pressibility data in the region of low $1/V$ must be available to determine the intercept precisely. This was not the case for 3-methylpentane, since the lowest density $(1/V)$ measured was 3.2 gram moles/liter at pressures of 41 and 55 atm., respectively.

A certain amount of scatter was found when the reduced third virial coefficients for the isomeric hexanes were plotted against reduced temperature (Figure 2). This result was not surprising, since David and Hamann (*2*) had found about the same degree of scatter (except the deviation of the 3-methylpentane points) for varied data in determining the solid line of Figure 2.

Here again the greater deviation of the 3-methylpentane data is probably due to lack of low density compressibility data.

NOMENCLATURE

B = second virial coefficient, liter/gram mole
C = third virial coefficient, liter/gram mole
R = gas constant, liter atmosphere/gram mole ° K.
T = temperature, ° K.
V = molal volume, liter/gram mole
Z = compressibility factor
$B^* = B/V_c$ reduced second virial coefficient
$C^* = C/V_c^2$ = reduced third virial coefficient
$T^* = T/T_c$ = reduced temperature
T_c = critical temperature, ° K.
V_c = critical molal volume, liter/gram mole

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Heats and Free Energies of Formation of the Alkali Aluminum Hydrides and of Cesium Hydride

Heat of Formation of Aluminum Chloride in Hydrochloric Acid

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Standard heats of formation of five metal hydrides were determined by measuring their heats of reaction with HCl. The following values are reported for ΔH_f^0 (25° C.) in kcal./mole: $LiAlH_4$(c), −28.4; $NaAlH_4$(c), −27.0; $KAlH_4$(c), −39.8; $CsAlH_4$(c), −39.4; CsH(c), −11.92. Free energies of formation were calculated. Thermodynamic stabilities of the alkali aluminum hydrides are discussed. Heat of formation of aluminum chloride in HCl was determined for the HCl molality range 0 to 4.6 and for $H_2O/AlCl_3$ mole ratios ranging from 200 to 500.

WITH THE EXCEPTION of lithium aluminum hydride (*2*), the heats of formation of the alkali aluminum hydrides have not been reported. Published values for cesium hydride, −19.9 (*7*) and −13.48 (*6*), agree poorly. These hydrides react rapidly and completely with HCl to form products having known heats of formation. Measurement of the heats of these reactions therefore provided an excellent method of obtaining the heats of formation. To facilitate the calculation of the heats of formation, the heat of formation of aluminum chloride in HCl was determined over suitable ranges of HCl molality and $H_2O/AlCl_3$ ratio.

EXPERIMENTAL

Materials. The five crystalline hydrides were synthesized in this laboratory. Analyses and estimated purities are summarized in Table I. The potassium aluminum hydride was assumed to contain 2.3% KH, and the heats of hydrolysis were corrected accordingly.

All other chemicals used were reagent-grade materials.

APPARATUS AND PROCEDURE

The calorimeter consisted of a 300-ml. silvered borosilicate glass Dewar fitted with a rubber stopper. Temperatures were read to the nearest 0.01° C. with an accurately cali-

Table I. Purities of Hydrides

Hydride	% of Theoretical			Estimated Purity[b], Wt. %
	Alkali Metal	Al	H	
$LiAlH_4$	100.0	99.0	96.8	99.0
$NaAlH_4$	100.0	99.9	96.0	99.5
$KAlH_4$	102.9	97.7	96.7	97.7
$CsAlH_4$	99.8	99.6	95.9	99.6
CsH[a]	98.7	...	94.7	98.0

[a] Contained 0.3 wt. % organic matter. [b] Based on alkali metal and aluminum analyses, disregarding hydrogen analyses, which are characteristically low.

brated thermometer. About 60 ml. of aqueous HCl and 100 ml. of high-boiling liquid hydrocarbon (*n*-undecane) were weighed into the calorimeter. The liquids were stirred magnetically with a Teflon-covered bar. The sample was introduced into the calorimeter from an accurately weighed vial. To minimize exposure of the sample during its addition, the experiments were performed in a nitrogen dry-box. The deep hydrocarbon layer covering the acid served to slow the reaction, which otherwise would have been too vigorous.

The temperature rise for the experiment was determined from a temperature-time plot. The heat of reaction was calculated from the temperature rise, the total heat capacity of the system, and the amount of sample. The heat of reaction was corrected for the heat loss due to the vaporization of water from the calorimetric liquids (the amount of water vaporized was measured). It was also corrected from the initial reaction temperature to 25° C. All weights of materials were corrected for buoyancy. The densities of the hydrides, required for the latter corrections, were determined by liquid displacement. The values at 25° C. so obtained, in gram/ml., are: $LiAlH_4$, 0.918; $NaAlH_4$, 1.28; $KAlH_4$, 1.33; $CsAlH_4$, 2.84; CsH, 3.42.

Numerical Constants. The energy measurements were made in terms of the thermochemical calorie, one calorie being the equivalent of 4.1840 absolute joules. The 1959 International Atomic Weights were used.

Heat Capacity Data. The specific heat of *n*-undecane was read from a plot based on the measurements of Finke and others (*4*). The specific heat of HCl was determined by interpolating the measurements of Wrewsky and Kaigorodoff (*13*). The small adjustments in the heat capacities of the aqueous phase required to correct for the presence of dissolved salts were based on specific heat data given in the International Critical Tables for aqueous solutions of these salts. Specific heats for the hydrides were estimated since their contributions to the total heat capacities were small.

The heat capacity of the calorimeter (including the thermometer and stirring bar) was evaluated by pouring a measured quantity of *n*-undecane at a known temperature near ambient into the calorimeter, which had been preheated to a higher, known temperature. Values were obtained in this manner at several mean temperatures, the value at 30° C. being 19.13 cal./degree.

Over-all Check of Apparatus. As a check on the accuracy of the method, the heat of solution of anhydrous magnesium chloride (about 99% pure) in 1*N* HCl was measured and compared with the results of Shomate and Huffman (*12*). The results obtained for ΔH_s at the concentration $MgCl_2 \cdot 200\,H_2O$ are −36.07, −35.97, and −36.05 kcal./mole, or an average of −36.03 kcal./mole. The results of Shomate and Huffman, adjusted to the same concentration using dilution data from NBS Circular 500 (*11*), give −36.06 kcal./mole. The agreement is quite satisfactory.

HEAT OF FORMATION OF ALUMINUM CHLORIDE IN HYDROCHLORIC ACID

Method and Experimental Data. The heat of formation of aluminum chloride in HCl was evaluated by measuring the heat of solution of the hexahydrate at various acid strengths and dilutions. The experimental data and the calculated heats of solution are shown in Table II.

Calculations and Correlations. The heats of solution corresponding to $H_2O/AlCl_3$ mole ratios in the vicinity of 230, adjusted slightly to make them correspond to mole ratios of exactly 230, are re-listed in Table III. The heats of formation shown in the last column were calculated from the following equation (the "heat of formation of $AlCl_3$" is defined as the apparent molal heat content of $AlCl_3$, the HCl being regarded as part of the solvent):

$$\Delta H_f = -233.70 + \Delta H(\text{soln.}) - \Delta H(\text{diln.}) \tag{1}$$

In this equation, the constant −233.70 is the ΔH_f of aluminum chloride in the hexahydrate as determined recently by Coughlin (*1*), and ΔH(diln.) is the heat of dilution of the HCl by the water in the hexahydrate. As shown in Figure 1, the heat of formation of aluminum chloride is a linear function of HCl molality (final), within experimental error. The following equation was fitted to the data:

$$\Delta H_f(m, 230) = -\,246.87 + 1.518\,m \tag{2}$$

Table II. Heat of Solution of $AlCl_3 \cdot 6H_2O$ in Aqueous HCl

Grams $AlCl_3 \cdot 6H_2O$	Wt. % HCl (Initial)	Moles H_2O per Mole $AlCl_3$ (Final)	Initial Temp., ° C.	Δt, ° C.	$-\Delta H$(Soln.) at 25° C., Kcal./Mole
8.3004	0.03778	230.7	25.22	2.71	13.13
8.3011	0.03778	231.4	24.99	2.71	13.18
8.3030	4.000	227.2	24.86	2.46	11.46
8.2979	4.000	227.1	24.55	2.47	11.53
8.2725	6.794	222.8	25.94	2.29	10.30
8.2813	6.794	224.1	25.87	2.29	10.37
8.3280	6.794	216.1	25.12	2.30	10.17
7.7135	13.27	228.5	26.44	1.63	7.34
7.6352	13.27	232.0	24.59	1.60	7.38
7.6671	14.72	229.5	24.74	1.45	6.59
7.7076	14.72	227.0	24.58	1.47	6.60
4.1842	0.03778	452.2	25.14	1.39	13.36
3.1271	0.03778	599.7	25.17	1.06	13.55
3.8856	0.03778	488.7	25.05	1.30	13.42
4.1667	6.790	439.3	25.83	1.17	10.45
4.1654	6.790	439.9	24.43	1.15	10.40
5.1480	14.72	336.5	24.77	0.98	6.57
5.1179	14.72	340.9	24.53	1.00	6.78
3.8766	14.72	446.1	25.63	0.78	6.87
3.9058	14.72	441.0	26.06	0.77	6.75

Table III. Heat of Formation of $AlCl_3$ in Aqueous HCl at $H_2O/AlCl_3$ Ratio of 230

HCl Molality (Final)	Kcal./Mole at 25° C.		
	$-\Delta H$ of solution	$-\Delta H$ of dilution	$-\Delta H_f$ of $AlCl_3$
0.01010	13.13	0.00	246.83
0.01010	13.18	0.00	246.88
1.112	11.47	0.03	245.14
1.112	11.53	0.03	245.20
1.945	10.31	0.09	243.92
1.945	10.38	0.09	243.99
1.944	10.20	0.09	243.81
4.086	7.34	0.36	240.68
4.087	7.38	0.36	240.72
4.610	6.59	0.44	239.85
4.609	6.60	0.44	239.86

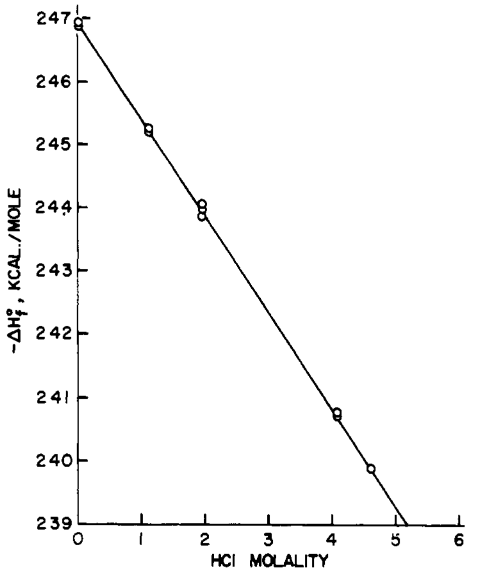

Figure 1. Heat of formation of $AlCl_3$ in aqueous HCl *vs.* molality at $nH_2O/nAlCl_3 = 230$

The heat of solution data taken at varying dilutions indicate that, for the ranges studied, the heat of dilution of aluminum chloride in HCl (with HCl of the same molality) does not vary with the molality within experimental error. The heat of formation of aluminum chloride at any dilution in the $H_2O/AlCl_3$ range of 200 to 600 may therefore be calculated from the equation:

$$\Delta H_f\,(m, r_n) = -246.87 + 1.518m + \Delta H\,(\text{diln., 230 to } r_n) \quad (3)$$

Approximate values of the heat of dilution are shown in Table IV.

As may be seen from Figure 1, the decrease in $-\Delta H_f^0$ of $AlCl_3$ with increasing HCl molality is considerable, amounting to 7.6 kcal. from $m = 0$ to $m = 5$. This indicates that one or more endothermic reactions may occur in aqueous solution between HCl and whatever species are produced when $AlCl_3$ is dissolved in dilute HCl. This phenomenon has been observed by other investigators, for example by Klemm (*10*), who suggested the formation of complex ions of the type $AlCl_6^{=}$. Other possible endothermic reactions include the formation of $AlCl_4^-$ ions, which are known to exist in nonaqueous systems.

Table IV. Approximate Heats of Dilution of $AlCl_3$

r_n $nH_2O/nAlCl_3$	ΔH(Diln.) 230 to r_n Kcal./Mole
200	+0.06
220	+0.02
240	−0.02
260	−0.05
280	−0.08
300	−0.10
350	−0.15
400	−0.20
450	−0.24
500	−0.27
550	−0.29
600	−0.31

HEATS OF HYDROLYSIS OF HYDRIDES

The experimental data and calculated heats of hydrolysis at 25.00° C. are listed in Table V. The equation representing the hydrolysis reaction for a complex hydride is of the form:

$$XAlH_4(c) + a(HCl\cdot bH_2O) \rightarrow [XCl + AlCl_3 + (a-4)(HCl\cdot dH_2O)] + 4H_2(g) \quad (4)$$

$$\Delta H = \Delta H_h$$

Since the heat of formation of XCl in solution was not known, it was necessary to measure the heat of solution of the anhydrous salt. The amounts of materials used were such that the resulting solution had the same composition as the final solution from the hydrolysis reaction. The equation representing this process is of the form:

$$XCl(c) + [AlCl_3 + (a-4)(HCl\cdot dH_2O)] \rightarrow [XCl + AlCl_3 + (a-4)(HCl\cdot dH_2O)] \quad (5)$$

$$\Delta H = \Delta H_s$$

Subtraction of Equation 5 from Equation 4 gives the following "*modified hydrolysis*" equation:

$$XAlH_4(c) + a(HCl\cdot bH_2O) \rightarrow XCl(c) + [AlCl_3 + (a-4)(HCl\cdot dH_2O)] + 4H_2(g) \quad (6)$$

$$\Delta H_{mh} = \Delta H_h - \Delta H_s$$

Similarly, the modified hydrolysis equation for CsH is:

$$CsH(c) + a(HCl\cdot bH_2O) \rightarrow CsCl(c) + (a-1)(HCl\cdot dH_2O) + H_2(g) \quad (7)$$

$$\Delta H_{mh} = \Delta H_h - \Delta H_s$$

The calculation of heats of modified hydrolysis from mean heats of hydrolysis and heats of solution is shown In Table VI. Values of *a*, *b*, and *d* are also listed.

HEATS OF FORMATION OF HYDRIDES

Heats of formation of HCl(*aq*) and XCl(*c*) were obtained from NBS Circular 500 (*11*). Heats of formation of $AlCl_3$(*aq* HCl) were calculated from Equation 3 using the following values of m and r_n: $LiAlH_4$, 4.294, 462; $NaAlH_4$, 4.156, 377; $KAlH_4$, 4.227, 449; $CsAlH_4$, 4.416, 618. Heats of formation were combined with heats of modified hydrolysis (Table VI), according to Equations 6 and 7, to yield the heats of formation of the hydrides shown in Table VII. The uncertainty assigned to each experimental heat of formation was determined by combining twice the standard deviation of the mean (Table V) with estimates of all other known sources of error.

The agreement between the results of this investigation and the literature results included in Table VII is excellent in the case of $LiAlH_4$. In the case of CsH, the agreement with the value −13.48 reported by Herold (*6*) is reasonable considering the indirectness of the latter's method (the heat of formation was calculated from measurements of decomp-

Table V. Experimental Data and Calculated Heats of Hydrolysis

Hydride	Wt. % HCl	Hydride, Grams	Initial Temp., °C.	Δt, °C.	$-\Delta H_h$ at 25.00° C., Kcal./Mole
$LiAlH_4$	14.83	0.2560	26.75	10.29	165.2
		0.2652	24.37	10.57	163.8
		0.2480	23.45	10.04	166.2
		0.2449	24.85	9.71	162.9
		0.2509	24.22	9.94	162.9
		0.2475	24.35	10.01	166.4
				Mean	164.6
				Std. dev. of the mean	±0.65
$NaAlH_4$	14.75	0.4389	25.33	11.86	157.0
		0.3998	25.44	10.92	158.7
		0.4202	25.51	11.36	157.3
		0.4494	25.46	12.29	158.5
				Mean	157.9
				Std. dev. of the mean	±0.4
$KAlH_4$	14.72	0.4739	24.06	9.14	147.6
		0.4799	24.74	9.35	149.2
		0.4888	25.31	9.47	148.5
				Mean	148.4
				Av. dev. from the mean	±0.5
$CsAlH_4$	14.83	0.7853	23.43	6.64	149.7
		0.7785	23.88	6.58	149.6
		0.8277	23.49	6.94	148.6
				Mean	149.3
				Av. dev. from the mean	±0.5
CsH	5.07	2.0414	25.36	6.57	48.32
		2.0637	25.35	6.63	48.58
		2.0113	25.56	6.44	48.14
		1.9973	24.77	6.40	48.28
		2.0022	23.73	6.39	48.37
		1.9976	24.33	6.40	48.28
				Mean	48.33
				Std. dev. of the mean	±0.06

Table VII. Standard Heats of Formation of Hydrides

Hydride	$-\Delta H_f^0$ (25° C.), Kcal./Mole Found	Lit. Value	Ref.
$LiAlH_4$	28.4 ±1.5	28.51[a]	(2)
$NaAlH_4$	27.0 ±1.0		
$KAlH_4$	39.8 ±0.9		
$CsAlH_4$	39.4 ±0.8		
CsH	11.92±0.25	13.48	(*6*)
		19.9	(*7*)

[a] Recalculated from experimental heat of hydrolysis. Heat of solution of LiCl(*c*) in HCl(*aq*) + $AlCl_3$ was measured as $\Delta H_s = -8.22$ kcal./mole. The ΔH_f^0 of $AlCl_3$ (*aq* HCl) was calculated from Equation 3 as −246.13 kcal./mole.

Table VIII. Heats and Free Energies of Formation at 25° C.

Crystalline Compound	$-\Delta H_f^0$, Kcal./Mole	$-\Delta F_f^0$, Kcal./Mole	$-298.15\ \Delta S_f^0$, Kcal./Mole	S^0 (25° C.)
LiH	21.67[a]	16.8	4.9	5.9[b]
NaH	13.49[a]	8.6	4.9[c]	11.4
KH	13.82[a]	8.9	4.9[c]	14.5
CsH	11.92[d]	7.0	4.9[c]	19.3
$LiAlH_4$	28.5[d, e]	12.9	15.6[f]	23.5
$NaAlH_4$	27.0[d]	11.6	15.4[f]	29.6
$KAlH_4$	39.8[d]	23.8	16.0[f]	30.8
$CsAlH_4$	39.4[d]	23.5	15.9[g]	36.0
$LiBH_4$	46.37[e]	30.75	15.62	18.13[b]
$NaBH_4$	45.63[e]	30.19	15.44	24.21[b]
KBH_4	54.70[h]	38.70	16.00	25.48[i]

[a] From (*5*). [b] From (*9*). [c] Estimated as equal to corresponding value for LiH. [d] This investigation. [e] Recalculated from (*2*) using more recent heats of formation. [f] Estimated as equal to value for corresponding borohydride. [g] Estimated from the other aluminum hydrides. [h] From (*8*). [i] From (*3*).

osition pressure at elevated temperatures). Both our value and Herold's value disagree markedly with the value −19.9 given by Hurd (*7*), which seems unduly larger in magnitude than the well-established values for sodium and potassium hydrides [−13.49 and −13.82. (*5*)].

COMPILATION OF HEATS AND FREE ENERGIES OF FORMATION OF ALKALI HYDRIDES

The experimental heats of formation are summarized in Table VIII along with literature values for three other alkali simple hydrides and three alkali borohydrides. Literature values of absolute entropy were used to calculate values of $-298.15\ \Delta S_f^0$ which were combined with heats of formation to give free energies of formation. Where literature entropies were unavailable, values of $-298.15\ \Delta S_f^0$ were estimated and used in the calculation of free energies of formation and absolute entropies. Among the simple hydrides, the magnitude of the heat (or free energy) of formation is much greater for the lithium compound than for the other compounds, and is least for the cesium compound. In both the aluminum hydrides and the borohydrides, on the other hand, there is a small decrease in the magnitude of the heat (or free energy) of formation from the lithium to the sodium compound, followed by a large increase from the sodium to the potassium compound. Since all the hydrides listed have negative free energies of formation, they are all thermodynamically stable with respect to complete decomposition into the elements.

THERMODYNAMIC STABILITIES OF COMPLEX HYDRIDES

Heats and free energies for the decomposition of the complex hydrides into simple alkali hydride, metal, and hydrogen were calculated from the data in Table VIII. The results are listed in Table IX. As indicated by the

Table VI. Heats of Solution and Heats of Modified Hydrolysis at 25° C.

Hydride	*a*	*b*	*d*	Kcal./Mole $-\Delta H_h$	ΔH_s	$-\Delta H_{mh}$
$LiAlH_4$	39.715	11.624	12.926	164.6	−6.75	157.85
$NaAlH_4$	32.257	11.698	13.354	157.9	+1.9	159.8
$KAlH_4$	37.421	11.726	13.130	148.4	+4.5	152.9
$CsAlH_4$	53.191	11.624	12.569	149.3	+3.3	152.6
CsH	5.604	37.899	46.130	48.33	+4.02	52.35

Table IX. Heats and Free Energies at 25° C. of Decomposition Reactions of the Type $XMH_4(c) \longrightarrow XH(c) + M(c) + 3/2H_2(g)$

Complex Hydride	ΔH^0, Kcal./Mole	ΔF^0, Kcal./Mole
$LiAlH_4$	+6.8	−3.9
$NaAlH_4$	+13.5	+3.0
$KAlH_4$	+26.0	+14.9
$CsAlH_4$	+27.5	+16.5
$LiBH_4$	+24.70	+14.0
$NaBH_4$	+32.14	+21.6
KBH_4	+40.88	+29.8

free energies, the aluminum hydrides are considerably (15 to 18 kcal. at 25° C.) less stable toward this type of decomposition than the corresponding borohydrides. Of the seven complex hydrides, lithium aluminum hydride is the only one for which the ΔF^0 is negative in sign. It is therefore the only hydride which is thermodynamically unstable at 25° C.

Lithium aluminum hydride is about 7 kcal. less stable thermodynamically than sodium aluminum hydride, which in turn is about 12 kcal. less stable than potassium aluminum hydride. Isothermal rate-of-decomposition data place the three compounds in the same order of increasing stability. This stepwise increase in stability from the lithium to the sodium to the potassium compound is also exhibited by the borohydrides. Finally, the decomposition reactions for all seven hydrides are endothermic, hence these reactions cannot become "runaway."

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Latent Heat of Vaporization of *n*-Decane

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The latent heat of vaporization of *n*-decane was determined by calorimetric measurements at temperatures between 160° and 340° F. The results are presented in graphical and tabular form by application of the Clapeyron equation. The specific volume of the dew-point gas was evaluated throughout the above-indicated range of temperature. The current measurements are in good agreement with earlier volumetric data and critically chosen values of the latent heat of vaporization which have been reported.

DIRECT CALORIMETRIC MEASUREMENTS of the latent heat of vaporization of *n*-decane over a range of temperatures do not appear to be available. Rossini (*6*) reports values at 77° and 345° F. The volumetric behavior of the liquid phase of *n*-decane has been studied by Reamer and others (*5*). Likewise, the vapor pressure has been determined over a wide range of temperatures by Young (*9*), Reamer (*5*), and Willingham and coworkers (*8*). The volumetric data extend from a temperature of 40° to 460° F., and the vapor pressures cover a similar range of temperatures. Rossini (*6*) has also recorded several critically chosen values of the properties of *n*-decane at 77° F. As a result of the absence of directly measured values of the latent heat of vaporization of this paraffin hydrocarbon over a wide range of temperatures, the current investigation was initiated. This study involves measurements of the latent heat of vaporization of *n*-decane at temperatures between 160° and 340° F.