be suitable for lithium service for short periods at temperatures of 200-240°C. If either of these gaskets are [sic] used, then 300-p.s.i. flanges (minimum) should be used for jacketed pipe since a large applied force is required to make a gas-tight seal when flanged joints are used. Pipe flanges made for less than 300 p.s.i. service bend when bolts are tightened to make a gas-tight seal." (Ref. 237c(U))

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[redacted]

Olin Mathieson continues on corrosion tests with lithium as follows:

> "Tests were performed in an inert (argon) atmosphere and were continued for approximately 100 hours of exposure. Each metal was tested individually, thereby eliminating galvanic cell action between dissimilar metals. The liquid lithium was maintained at approximately 527°F, one specimen of the metal being immersed in lithium vapor and another half immersed in lithium liquid.
>
> "Metals which appeared to be satisfactory after the short term tests were cast iron and Types 316, 316LC, 321, 347, 416, and 446, Stainless Steel. Unsatisfactory metals included phosphorbronze, monel metal, mild steel, 304 stainless steel and ingot iron. The fact that ingot iron and 304 stainless steel exhibited intergranular attack is surprising as these two metals were used in laboratory work and appeared to be satisfactory. (See Table [V-IV]).
>
> "Type 304 stainless steel was used to handle the molten lithium and mild steel was used where slurries of the metal were encountered. No corrosion failures were observed.
>
> "Practical experience indicates, therefore, that the 300 series of stainless steels are most satisfactory for use in molten lithium service. The extra low carbon stainless steels (304 and 316) or type 347 should be used for field work and welding, because of the minimum danger of carbide precipitation at grain boundaries. This would minimize the danger of lithium attack on precipitated carbide which in turn would cause structural weaknesses." (Ref. 238c(U))

3. Some corrosion tests have been performed by the Materials Laboratory of the Thompson Ramo Wooldridge Corporation in Cleveland, Ohio. These are in conjunction with work being done to try to use lithium hydride as a thermal energy storage material. The initial work is reported as follows (refs. 85(U), 259(U), and 260(U):

Tubes of 347 stainless steel, with a 0.5-inch outer diameter and a 0.060-inch wall, were cut into 15-inch lengths and flattened at one end. The flattened end was inert-gas arc-welded, and the tube was filled with lithium hydride. The tubes were then placed in a vacuum and heated to 900° F to drive the $LiOH + LiH \rightarrow Li_2O + H_2$ reaction to completion. A tab of the material to be tested was then placed in the tube, and the open end of the tube was flattened and inert-gas arc-welded under vacuum. These sealed tubes were then placed in the furnace and cycled between 1200° and 1500° F, each complete cycle taking about 90 minutes.

There was excessive loss of hydrogen from the lithium hydride in the first series of capsule tests. Loss was so excessive that free

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lithium metal was found in some capsules. Corrosion of the capsule material was not as great as had been expected from work performed by earlier investigators. Diffusion of hydrogen through the container material appeared to be the main problem. The data are presented in tables V-V(a) to (f).

In other tests, Thompson Ramo Wooldridge (ref. 259(U)) was concerned with the compatibility of molten lithium hydride with good fin materials - those that would have low density and high thermal conductivity. A test was performed with graphite fins. The chemical reaction produced methane, hydrogen, and probably some lithium carbide. The fins disintegrated.

Further work done by Thompson Ramo Wooldridge (personal communication from E. G. Rapp) indicates that very little trouble should be expected with weld failures if extreme care is used during the welding operation. Figure V-7 shows photomicrographs of sections taken from a type-347 stainless steel container. This container had been charged with lithium hydride of unknown purity furnished by the Lithium Corporation of America. The temperature-time history of the unit is as follows: 3 hours at 1400° F, 20 hours at 300° F, 1 year at room temperature, cycling between 800° and 1600° F for a total number of five cycles giving an additional exposure of $4\frac{1}{2}$ hours to molten lithium hydride. The container was then opened in air with a hacksaw, and metallographic samples were taken. Metallographic preparation was carried out by conventional methods.

As shown in figure V-7, the attack appears to be intergranular, and the depth of penetration varied between about 0.0015 inch for the weld material to 0.003 inch for the wrought 347 stainless steel. A thin layer, assumed to be a corrosion product, was present as revealed by these photomicrographs. No identification of this layer was attempted.

4. The Sundstrand Turbo Co. did some work which concluded that, of the materials tested in the corrosion of liquid lithium hydride, molybdenum is the only test material investigated that appeared to withstand corrosive attack of molten lithium hydride at 1300° F. The materials included Hastelloy C, molybdenum, Inconel, Hastelloy N, and columbium.

It should be noted that the preceding information was from a limited test program, and that the low-carbon stainless steels were not tested (ref. 85(U)).

[redacted]

5. Mr. Hammill of the Union Carbide Co. (ref. 85(U)) presented the results of stress corrosion testing done in the 700° to 1100° F range for 1000 hours at stresses in the vicinity of 100,000 pounds per square inch. Three types of tests were performed:

(a) A tube specimen was filled with powdered lithium hydride, and the interior of the tube was kept at a positive pressure with 1 pound per square inch gage of dry hydrogen.

(b) A tube specimen was filled with powdered lithium hydride which was then melted and cooled.

(c) A "dogbone sheet" specimen was contained and stressed in lithium hydride which was under an oxygen-free nitrogen atmosphere with a positive pressure. When a little lithium chloride or lithium fluoride was added to the lithium hydride, the test results were improved slightly.

The interior finish of the tube specimens affected the results. The better the finish, the longer was the stress life of the tube. The exterior of the tubes was in air at atmospheric pressure. Wherever there were specimens that had been welded, poor results were obtained in the heat-affected zones of the weldments. None of the high-strength alloys (Haynes 25, Hastelloy C, Inconel X, etc.) showed any better results than those mentioned in the following table:

Material	Temperature, °F	Results
Inconel	700-900	Good
Stainless 301	700-900	Good; the best of all the materials tested at 1000° F
Stainless 316, low-carbon	700-900	No rupture in 1000 hr

6a. The results of tests by the General Electric Company with lithium hydride in contact with stainless steel follow:

"<u>SUMMARY</u>

"Solution heat-treated 19-9DL sheet (unwelded and welded) generally exhibited a slight decrease in ultimate tensile strength, 0.2 percent yield strength, and percent elongation after exposure to molten lithium hydride at

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[redacted]

1325 - 1425F for 65 - 100 hours, although the changes were essentially the same as those caused by exposure to heat and hydrogen alone.

"Metallographic examination defined the extent of attack to be slight with no definite indication of intergranular attack. A grain boundary precipitate was observed in the exposed specimens, although it is believed that the precipitate was the result of temperature rather than the corrosive condition.

"Weight change measurements and chemical analyses of the 19-9DL specimens and the lithium hydride confirmed that no appreciable attack occurred during these exposure conditions.

"CONCLUSIONS

"It is concluded that solution heat-treated 19-9DL sheet (unwelded and welded) is not seriously affected by exposure to molten lithium hydride and that the alloy is a useful structural material for the containment of molten lithium hydride at 1325 - 1425F for periods up to 100 hours."
(Ref. 261(U))

6b. Further tests by the General Electric Company with lithium hydride in contact with various stainless steels are reported in detail in reference 262(U). The abstract is as follows:

"The mechanical properties (ultimate tensile strength, 0.2 percent yield strength, and percent elongation) of unwelded and welded types 301, 304, 316, and 321 and welded 316 ELC, 317, 318, and 347 stainless steels were determined after exposure to molten lithium hydride at 1325 ±25°F for periods up to 100 hours. In general, the tensile and yield strengths were not markedly different from the control specimen values, although the percent elongation in all cases decreased. Metallographic examination showed negligible attack on all alloys after 100 hours exposure at 1325°F. Increasing the temperature to 1425°F produced attack (up to 0.004 inch) after 100 hours exposure. Type 321 stainless steel exhibited the largest amount of damage. Weight change measurements showed insignificant losses at all exposure conditions."
(Ref. 262(U))

7. The Pratt & Whitney Division did some work on the compatibility of lithium hydride with NaK, which is reported as follows:

"Pressed lithium hydride specimens were found to be completely compatible with NaK after 500 hours at 500F in

[redacted]

static capsule tests. The pellet surfaces were wetted by an adherent coat of NaK but penetration into the specimen was less than 30 mils. The specimens gained an average of six percent in weight after NaK immersion." (Ref. 112(CRD))

"Compatibility tests of lithium hydride in lithium and organic fluids contained in type 316 stainless steel capsules have been extended to 260 hours at 500F. . . . There were no visual corrosion or penetration effects in any of the tests except those in lithium at 500F. In these latter tests, the pellets were pitted to 10 to 20 mils. [The results of these tests are shown in Table V-VI.]" (Ref. 263(CRD))

8. Battelle Memorial Institute reports on its handling problems, especially regarding "creeping" up container walls, as follows:

> "Since liquid LiH has a tendency to creep out of containing vessels, an effort was made to find a material of construction which would decrease the creep to a minimum. Boats of tantalum, molybdenum, Types 304 and 347 stainless steel were tested, but creep occurred in all cases. In an effort to determine if the oxide coating on the boats was responsible for the creep, the stainless steel boats were loaded again with LiH and another run made. It appeared that even more of the hydride creeped out of the boats during this second run than in the initial firing.
>
> "All the materials tested seemed to withstand the corrosive effects of LiH sufficiently. Tantalum was darkened somewhat but did not seem to be otherwise affected.
>
> "Since it was not possible to contain the LiH in a conventional boat, a new scheme was tried. A stainless steel tube was selected for a container. The tube was of sufficient length so that the ends would at all times remain at a temperature below the melting point of LiH. By filling the tube with the hydride along its entire length, it was possible to contain the liquid LiH within the central portion of the tube and the solid LiH actually formed the boat which protected the ceramic portion of the apparatus. Preliminary tests indicated that this arrangement was quite feasible." (Ref. 264(SRD))

9. Tannenbaum and Ellinger (ref. 140(U)) report that lithium hydride very rapidly corrodes fused quartz at elevated temperatures. This was discovered when fused quartz was used to contain powdered lithium hydride for X-ray diffraction studies.

E. AVAILABILITY

The following companies are known to produce lithium hydride:

Foote Mineral Company, Philadelphia, Pennsylvania
Lithium Corporation of America, Bessemer City, North Carolina
Maywood Chemical Works, Maywood, New Jersey
Metal Hydrides, Inc., Beverly, Massachusetts - a secondary producer, carrying out the hydriding operation only
Fairmount Chemical Company, Newark, New Jersey
Bios Lab Inc., 17 W. 60th St., New York 23, N. Y. (small quantities chemically pure)

Lithium hydride is usually furnished in powder form, but lump form is also available.

Messer describes the impurities present in the commercial product as follows:

> "The commercial lithium metal and the hydride prepared therefrom are sometimes contaminated with carbon, as graphite or as Li_2C_2, by a brownish material which occurs in marble-like veins (in the metal), by other alkali metals, chloride, and by the products resulting from exposure to air (oxide, hydroxide, carbonate). The commercial hydride will ordinarily contain the same impurities although not in the same proportion. Thus, some of the alkali metals are removed by the 700° C. treatment with hydrogen, distilling out of the charge as the lithium is hydrided. The commercial hydride is usually gray and resembles calcium carbide or slate in appearance. Part of this coloration may be due to traces of colloidal metal or F-centers. Analyses (by hydrogen evolution) usually run over 98% LiH. Since the evolution method of analysis is not sensitive to lithium content, up to 3 or 4% lithium metal may be present. . . . So far as may be ascertained, vacuum-distilled lithium is not used in manufacturing commercial hydride, nor is vacuum-distilled lithium metal available through ordinary commercial channels." (Ref. 2c(U))

See also section I.C. ANALYSIS.

In recent months 99.4 percent pure lithium hydride has been produced for commercial use in small quantities, with a guaranteed purity of at least 99 percent (personal communication with Mr. V. I. Purcell, Foote Mineral Co.).

The Lithium Corp. of America (ref. 117b(U)) gives the following information:

> "Lithium hydride is available in large commercial quantities with the following abundance of particle sizes:
>
> | | | | |
> |---|---|---|---|
> | Class VI | -35 mesh | 100% | approximately |
> | | -60 mesh | 90% | approximately |
> | | -100 mesh | 80% | approximately |
> | | -150 mesh | 65% | approximately |
> | | -200 mesh | 45% | approximately" |
>
> "The LiH assay, based on the hydrogen yield on reaction with water, ranges from 93 to 98%. The assay of the finer grinds is usually lower than that of the coarse material. The guaranteed assay of . . . Class VI is 93%." (Ref. 117(U))

Messer (ref. 2c(U)) states that "assays of the commercial material run from 95 to over 98% LiH."

During a panel discussion at a meeting on LiH (ref. 85(U)) it was brought out that 99 percent pure material can be produced if desired. The majority of users at the present time do not require this purity; hence, it is not commonly available. It was suggested that a user who needs excellent purity should deal directly with the producer to obtain such a sample.

It was the general consensus of opinion at this meeting (ref. 85(U)) that it is much easier and more satisfactory to see that the lithium and hydrogen are pure before hydrogenation of the lithium. It is very difficult to remove impurities from the compound after it is formed.

Lithium hydroxide changes to lithium oxide and hydrogen at approximately 880° F, and Waldrop (ref. 85(U)) stated that reduction of lithium oxide takes place in the presence of iron at high temperatures. Thus these two impurities can be removed from the compound if necessary.

The price in year 1961 of the commercially available product is in the range of $9.00 to $12.00 per pound, depending on the quantity and purity desired.

F. TOXICITY

The summary and conclusions of Spiegl, et al. are as follows:

> "Lithium hydride in atmospheric concentrations of from 5 to 55 mg./cu.m. is an intensely irritating and corrosive material. Its action, however, can be ascribed to the alkalinity of the hydrolysis product, and is in no way characteristic of the lithium ion <u>per se</u>.
>
> "Mortality among rats, rabbits, guinea pigs, and mice exposed to levels of from 5 to 55 mg.LiH/cu.m. for one day, or to 5 mg.LiH/cu.m. for one week was low and probably unrelated to the exposure.
>
> "No chronic effects were observed during a postexposure period of up to 5 months.
>
> "There is no evidence that inhaling work area atmospheres containing 25 μLi/cu.m. would constitute a toxic hazard." (Ref. 265(U))

Aitken and Tetenbaum have the following to say about toxicity:

> "Because of the extreme degree of irritation produced by the inhalation of lithium hydride, it appears unlikely that a sufficiently high concentration of lithium hydride could be breathed to constitute a hazard . . . . Existing tolerance values for strong bases should probably apply for lithium metal, lithium hydride, and lithium hydroxide. The build up of tritium due to neutron capture must also be considered as a health hazard." (Ref. 19e(CRD))

TABLE V-I. - RELATION OF PARTICLE-SIZE DISTRIBUTION OF POWDER TO DENSITY AND WEIGHT LOSS OF HOT-PRESSED COMPACT (Ref. 254(U))

Run	Particle size, mesh	Density, g/cc	Weight loss, %
1	-10 +20	0.736	8
2	-10 +35	.736	7
3	-10 +50	.736	7
4	-10 +80	.748	7
5	-10 -80	.767	8
6	-20 +80	.766	9
7	-35 +80	.775	10
8	-35 +100	.733	6.7
9	-20 +100	.737	7
10	-10 +100	.736	8

TABLE V-II. - MOLD SURFACE TREATMENT RELATED TO COMPACT DENSITY AND WEIGHT LOSS OF LITHIUM HYDRIDE (Ref. 254(U))

Run	Metal plate	Compact analyses, %		Compact density, g/cc	Weight loss, %
		Hydride, internal (a)	Hydride, external (a)		
XH-37	Thin Fe, 1-1.5 mils (std.)	93.49	93.52	0.790	0.1
XH-40	Thin Fe (prefired)	93.81	93.83	.789	.8
XH-41	Thick Fe, 2-3 mils	93.49	93.75	Piece cracked	
XH-42	0.1 mil Cr on 1-2 mils Fe	93.70	94.45	.779	.2
XH-43	Ag, 1-2 mils	92.70	93.00	.797	1.9
XH-44	0.2 mil Au on 0.8 mil Ag	93.56	89.34	.789	1.9
XH-45	0.01 mil Rh on 0.2 Ni, 0.8 Fe	93.66	93.54	.783	.04

[a] The external samples were taken from the outer 3/16 in. of the compacts and the internal samples from the centers.

TABLE V-III. - MATERIALS WITH WHICH LITHIUM HYDRIDE HAS BEEN TESTED FOR COMPATIBILITY AND CORROSION

Material	Item number in text, section V.D.	Table number	Comments (regarding reaction with lithium hydride unless otherwise stated)
AISI 1010 steel		V-V(a)	Corrosion observed after 264 hr at 1165° to 1600° F
Alkylbenzene (250)	7	V-VI	Some reaction
Argon		V-VI	No reaction
A-286		V-V(a)	Carbon depletion and oxidation after 503 hr at 1165° to 1600° F
Beryllium		V-V(a), V-V(e), V-V(f)	65 percent of Be lost after 529 hr at 1165° to 1600° F
Columbium	4	----------	Unsatisfactory at 1300° F
Gaskets:			
1. Darcoid and asbestos	2	----------	Reacted with lithium, hardened sufficiently at 200° to 300° C to cause leaks
2. Flexitallic	2	----------	Most suitable gasket found for lithium service
3. Garlock (a form of asbestos)	2	----------	Suitable for short periods at 200° to 240° C in lithium
Graphite	3	----------	Chemical reaction produced methane, hydrogen, and probably some lithium carbide; the graphite disintegrated
Hastelloy C	4	----------	Unsatisfactory at 1300° F
	5	----------	Not any better than Inconel, stainless 301 or 316LC for 1000 hr at 100,000 psi and 700° to 900° F
		V-V(a) to V-V(f)	Carbon depletion and oxidation after 503 hr at 1165° to 1600° F
Hastelloy N	4	----------	Unsatisfactory at 1300° F
Hastelloy X		V-V(a)	Carbon depletion after 501 hr at 1165° to 1600° F
Haynes 25		V-V(a) to V-V(f)	Slight reaction after 503 hr at 1165° to 1600° F
	5	----------	Not any better than Inconel, stainless 301, or 316LC for 1000 hr at 100,000 psi and 700° to 900° F
Inconel	4	----------	Unsatisfactory at 1300° F
		V-V(a)	Carbon depletion and grain boundary corrosion after 1005 hr at 1165° to 1600° F
	5	----------	Very little stress corrosion at 100,000 psi and 700° to 900° F for 1000 hr
Inconel X	5	----------	Not any better than Inconel, stainless 301, or 316LC for 1000 hr at 100,000 psi and 700° to 900° F
		V-V(a) to V-V(f)	Weight gain after 555 hr at 1165° to 1600° F

TABLE V-III. - Continued. MATERIALS WITH WHICH LITHIUM HYDRIDE HAS BEEN TESTED FOR COMPATIBILITY AND CORROSION

Material	Item number in text, section V.D.	Table number	Comments (regarding reaction with lithium hydride unless otherwise stated)
Iron, Armco	1	----------	No objectionable corrosion below 720° C
Iron, cast	 2	V-IV ----------	No corrosion reported Satisfactory after 100-hr test in lithium at 527° F
Iron, ingot	 2	V-IV ----------	Considerable corrosion (like mild steel) Unsatisfactory after 100-hr test in lithium at 527° F
Lithium		V-VI	Some reaction
Molybdenum	4 8	---------- V-V(a) ----------	Better than Hastelloy C or N, Inconel, and columbium at 1300° F Intergranular corrosion observed after 1004 hr at 1165° to 1600° F; excellent hydrogen retention No reaction reported
Monel	2	V-IV	Unsatisfactory after 100-hr test in lithium at 527° F
Organic fluids: see Santowax and alkylbenzene (250)	7	V-VI	Some reaction
Phosphorbronze	2	V-IV	Unsatisfactory after 100-hr test in lithium at 527° F
Quartz (fused)	9	----------	Corrodes very rapidly at elevated temperatures
Santowax R	7	V-VI	Some reaction
Silica	1	----------	Not recommended for use in contact with lithium hydride
Sodium-potassium alloy	7	----------	Completely compatible after 500 hr at 500° F
Stainless 19-9DL	6a	---------- V-V(a)	Satisfactory for containing molten lithium hydride for up to 100 hr at 1325° to 1425° F Some corrosion after 500 hr at 1165° to 1600° F
Stainless 301	5 6b	---------- ----------	Very little stress corrosion at 100,000 psi and 700° to 900° F for 1000 hr; better than Inconel, stainless 316LC, Haynes 25, Hastelloy C, and Inconel X at 1000° F Very little corrosion or other effects after 100 hr at 1325° F

TABLE V-III. - Continued. MATERIALS WITH WHICH LITHIUM HYDRIDE HAS BEEN TESTED FOR COMPATIBILITY AND CORROSION

Material	Item number in text, section V.D.	Table number	Comments (regarding reaction with lithium hydride unless otherwise stated)
Stainless 304	2		Low-carbon or extra-low-carbon 304 is used for lithium service at about 430° F
		V-IV	Intergranular attack after 105 hr at 250° C
	2	----------	Unsatisfactory after 100-hr test in lithium at 527° F, yet was satisfactory for handling molten lithium slurries
	8	----------	No reaction reported
	6b	----------	Very little corrosion or other effects after 100 hr at 1325° F
Stainless 310		V-V(a)	Some corrosion after 501 hr at 1165° to 1600° F
Stainless 312	1	----------	No objectionable corrosion below 720° C
Stainless 316		V-V(a) to V-V(f)	Some corrosion after 1002 hr at 1165° to 1600° F
		V-IV	Some corrosion
	2	----------	Satisfactory after 100-hr test in lithium at 527° F
Stainless 316LC	2	----------	Satisfactory after 100-hr test in lithium at 527° F
		V-IV	Some corrosion
	5	----------	No rupture in 1000 hr during stress-corrosion testing at 100,000 psi and 700° to 900° F
Stainless 316ELC		V-V(a)	Slight corrosion after 503 hr at 1165° to 1600° F
Stainless 316ELC, welded only	6b	----------	Very little corrosion or other effects after 100 hr at 1325° F
Stainless 317, welded only	6b	----------	Very little corrosion or other effects after 100 hr at 1325° F
Stainless 318, welded only	6b	----------	Very little corrosion or other effects after 100 hr at 1325° F
Stainless 321		V-IV	Some corrosion
	2	----------	Satisfactory after 100-hr test in lithium at 527° F
	6b	----------	Very little corrosion or other effects after 100 hr at 1325° F
		V-V(a)	Some corrosion after 501 hr at 1165° to 1600° F

[redacted]

TABLE V-III. - Concluded. MATERIALS WITH WHICH LITHIUM HYDRIDE HAS BEEN TESTED FOR COMPATIBILITY AND CORROSION

Material	Item number in text, section V.D.	Table number	Comments (regarding reaction with lithium hydride unless otherwise stated)
Stainless 347	1	----------	No objectionable corrosion below 720° C; for 150 hr at temperatures to 1050° C, the surface was roughened to a thickness of 0.01 mm
		V-V(a) to V-V(f)	Very slight reaction after 500 hr; some corrosion after 1001 hr at 1165° to 1600° F
	3	Fig. V-7	Intergranular attack when exposed to LiH varied between 0.0015 in. for weld material to 0.003 in. for wrought 347 SS. The temperature-time history was: 3 hr at 1400° F, 20 hr at 300° F; 1 yr at room temperature, five cycles between 800° and 1600° F for $4\frac{1}{2}$ hr
		V-IV	No corrosion reported
	2	----------	Satisfactory after 100 hr test in lithium at 527° F
	8	----------	No reaction reported
Stainless 347, welded only	6b	----------	Very little corrosion or other effects after 100 hr at 1325° F
Stainless 410		V-IV	Some corrosion
Stainless 416	2	----------	Satisfactory after 100-hr test in lithium at 527° F
Stainless 446		V-IV	Some corrosion
	2	----------	Satisfactory after 100-hr test in lithium at 527° F
	3	V-V(a)	Some corrosion after 500 hr at 1165° to 1600° F
Steel, carbon	2	----------	Not satisfactory for service with lithium or lithium hydride - dispersion oil-hydrogen gas slurries at temperatures of 400° to 550° F and at pressures of 0 to 30 lb/sq in. gage
Steel, mild	2	V-IV	Unsatisfactory after 100-hr test in lithium at 527° F; satisfactory to handle molten lithium where slurries of the metal were handled
	2	----------	Subject to excessive intergranular attack when in contact with lithium; lithium diffuses through mild steel at a rate of about 2 mm in 4 hr
	1	----------	Not recommended for use in contact with lithium hydride
Tantalum	8	----------	Darkened somewhat but did not seem to be otherwise affected by molten lithium hydride (time unknown)

[redacted]

TABLE V-IV. - CORROSION TESTS WITH LITHIUM HYDRIDE (Ref. 238(U))

Material	Temperature, °C	Exposure, hr	Corrosion rate, mils/year		Remarks
			LiH liquid	LiH vapor	
304 SS	250	105	0.09	0.13	Intergranular attack observed on immersed sample
316 SS	275	89	.55	.38	Both samples satisfactory
316LC SS	270	113	.40	.51	No intergranular attack visible
410 SS	270	89.5	.58	.80	No intergranular attack visible
446 SS	260-270	89	.25	.06	No intergranular attack visible
321 SS	270	89	.12	.38	No intergranular attack visible
347 SS	270	89	----	----	No intergranular attack visible; bent 90° with no loss of ductility
Mild steel	270	114	.92	.78	Intergranular attack visible for both samples; liquid sample cracked visibly when bent 90°; vapor sample did not
Cast iron	270	64	----	----	No corrosion visible for either sample when examined under microscope
Ingot iron	270	138	.57	.12	Same as mild steel results
Ingot iron	270	64.5	----	----	Intergranular attack visible for both samples; neither cracked upon bending; fresh lithium was used
Monel					Unsatisfactory
Phosphorbronze					Unsatisfactory

TABLE V-V. - LITHIUM HYDRIDE CONTAINMENT TEST

(a) Summary of results on single metal test capsules (Refs. 260(U), 266(U), and 267(U))

Material	Capsule number	Time of exposure, hr	Number of cycles between 1165° and 1600° F	Weight change of capsule, %	Lithium hydride after test, %	Cleaning procedures	Mode of corrosion	Depth of corrosion, in.	Remarks
310 SS	31 to 34	501	290	+0.10 to -0.27	Yes	C or C-Va	----------	----------	----------
316 SS	[a]4 to 7	116 to 529	70 to 312	-0.018 to -0.49	91.3(529 hr)	----------	----------	----------	----------
	38, 40	501	290	+0.02 to -0.44	Yes	C-Va	----------	----------	----------
	55, 56, 57, 63, 65, 80	500	310	-0.41 to -0.44	0.8 to 6.1	Va or C	----------	----------	End-cap type, argon gas
	62, 64	1002	621	-0.42 to -0.43	2.3 to 2.8	C	Grain boundary penetration	<0.001	----------
	69 to 72	503	311	-0.004 to -0.42	4.1 to 4.9	C or H_2	Grain boundary penetration	<0.001	Oxidized thermocouple well
	84	501	292	-0.31	----------	H_2	----------	----------	
	87	503	312	-0.23	65.8	C	Grain boundary penetration	0.0025	Aluminum dipped
316ELC SS	161 to 164	503	312	-0.31 to -0.36	7.3 to 24.6	C	Grain boundary penetration	<0.001	Test continued
321 SS	25, 26, 28 to 30, 42	501	290	+0.12 to -0.23	Yes	C or C-Va	----------	----------	----------
347 SS	[a]1, 3	290	160	0.051	----------	----------	----------	----------	----------
	23, 24, 21	501	290	+0.07 to -0.56	16.2	C-Va	----------	----------	----------
	22	1001	600	-0.39	1.7	C	----------	----------	----------
	88 to 92		291			C or Va or H_2	Intergranular on Hastelloy W		Capsule 88 had electrolytic chrome plate
	95 to 97	498 to 501	292	+0.015 to -0.50	1.29 to 3.90		Weld only	0.004	
	93	501	292	-0.83	43.20	C	None	----------	"Solaramic" surface
	94	503	312	-0.05	----------	C	----------	----------	Glass-coated
	141 to 143	503	312	-0.26 to -0.29	10.3 to 16.2	C	----------	----------	Aluminum-dipped
446 SS	35 to 37, 41	500	310	-0.01 to -0.42	1.1 to 1.8	C or H_2	----------	----------	----------
19-9DL SS	58 to 61	500	310	+0.02 to -0.36	2.5 to 5.9	C or H_2	----------	----------	*Welded slots using 19-9W Mo filler rod*
Haynes 25	12, 13, 14	173	100	-0.179	90.76	----------	----------	----------	----------
	43 to 45	501	290	+0.01 to -0.22	Yes	C or C-Va	----------	----------	Preoxidized
	102 to 105	501	292	+0.003 to -0.29	21.3 to 23.0	C or Va or H_2	None	----------	Electrolytic chrome plate on 102 and 103
	155, 157	503	312	-0.17, -0.22	40.0, 53.7	C	Grain boundary penetration	<0.001	Etched, welds of Hastelloy W and 19-9 attacked
AISI 1010	81 to 83	264	161	-0.03 to -0.48	1.9 to 3.7	C	----------	----------	----------
Molybdenum	47, 48, 50, 68	503	311	-0.07 to -0.105	66.2 to 77.9	C	Intergranular (end cap and weld only)	0.005 end cap 0.002 weld	Argon, end caps welded
	49, 51	1004	603	-0.15	40.7 to 45.5	C	Intergranular	0.0075	Attack on end cap only
A-286	73 to 76	503	311	+0.005 to -0.42	2.5 to 3.8	C or H_2	Carbon depletion	0.001	Oxidized
Hastelloy C	[a]15, 17	555	327	0.052 to -0.226	84.76	----------	----------	----------	----------
	77 to 79	503	311	-0.40 to -0.57	1.9 to 2.5	C or H_2	Carbon depletion	0.0015 to 0.005	Oxidized
Hastelloy X	98, 99, 101	501	292	-0.41 to -0.43	1.96 to 2.96	C	Carbon depletion	0.0025	----------
Inconel	99	1005	604	-0.40	4.3	C	Grain boundary penetration	0.0025	Carbon depletion
316 SS + Mo insert	39	501	290	-0.30	Yes	C-Va	----------	----------	----------
316 SS + Haynes 25	67	1002	621	-0.40	2.3	C	Haynes 25 - grain boundary penetration 316 SS - carbon depletion	<0.001 0.003	Bimetallic
316 SS + Haynes 25	151, 152	503	312	-0.30, -0.31	15.11	C	None	----------	Haynes 25 on top
316 SS + Mo insert	153	503	312	-0.27	17.8	C	Haynes 25 - etched 316 SS - deposit	<0.001 <0.001	316 SS on top
316 SS + Haynes 25 + Mo - 1/2 Ti insert	154	503	312	-0.25	35.4	C	Haynes 25 - none 316 SS - deposit Insert - grain boundary penetration	0.001	316 SS on top
Inconel X	[a]11, 20	555	327	0.045 to 0.137	73.79	----------	----------	----------	----------
316 SS + Be	[a]18	529	312	-1.75	59.22	----------	----------	----------	65% of Be gone
347 SS + Be	[a]19	290	160	0.066	----------	----------	----------	----------	52% of Be gone

[a] These samples contained a charge of 4.6 g of LiH, while all others contained 3.6 g of LiH.

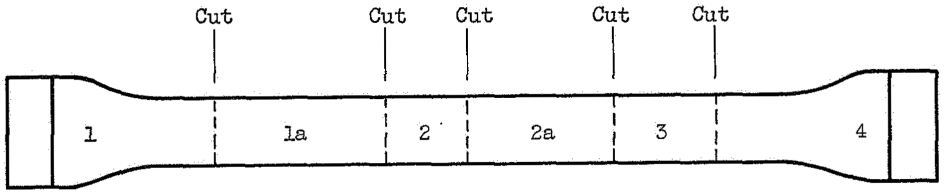
TABLE V-V. - Continued. LITHIUM HYDRIDE CONTAINMENT TEST

(b) Composition of capsule materials
(Data obtained from E. J. Vargo and D. B. Cooper, Thompson Ramo Wooldridge, Inc.)

Material	Composition, weight percent													
	C	Mn	Si	Cr	Ni	Mo	Cu	Cb/Ta	Al	Cb	Fe	Ti	W	Co
Type 347 SS	0.042	1.65	0.56	18.00	12.34	0.14	0.16	0.637	----	----	Bal.	----	-----	----
Type 316 SS	.05	1.69	.51	17.60	12.49	2.16	----	-----	----	----	Bal.	----	-----	----
Inconel X	.06	.62	.29	15.10	Bal.	-----	----	-----	0.95	0.71	6.86	2.52	-----	----
Haynes 25	.09	1.74	.50	20.05	11.01	-----	----	-----	----	----	2.14	----	15.23	Bal.
Hastelloy C	.06	.66	.55	15.60	Bal.	15.11	----	-----	----	----	6.91	----	4.16	2.64

TABLE V-V. - Continued. LITHIUM HYDRIDE CONTAINMENT TEST

(c) Ultrasonic thickness tester measurements of container
(Data obtained from E. J. Vargo and D. B. Cooper, Thompson Ramo Wooldridge, Inc.)



Capsule with its sections shown

Capsule material	Capsule number	Wall thickness readings, mils											
		Lithium hydride zone						Above lithium hydride zone					
		Before test		After test		Change		Before test		After test		Change	
Capsule section		1	2	1	2	1	2	3	4	3	4	3	4
Type 347 SS	1	65.0	66.8	64.8	64.0	-0.2	-2.8	63.8	64.9	63.9	64.5	0.1	-0.4
Type 347 SS	3	65.0	64.0	64.8	63.8	-.2	-.2	64.8	63.5	64.6	63.6	-.2	.1
Type 347 SS	19	65.6	65.4	67.2	66.1	1.6	.7	63.8	63.7	64.1	65.6	.3	1.9
Type 316 SS	5	66.2	67.0	61.9	62.7	-4.3	-4.3	67.1	66.1	62.7	62.0	-4.4	-4.1
Type 316 SS	6	67.2	66.5	61.2	61.9	-6.0	-4.6	67.2	67.1	61.8	61.5	-5.4	-5.6
Type 316 SS	7	65.6	67.8	62.0	64.0	-3.6	-3.8	65.2	68.5	61.5	64.2	-3.7	-4.3
Type 316 SS	18	64.7	68.8	60.9	61.0	-3.8	-7.8	65.1	69.3	61.0	61.1	-4.1	-8.2
Inconel X	11	64.2	65.0	65.3	66.1	1.1	1.1	64.3	65.7	65.1	66.0	.8	.3
Inconel X	20	63.5	65.5	64.5	66.2	1.0	.7	63.4	65.9	64.2	66.0	.8	.1
Haynes 25	12	68.1	68.2	68.2	67.4	.1	-.8	67.9	68.3	67.5	68.2	-.4	-.1
Haynes 25	14	67.9	67.8	68.2	68.0	.3	.2	68.0	68.0	67.9	67.5	-.1	-.5
Hastelloy C	15	59.5	59.7	55.7	55.5	-3.8	-4.2	59.8	59.5	54.9	55.5	-4.9	-4.0
Hastelloy C	17	58.2	59.8	55.0	54.5	-3.2	-5.3	59.5	59.9	53.8	54.3	-5.7	-5.6

TABLE V-V. - Continued. LITHIUM HYDRIDE CONTAINMENT TEST

(d) Summary of microhardness readings on single metal test capsules
(Data obtained from E. J. Vargo and D. B. Cooper, Thompson Ramo Wooldridge, Inc.)

Capsule material	Capsule number	Section of capsule (see table V-V(c))	Microhardness, KHN at distance below surface of capsule material, in.				Remarks and exposure times
			0.0005	0.001	0.003	0.010	
Type 347 SS	--	-	176	204	219	211	Before test
	1	2	120	165	180	185	290 hr
	3	2	120	190	200	190	Blank - 290 hr
Type 316 SS	--	-	139	151	161	158	Before test
	4	2	180	175	200	200	290 hr
	5	2	141	163	215	229	116 hr
	5	3	132	171	211	219	116 hr
	6	2	135	160	190	173	529 hr
	6	3	114	150	170	208	529 hr
	7	2	142	163	177	177	Blank - 529 hr
Inconel X	--	-	244	249	244	258	Before test
	11	2	232	277	310	310	Blank - 555 hr
	20	2	---	---	333	359	555 hr
	20	3	170	255	345	317	555 hr
Haynes 25	--	-	279	305	344	344	After heat treatment
	12	2	282	403	412	356	173 hr
	12	3	316	370	382	394	173 hr
	13	2	235	300	330	330	32 hr
	14	2	300	416	413	403	Blank - 173 hr
Hastelloy C	--	-	253	268	235	227	After heat treatment
	15	2	---	---	333	363	Voids - 555 hr
	15	3	---	305	355	363	Voids - 555 hr
	17	2	355	355	355	413	Blank - 555 hr

TABLE V-V. - Continued. LITHIUM HYDRIDE CONTAINMENT TEST

(e) Spectrographic analyses of lithium hydride samples
(Data obtained from E. J. Vargo and D. B. Cooper, Thompson Ramo Wooldridge, Inc.)

LiH from capsule number	Capsule material	Impurities			
		Appreciable	Minor	Trace	Faint trace
--	Starting LiH			Al, Mg, Si	B, Ca, Cu, Fe, Mn
1	Type 347 SS		Cr, Cu, Ni	Cb, Fe, Si	Al, B, Ca, Mg, Mn, Mo
19	Type 347 SS + Be insert	Be	Cr, Fe	Ni, Si	Al, B, Ca, Cu, Mg, Mn, Mo, V
[a]19-1	Type 347 SS + Be insert	Be	Cu, Fe, Mn, Ni, Si	Cr, Li, Ti, V	
4	Type 316 SS		Cr, Fe, Mn, Mo, Ni	B, Si	Al, Ca, Cu, Mg, V
5	Type 316 SS		Cr, Fe, Ni	Mo, Si	Al, B, Ca, Cu, Mg, Mn, V
6	Type 316 SS		Cr, Ni	B, Ca, Cu, Mg, Mo, Si, V	Fe
18	Type 316 SS + Be insert		Be, Cr, Cu, Fe, Mo, Ni	Ca, Mg, Mn	
20	Inconel X		Cu, Ni		Cr, Fe, Mn, Si
12	Haynes 25		Ni	Ca, Co, Cu, Mg, Si	Cr, Fe, W
15	Hastelloy C		Ni	Ca, Cu, Mg, Mo, V	Cr, Co, Fe, Si, W

[a] Corrosion product from beryllium insert.

TABLE V-V. - Concluded. LITHIUM HYDRIDE CONTAINMENT TEST

(f) Chemical analyses of LiH samples
(Data obtained from E. J. Vargo and D. B. Cooper,
Thompson Ramo Wooldridge, Inc.)

LiH from capsule number	Capsule material	Analysis, weight percent							
		LiH	Be	Ni	Fe	Cr	Mo	Mn	N_2
--	Starting LiH	98.45	------	-----	-----	------	------	------	0.066
1	Type 347 SS	------	------	1.52	0.90	>0.01	------	<0.01	------
19	Type 347 SS + Be insert	------	1.00	<.01	3.11	>.01	------	.01	------
[a]19-1	Type 347 SS + Be insert	------	90.0	9.20	<.01	<.01	------	<.01	------
4	Type 316 SS	------	------	.94	2.98	.44	<0.01	<.10	------
5	Type 316 SS	------	------	.69	.88	>.01	------	.01	------
6	Type 316 SS	91.30	------	1.73	>.01	<.01	------	<.01	.279
18	Type 316 SS + Be insert	59.22	1.05	<.01	1.88	<.01	<.01	<.01	.062
20	Inconel X	73.79	------	6.11	<.01	<.01	------	<.01	1.32
12	Haynes 25	90.76	------	1.50	>.01	<.01	------	<.01	.800
15	Hastelloy C	84.76	------	5.21	<.01	<.01	------	<.01	.572

[a] Corrosion product from beryllium insert.

TABLE V-VI. - RESULTS OF COMPATIBILITY TESTS OF LITHIUM HYDRIDE AND COOLANT IN STAINLESS 316 CAPSULES (Ref. 263(CRD))

Coolant	Temperature, °F	Time, hr	Weight change, percent (assumed to be of lithium hydride)
Lithium	400	160	+10
	400	260	+14
	500	160	Broken
	500	260	Broken
Santowax R	400	160	+12
	400	260	+11
	500	160	Broken
	500	260	+10
Alkylbenzene (250)	400	160	+6
	400	260	+7
	500	160	+11
	500	260	+8
Argon	400	260	0
	500	260	0

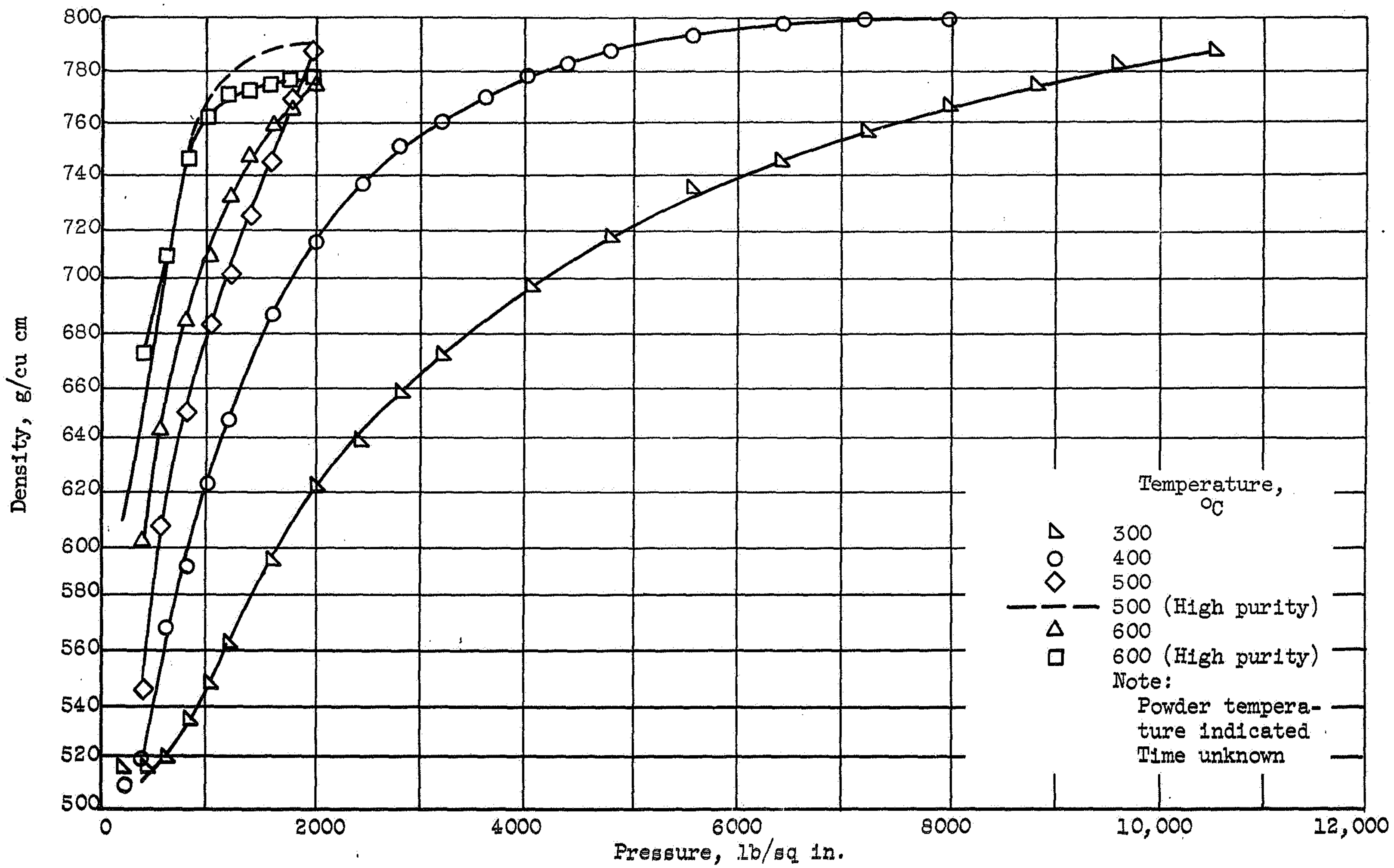


Figure V-1. - Density of compacted lithium hydride as a function of pressing pressure at various temperatures (ref. 254(U)).

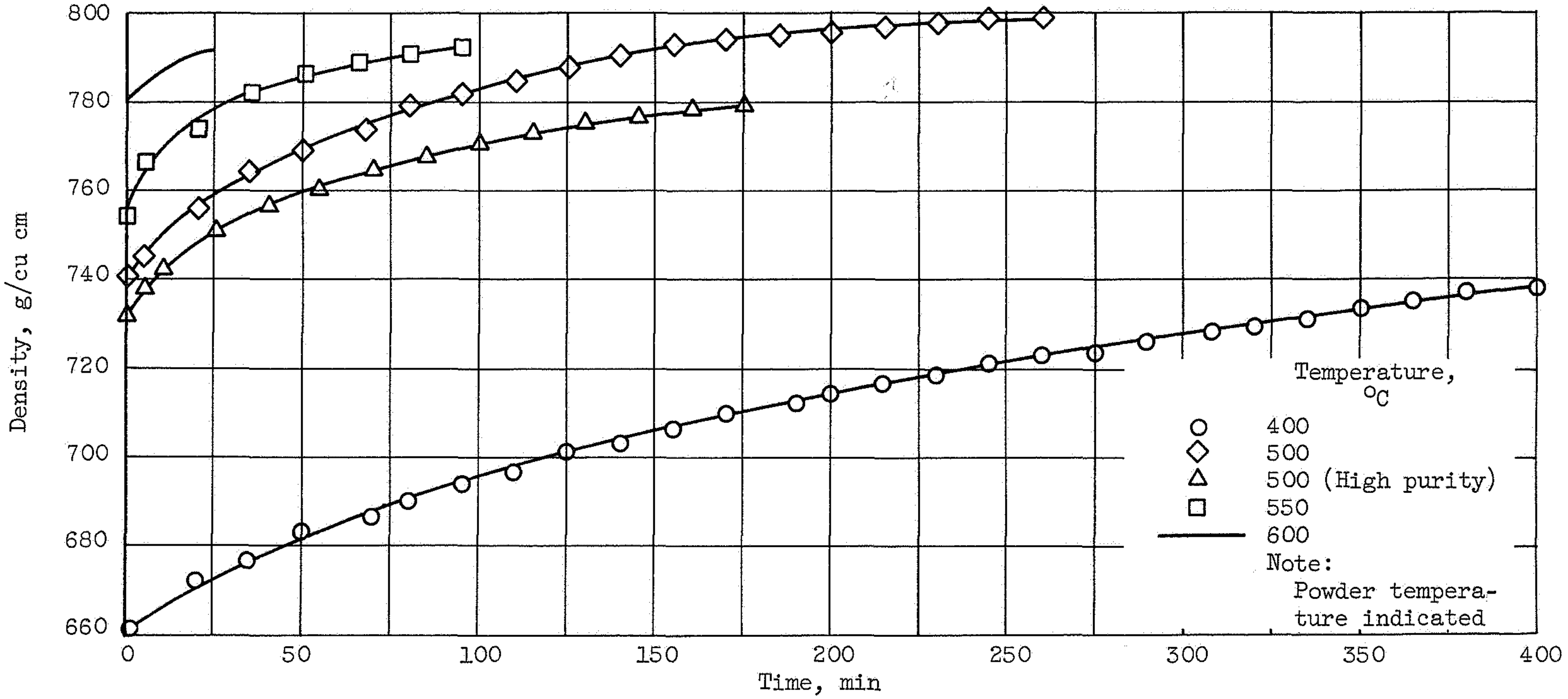


Figure V-2. - Density of compacted lithium hydride as a function of pressing time at various temperatures and at pressing pressure of 1000 psi (ref. 254(U)).

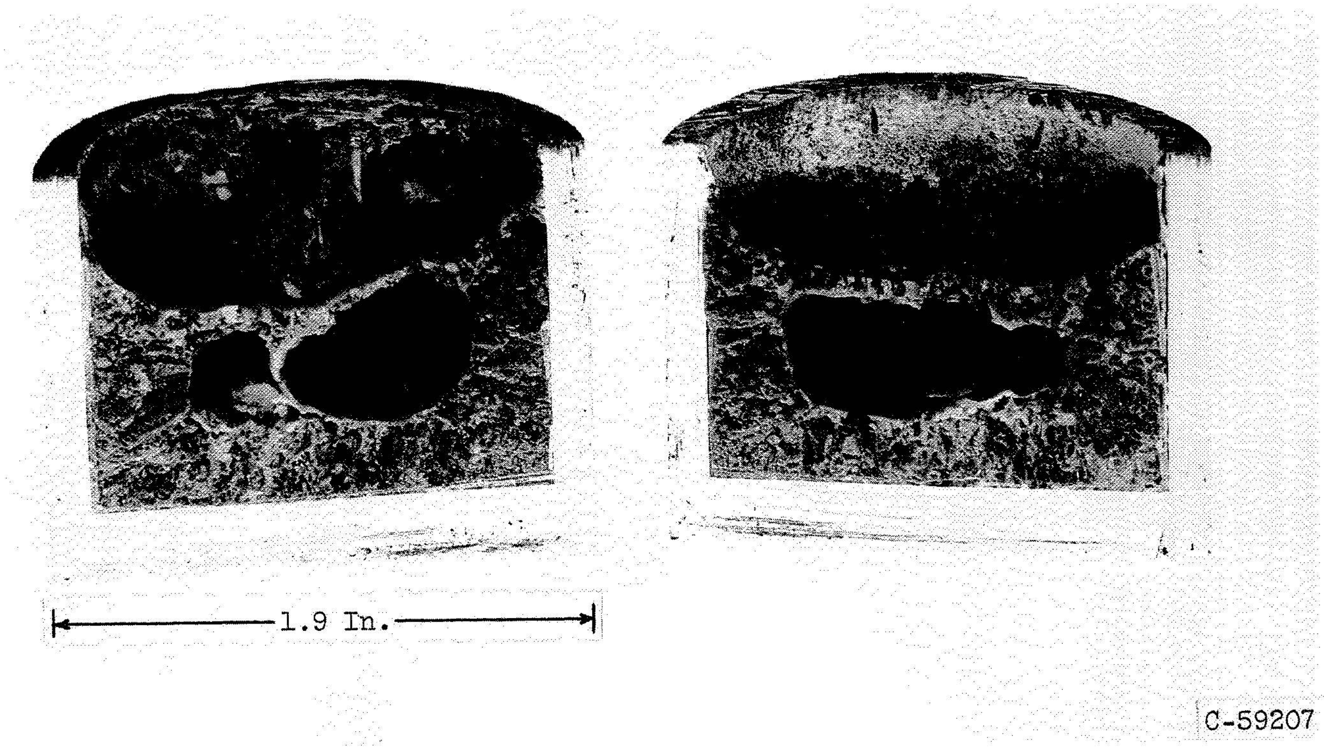


Figure V-3. - Cross section of LiH in 347 stainless steel container. (Personal communication from E. G. Rapp, Thompson Ramo Wooldridge, Inc.)

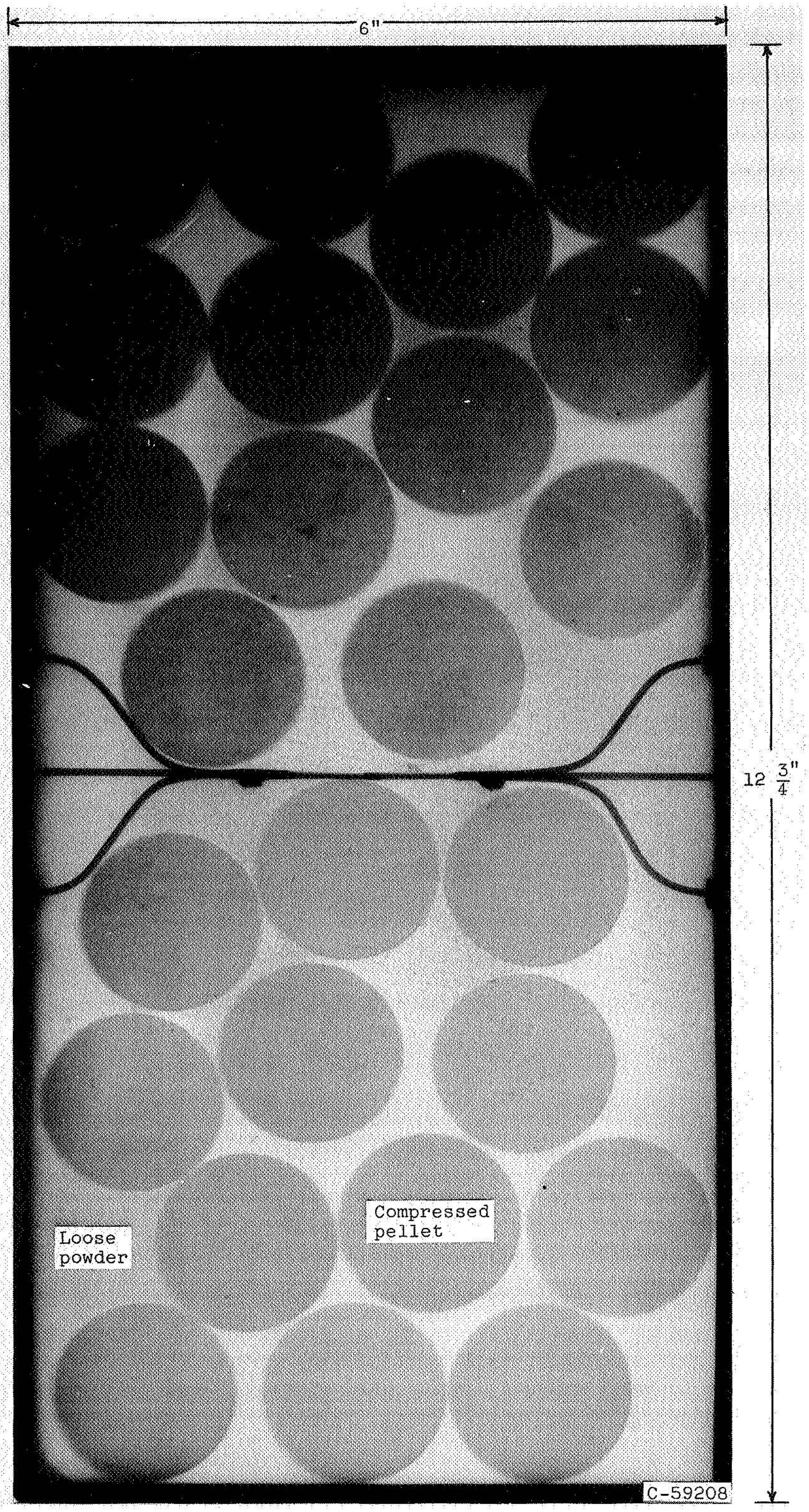


Figure V-4. - Compressed pellets and powder before melting. (Personal communication from E. G. Rapp, Thompson Ramo Wooldridge, Inc.)

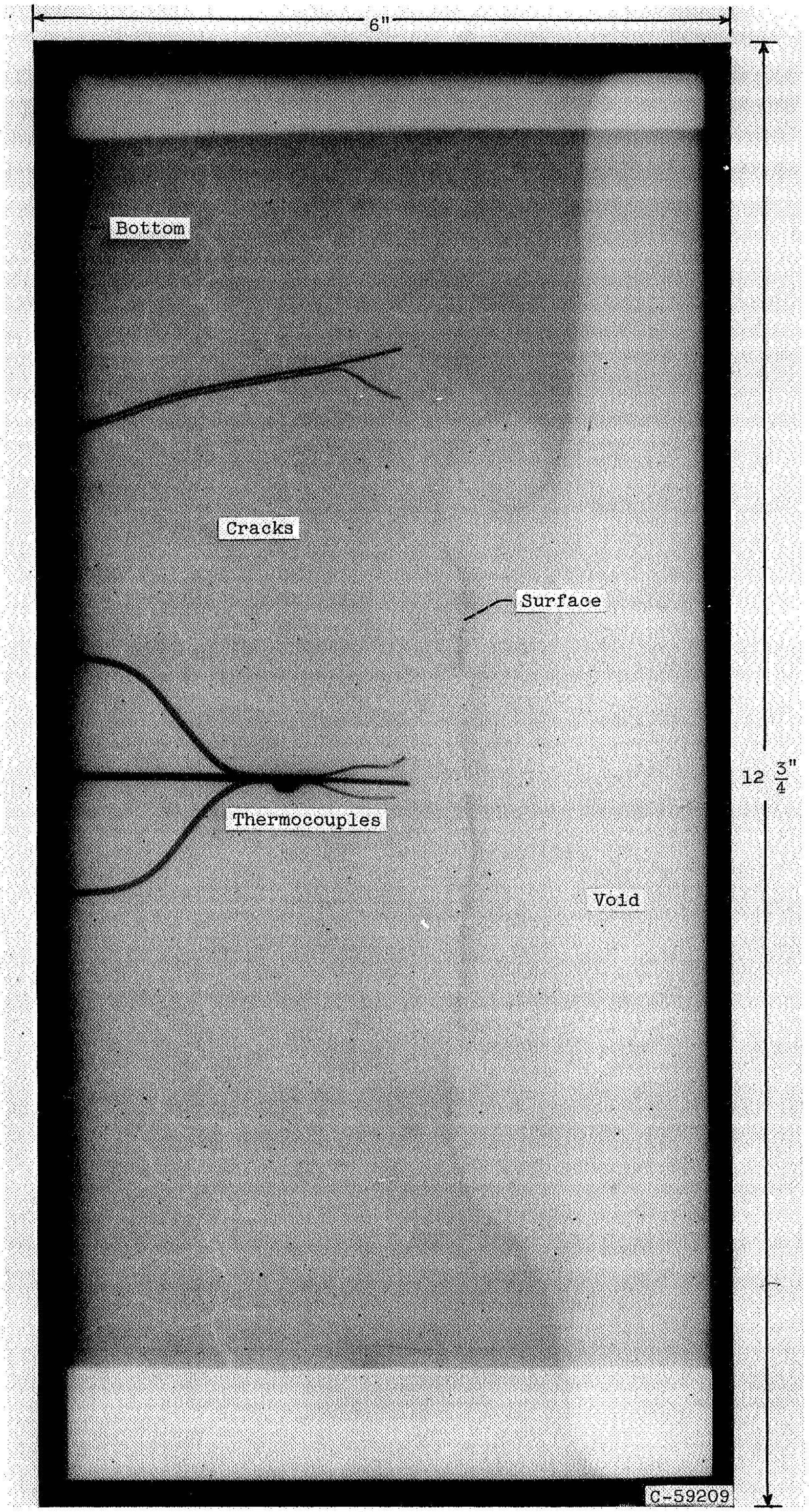


Figure V-5. - Lithium hydride after melting and freezing. (Personal communication from E. G. Rapp, Thompson Ramo Wooldridge, Inc.)

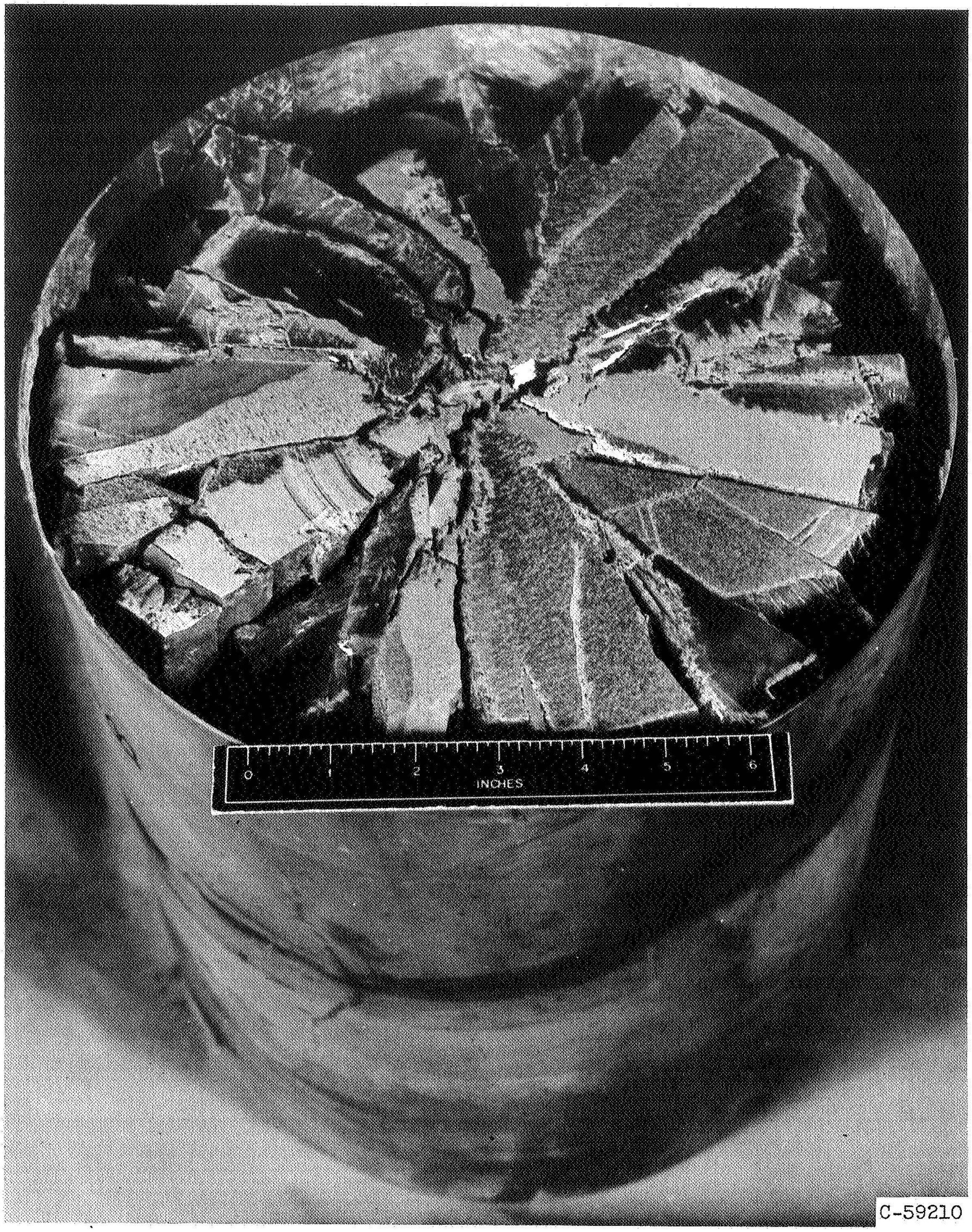


Figure V-6. - Example of cracking which occurs on machining cast hydride with a fly cutter. (Ref. 141(CRD)).

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