a maximum at main-group and sub-group IV elements. The relationship is probably true for the gaseous sulfides, selenides, and tellurides. There is also a decrease in dissociation energy as the molecular weight increases, as expected.

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The Enthalpy of Formation of Lithium Aluminum Hydride

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The following heats of solution have been measured in 4.0*N* hydrochloric acid:

	ΔH_{298} kcal./mole
$Al(c) + 3HCl(aq.\ 4N) \rightarrow AlCl_3(in\ 4N\ HCl) + \frac{3}{2} H_2(g)$	-128.27 ± 0.38
$Li(c) + HCl(aq.\ 4N) \rightarrow LiCl(in\ 4N\ HCl) + \frac{1}{2} H_2(g)$	-67.05 ± 0.53
$LiAlH_4(c) + 4HCl(aq.\ 4N) \rightarrow (LiCl + AlCl_3)(in\ 4N\ HCl) + 4\ H_2(g)$	-170.59 ± 1.29

From these data the enthalpy of formation of lithium aluminum hydride was calculated to be -24.67 ± 2.21 kcal./mole.

ACCURATE CALCULATIONS in chemical thermodynamics depend upon the existence of reliable thermochemical information. A literature search has shown that the only reported value for the heat of formation of lithium aluminum hydride was determined by Davis, Mason, and Stegman (*4*). In that work, the heat of reaction of the hydride with $HCl \cdot H_2O$ was measured. Calculations with available data, gave a heat of formation of -24.08 ± 0.35 kcal./mole for the hydride. The auxiliary data used by Davis have since been updated by Evans (*5*), and the Davis value for the heat of formation of lithium aluminum hydride has been changed accordingly. The corrected value is -25.74 ± 0.40 kcal./mole. Other auxiliary data used by Davis are presently being updated by Evans and, therefore, the heat of formation value remains subject to slight change.

The calculation of the enthalpy of formation of lithium aluminum hydride by measuring separately the heats of solution of lithium, aluminum, and lithium aluminum hydride in 4.0*N* hydrochloride acid is described. From the heats of solution data, the enthalpy of formation of the hydride was calculated.

EXPERIMENTAL

Calorimeter. The heats of solution were measured in a modified Parr combustion calorimeter operated adiabatically. The reaction bomb consisted of a nickel alloy body, the inner wall of which was gold-plated. A special bomb lid was constructed through which passed a centered, moveable shaft. The shaft was made gas-tight by using a double O-ring and grease packing in the lid passage. Two baffle plates were attached to the bottom section of the shaft which, when lowered, crushed the submerged, sample-containing, glass ampoule. The plates also prevented splattering resulting from violent reactions. A thin tantalum sheet was placed on the bottom of the bomb. The bottom plate was constructed of tantalum, also. The lid bottom, shaft, and upper baffle were gold-plated for protection against attack by the acid. A relief valve was built into the lid to exhaust the bomb prior to a run, and to release hydrogen following the run. The lid to the calorimeter jacket was modified to allow the crushing shaft, thermistor tube, and heater tube to pass through to the calorimeter bucket contained within. Beckmann thermometers were used to monitor the bucket and jacket water temperatures. Improvements in water circulation were made by increasing the rate of stirring (smaller pulley used) and by placing a tube around the stirrer blades.

Temperature measurements were made by employing a GB32P8 Fenwal thermistor of 2 kilohms in one arm of a Wheatstone bridge network which was powered by one mercury battery (Mallory RM42R, 1.35v.). The bridge was kept balanced by manipulating three 10-step resistance decades (the finest subdivision was 0.1 ohm) totaling 111 ohms located in series in a second arm of the bridge. The output was amplified by a L & N amplifier 9835-A. The sensitivity of the system was 6.8×10^{-5} °C. per 0.5 division on the amplifier meter. Operating under adiabatic conditions, the thermal leak rate was approximately 7×10^{-5} deg. min.$^{-1}$.

Calibration. The heater with which the calorimeter was electrically calibrated consisted of 15 inches of enameled Cupron wire noninductively wound on a threaded Plexiglass plug and immersed in mineral oil contained in a glass well. The total resistance of the heater was 23.96 ohms as determined from voltage drop measurements. Energy for the heater was supplied by a 12-volt storage battery. The timing of the heating was measured by a Model S-10 Standard Precision timer synchronized with the heater switch. The reactions studied created temperature increases of approximately 0.2°, 0.4°, and 0.6° C. Electrical calibrations were made because of the characteristic nonlinearity of the temperature coefficient of resistance of the thermistor. The heater current was measured by the potential drop across an 0.500 ohm standard wire-wound resistor, utilizing a K-2 potentiometer.

All experimental runs, including calibrations, were initiated at a temperature between 24.85° and 24.86° C. The heat input was calculated as follows:

$$q, \text{cal.} = 0.2390\,(E_s/R_s)\,R_h\,(t_{sec.})$$

The energy equivalent of the calorimeter system over the three ranges of thermistor resistance is shown below.

ΔR, Ohm	E, Cal./Ohm
18–20	35.97 ± 0.06
20–36	36.03 ± 0.08
44–50	36.28 ± 0.14

Each of the results is the average of five determinations. The uncertainties listed throughout this work are twice the standard deviation, in accordance with Rossini (*10*). The results are expressed in the defined calorie (1 cal. = 4.1840 abs. joules). All formula weights were calculated from the 1961 International Atomic Weights. In addition to the electrical calibrations, a chemical check of the apparatus was made by measuring the heat of reaction of magnesium with 1*N* hydrochloric acid. The use of 1*N* hydrochloric acid for the magnesium runs necessitated separate energy equivalent determinations, since the acid concentration differed from that used in the remainder of the study. The heat of reaction of magnesium with 1*N* hydrochloric acid was -111.73 ± 0.56 kcal./mole. A favorable agreement was found with other available data in which 1*N* hydrochloric acid solvent was used.

Investigator	ΔH Results, Kcal./Mole
Shomate & Huffman (*11*)	-111.3 ± 0.02
Lohr (*6*)	-111.3 ± 2.4
Westrum (*12*)	-111.2
Argue (*1*)	-111.2 ± 0.25
This work	-111.7 ± 0.56

In this work, the evolved hydrogen was confined within the bomb; whereas, in the work of the others, the hydrogen was allowed to escape, with corrections made (−0.3 kcal.) for the heat of vaporization of the water necessary to saturate the evolved hydrogen.

Materials. The aluminum was purchased from Mallinkrodt Chemicals as 99.9+ % pure; the lithium was purchased from Lithium Corporation of America as 99.9+ % pure; the magnesium was purchased from New England Lime Company as 99.9+ % pure; and the lithium aluminum hydride was purchased from Metal Hydrides, Inc. as 97.08% pure.

Lithium, aluminum, and magnesium were spectrographically analyzed for 14 elements. The total impurities in aluminum and lithium were less than 100 p.p.m. in each, and in magnesium less than 300 p.p.m. The lithium aluminum hydride was analyzed: By hydrogen evolution, the material was found to liberate 97.01, 96.92, 97.27, and 97.10% (mean = $97.08 \pm 0.09\%$) of the theoretical. The lithium content (by weight) was determined by the flame photometric method to be 17.70, 17.71 (mean = 17.71%); theoretical = 17.76%. The aluminum content (per cent by weight) was determined by the EDTA method to be 69.64, 69.56, 69.24, 69.37 (mean = $69.45 \pm 0.09\%$). The material was also analyzed for chlorine by a gravimetric method. This analysis showed 0.77% chlorine by weight, and this was assumed to be in the form of lithium chloride. Utilizing this analytical information, the material was assayed as 97.08% $LiAlH_4$, 0.92% lithium chloride, 1.22% lithium aluminate, and 0.78% inert material.

PROCEDURE

The samples were sealed under argon in 2-inch lengths of 10-mm. glass tubing. The sample amounts correspond to approximately 0.01 mole. The sample-containing ampoule was immersed in 150 ml. of 4.0*N* hydrochloric acid, contained within the bomb. For the aluminum and lithium aluminum hydride runs, four drops of 10% chloroplatinic acid solution was added to hasten the rates of reaction. The thermal correction for the catalyst was +2.5 cal. and was based on the following equation and data in Circular 500 (*9*).

$$H_2PtCl_6(\text{soln.}) + 2\,H_2(g) \rightarrow Pt(c) + 6\,HCl\,(\text{soln.})$$

$$\Delta H_{298} = -72.8 \text{ kcal./mole}$$

The bomb was sealed, evacuated, back-filled with argon to atmospheric pressure, and immersed in 2000 ml. of water contained in the calorimeter bucket. The initial temperature of the calorimeter and jacket was adjusted to 24.85° to 24.86° C. The reaction was initiated by turning down the central shaft and crushing the sample ampoule. The evolved hydrogen was confined within the bomb. All runs were performed adiabatically. From the amounts of materials used, the following equations represent the reactions studied:

$$Al + 60(HCl \cdot 14H_2O) \rightarrow (AlCl_3 + 57\,HCl)840H_2O + \tfrac{3}{2}H_2$$

$$Li + 60(HCl \cdot 14H_2O) \rightarrow (LiCl + 59HCl)\,840H_2O + \tfrac{1}{2}H_2$$

$$LiAlH_4 + 60(HCl \cdot 14H_2O) \rightarrow (LiCl + AlCl_3 + 56HCl)\,840H_2O + 4H_2$$

RESULTS

The experimental data are shown in Table I. The uncertainties listed include allowance for calibration uncertainties.

The run values were corrected for the energy of breaking the sample. ΔH was calculated from ΔU by the following relationship:

$$\Delta H = \Delta U + \Delta nRT$$

where: Δn refers to the number of moles of hydrogen produced.

Run Al-12 was made in hydrochloric acid to which had been added lithium chloride in an amount comparable to the concentration of lithium ions resulting from a lithium run. No indication of interaction was shown in this run. Run Li-9 was made using a heavier sample weight—no effect was seen in the results. Corrections for the impurities in the $LiAlH_4$ are based on the following equations:

$$LiCl(c) \rightarrow LiCl(800H_2O) \qquad \Delta H_{298} = -8.8 \text{ kcal./mole} \quad (1)$$

$$LiAlO_2 + 4\,HCl\,(aq.) \rightarrow (LiCl + AlCl_3)(\text{in } 4N\ HCl) + 2H_2O = -46.0 \text{ kcal./mole} \quad (2)$$

Both ΔH from Equation 1 and ΔH from Equation 2 were calculated from heat of formation data given in Circular 500 (*9*). The actual corrections for the impurities are as follows:

$$LiCl = -0.073 \text{ kcal.}$$

$$LiAlO_2 = -0.339 \text{ kcal.}$$

$$LiAlH_4 \text{ purity} = 97.08\%$$

$$\Delta H_{298}\,(LiAlH_4) = -165.953 - (-0.412) \text{ kcal./mole of } 97.08\%\ LiAlH_4 = -170.59 \text{ kcal./mole } LiAlH_4$$

A comparison of the aluminum data with other available data is presented below:

Investigator	Year	ΔH_{298}	Comments
Richards(*8*)	1910	−126.0	H_2 liberated
Biltz(*2*)	1922	−125.1	
Young(*13*)	1944	−126.8	H_2 liberated
Coughlin(*3*)	1956	−127.0	
Messer(*7*)	1960	−127.3	H_2 confined.
Circular 500(*14*)[a]	1952	−128.5	By calculation
This work	1963	−128.1	H_2 confined

[a] Data were chosen at dilutions encountered in this work.

No data on the heat of reaction of lithium with aqueous hydrochloric acid were found. Calculation based on Circular 500 data yield −67.44 kcal./mole. This work reports −67.05 kcal./mole as determined directly.

The heat of reaction of $LiAlH_4$ in $1N$ hydrochloric acid has been reported (*4*). The value for ΔH_{298} in $1N$ hydrochloric acid is given as −165.87 kcal./mole by the investigators. The value determined in this work, corrected for impurities, is −170.59 kcal./mole.

From the heats of reaction measured, the enthalpy of formation of $LiAlH_4$ was calculated as shown in Table II:

Table I. Heat of Reaction

Run	Wt., Grams	ΔR,	Heat Evolved, Cal.	ΔU_{298} Cal./Mole	ΔH_{298} Cal./Mole
Aluminum with $4.0N$ HCl					
Al- 4	0.2404	31.909	1147.2	128,757	127,869
Al- 5	0.2398	31.812	1143.7	128,685	127,797
Al- 6	0.2696	36.010	1294.9	129,593	128,705
Al- 7	0.2700	36.067	1297.0	129,611	128,723
Al- 9	0.2701	35.971	1293.5	129,213	128,325
Al-10	0.2719	36.304	1305.5	129,549	128,661
Al-11	0.2707	36.034	1295.8	129,156	128,268
Al-12	0.2703	36.111	1296.1	129,377	128,489
Mean = 128,355, Mean − (corr.) = 128,265, Uncertainty = 384, % = 0.30.					
Lithium with $4.0N$ HCl					
Li-1	0.0696	18.764	674.9	o7,286	66,990
Li-2	0.0777	20.935	753.0	67,247	66,951
Li-3	0.0768	20.758	746.7	67,465	67,169
Li-7	0.0835	22.911	824.1	68,484	68,188
Li-8	0.0954	25.446	915.3	66,575	66,279
Li-9	0.1395	37.701	1358.4	67,569	67,273
Mean = 67,142, Mean − (corr.) = 67,052, Uncertainty = 530, % = 0.79.					
Lithium Aluminum Hydride with $4.0N$ HCl					
	0.4108	50.327	1823.4	168,458	166,089
	0.4306	52.739	1910.9	168,424	166,055
	0.3992	48.823	1768.8	168,162	165,793
	0.3367	41.196	1492.1	168,188	165,819
	0.3862	47.130	1707.4	167,789	165,420
	0.3312	40.957	1480.9	169,697	167,328
	0.3369	41.359	1495.5	168,471	166,102
	0.3471	42.498	1536.8	168,036	165.667
	0.3799	46.241	1686.5	168,483	166,114
Mean = 166,043, Mean − (corr.) = 165,953, Uncertainty = 1,294, % = 0.78.					
Magnesium in $1N$ HCl					
	0.2400	30.627	1107.2	112,159	111,567
	0.2405	30.700	1107.3	111,936	111,344
	0.2404	30.899	1117.0	112,964	112,372
	0.2404	30.930	1113.1	112,569	111,977
Mean = 111,815, Mean − (corr.) = 111,725, Uncertainty = 454, % = 0.41.					

Table II. Enthalpy of Formation of $LiAlH_4$

Reaction	$\Delta H_{f_{298}}$ Kcal./Mole	Uncertainty
$Al + 3HCl\,(aq., 4N) \rightarrow AlCl_3(soln.) + \frac{3}{2}H_2(gas)$	−128.27	±0.38
$Li + HCl\,(aq.\ 4N) \rightarrow LiCl\,(soln.) + \frac{1}{2}H_2(gas)$	−67.05	±0.53
$LiAlH_4 + 4HCl\,(aq., 4N) \rightarrow LiCl\,(soln.) + AlCl_3(soln.) + 4H_2(gas)$	−170.59	±1.29

$$Li + Al + 2H_2(gas) \leftrightarrow LiAlH_4(s)$$

$$\Delta H_{f_{298}} = \Delta H_1 + \Delta H_2 - \Delta H_3 = -24.67 \pm 2.21 \text{ kcal./mole}$$

DISCUSSION

For comparison of the value for the enthalpy of formation of $LiAlH_4$, the Davis paper (*4*) remains the only source. In that work, the heat of reaction of $LiAlH_4$ with $1N$ hydrochloric acid was measured. By applying existing values of the heats of formation of the other reactants and products, the enthalpy of formation of $LiAlH_4$ was calculated to be −24.08 kcal./mole. The value determined in this work is −24.67 kcal./mole.

The Davis value has been updated by Evans (*5*) as a result of the availability of more recent data. This has changed the auxiliary data used by Davis and his value has now been corrected to −25.74 kcal./mole. The value changes with the availability of updated auxiliary data which may differ from that used by other workers. The method used in this work minimized the need for auxiliary data, including dilution information, since the sample weights were chosen to be equal to their weight in the hydride sample. The comparison of the two values is favorable, however.

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NOMENCLATURE

ΔU_{298} = heat of solution at constant volume and at 298° K., kcal./mole
ΔH_{298} = heat of solution at constant pressure and at 298° K., kcal./mole
$\Delta H_{f\,298}$ = enthalpy of formation at 298° K., kcal./mole
q = electrical energy supplied to the calorimeter, calories
E_s = voltage drop across standard resistor, volts
R_s = 0.500 ohm standard wire-wound resistor
R_h = heater resistance = 23.96 ohms
$t_{sec.}$ = heating time, seconds
E = energy equivalent, calories/ohm
ΔR = change in the resistance of the thermistor

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Thermal Conductivities of Aluminum and Zinc Powder Suspensions

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Thermal conductivities are presented for suspensions of powders that are highly conducting compared with the continuous phase. Aluminum and zinc in varying particle sizes and shapes dispersed in lubricating grease in amounts of up to 80% by weight comprised the suspensions. A comparison of experimental results with values predicted from a number of correlating expressions showed generally satisfactory agreement at the lower concentrations but considerable disparity at the higher concentrations. The experimental apparatus and technique are briefly described.

THE THERMAL CONDUCTIVITY of two-phase systems is a function of numerous variables and is difficult to predict, particularly when the thermal conductivity of the two phases differ by over two orders of magnitude. Experimental data are therefore desirable. Results are reported herein for several systems consisting of metal powder dispersions in lubricating grease and comparisons are made with analytical expressions.

EXPERIMENTAL

Materials. Aluminum and zinc powders with varying size and shape particles were employed as the dispersed phase and Marfak No. 1 lubrication grease of the Texas Company served as the continuous phase. One aluminum powder was composed of irregular particles having a mass mean diameter of 40μ and a geometric standard deviation of 2.0. Three other aluminum powders, hereafter referred to as aluminum A, aluminum B, and aluminum C, had mass mean diameters of 33, 40, and 53μ and geometric standard deviations of 2.5, 1.8, and 1.5, respectively. Two zinc powders had irregular particles of 60 and 80μ in mean diameter with standard deviations of 1.4 and 1.6, respectively. The thermal conductivities of zinc and aluminum are 64 and 118 B.t.u./hr. ft. ° F. (*2*), respectively, while the grease had a conductivity of approximately 0.16 B.t.u./hr. ft. ° F.

Apparatus. A steady state, parallel-disk type of conductivity apparatus was employed, and with it, as nearly as possible, a constant heat flux was maintained across the test material. Figure 1 shows the principal features of the calorimeter. The upper plate was maintained at a constant and higher-than-room temperature by the circulation of hot water from a constant temperature bath through an attached coil of copper pipe. This coil was dual-wound and attached to a copper plate, as shown in Figure 2, so that the heat from the water would be distributed evenly over the plate creating an essentially isothermal surface. The bottom portion of the hot plate was separated with a thin ring of transite into two sections so that the center part of the plate would be guarded against adverse temperature effects at the outer periphery of the plate. The chamfer at the bottom of the hot plate provided an escape for entrained air from beneath the hot plate during assembly with suspension in place. It was essential that the heat conducting medium contact the hot plate at all points.

The calorimeter was encased in a transite shell, the ends