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William L. Jolly

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HYDRIDES

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Hydrides

By the broadest definition, a hydride is any compound containing hydrogen. When one considers that almost all carbon compounds contain hydrogen and that hydrogen forms a multitude of compounds with all the elements except some of the rare gases, it seems probable that there are more known hydrogen compounds (hydrides) than any other type of compound. Hydrides are known with a wide variety of structures and bonding types. This versatility of hydrogen is attributable to the unique electronic structure of the hydrogen atom: the atom possesses no core electrons; it has only one valence electron (and thus is formally analogous to the alkali metal atoms), and it is only one electron short of a stable rare gas electronic configuration (and thus is analogous to halogen atoms). The known hydrides may be classified as saline hydrides, metallic hydrides, transition-metal hydride complexes, and covalent hydrides; however, some hydrides have the properties of more than one class, and the classification is rather arbitrary. We shall discuss these hydride categories separately.

Saline Hydrides.- At high temperatures, hydrogen reacts with the alkali metals and the alkaline-earth metals heavier than beryllium to form salts, such as NaH and CaH_2,

containing the hydride ion, H^-. Inasmuch as the ionic radii of H^- and F^- are very similar (1.40 and 1.36 Å, respectively), the physical properties of the saline hydrides are similar to those of the corresponding fluorides. Even the trend in the heats of formation of the alkali metal hydrides is like that for the alkali metal fluorides. In each case, the lithium compound is more stable than the cesium compound. (Seee Table I.) The heat of formation of a hydride, ΔH_f°, may be broken up into a number of quantities: the sublimation energy of the metal, S; one-half the dissociation energy of hydrogen, $\frac{1}{2}D$; the ionization potential of the metal, I; the electron affinity of hydrogen, E, and the lattice energy, U. Thus we may write

$$\Delta H_f^\circ = S + \tfrac{1}{2}D + I - E - U$$

The factor which is most important in determining the trend in ΔH_f° is the lattice energy. The lighter alkali metal hydrides are more stable because the trend in U more than compensates for the opposing trends in S and I.

Striking evidence for the negative charge of the hydrogen in lithium hydride is obtained when molten lithium hydride (m.p. 691°) is electrolyzed: lithium metal forms at the cathode, and hydrogen is evolved at the anode.

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Table I

Properties of the Saline Hydrides

Formula	Density g/ml	ΔH° of formation, kcal/mole	Crystal Structure
LiH	0.778	-21.67	NaCl
NaH	1.36	-13.49	NaCl
KH	1.43	-13.82	NaCl
RbH	2.60	-11.3	NaCl
CsH	3.41	-11.92	NaCl
MgH_2	1.42	-17.79	Rutile
CaH_2	1.9	-41.65	orthorhombic; distorted $PbCl_2$
SrH_2	3.27	-42.3	
BaH_2	4.15	-40.9	

The saline hydrides react vigorously with protonic solvents such as water, alcohol, and liquid ammonia to form molecular hydrogen and the basic anions of the solvents:

$$CaH_2 + 2H_2O \rightarrow Ca^{2+} + 2OH^- + 2H_2$$

$$LiH + C_2H_5OH \rightarrow Li^+ + C_2H_5O^- + H_2$$

$$KH + NH_3 \rightarrow K^+ + NH_2^- + H_2$$

Although the hydride ion cannot exist in aqueous solution in any appreciable concentration because of its extreme reactivity, the free energy of the ion has been estimated, permitting the inclusion of the H^- ion in the oxidation potential diagrams for hydrogen.

<u>Acidic Solutions</u> $\quad H^- \xrightarrow{2.25} H_2 \xrightarrow{0.0} H^+$

<u>Basic Solutions</u> $\quad H^- \xrightarrow{2.25} H_2 \xrightarrow{0.83} H_2O$

The H^- ion ranks between the metals aluminum and sodium in its reducing power.

Sodium hydride finds considerable use as a reducing agent in synthetic chemistry. Sodium hydroborate, $NaBH_4$, is prepared by the reaction of sodium hydride with methyl borate at elevated temperatures:

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$$4NaH + B(OCH_3)_3 \rightarrow NaBH_4 + 3NaOCH_3$$

Aqueous solutions of sodium hydroborate are kinetically stable at high pH; however, acidic solutions decompose rapidly to boric acid and hydrogen:

$$BH_4^- + H^+ + 3H_2O \rightarrow 4H_2 + H_3BO_3$$

Sodium hydroborate is often used as a reducing agent in both inorganic and organic syntheses. Two typical reactions are:

$$3BH_4^- + 4HSnO_2^- + 7H^+ + H_2O \rightarrow 4SnH_4 + 3H_3BO_3$$

$$4R_2C{=}O + BH_4^- + 4H_2O \rightarrow 4R_2CHOH + B(OH)_4^-$$

When ether solutions of sodium hydroborate are heated at 100° with diborane, the octahydrotriborate ion, $B_3H_8^-$, is formed:

$$B_2H_6 + NaBH_4 \rightarrow NaB_3H_8 + H_2$$

By suitable changes in reaction time, temperature, diborane pressure, and solvent, these same reagents will yield various anions of the general formula $B_nH_n^{2-}$ (n ranging from 6 to 12). These ions are remarkably resistant toward hydrolysis, and their boron skeletons remain intact

throughout a wide variety of reactions in which the hydrogen atoms are replaced by other groups.

Lithium hydride is an intermediate in the preparation of lithium aluminum hydride (lithium hydroaluminate), $LiAlH_4$:

$$4LiH + AlCl_3 \rightarrow LiAlH_4 + 3LiCl$$

Both $LiAlH_4$ and $NaAlH_4$ can be prepared directy from the elements:

$$M + Al + 2H_2 \xrightarrow[\substack{1000\text{-}5000 \text{ psi} \\ 120\text{-}150^\circ}]{\text{ether solvent}} MAlH_4$$

Lithium aluminum hydride is an important reagent in synthetic chemistry; it is a much more powerful reducing agent than hydroborates, and it reacts vigorously with protonic solvents such as alcohols.

Metallic Hydrides.- The binary hydrides of the transition metals have metallic properties and are usually obtained as powders or brittle solids which are dark and metallic in appearance, and which have electrical conductivities and magnetic properties characteristic of metals. Most of these hydrides have non-stoichiometric compositions; the stoichiometries and properties depend on the purity of the metals used in the preparations. An early theory

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of the metallic hydrides was implied in their classification, along with carbides, nitrides, borides, etc., as "interstitial." It was believed that the hydrogen entered the interstices of the metal lattice as atoms. This theory, which suggested very little interaction between the interstitial atoms and the metal, soon became untenable in the light of structural studies. Most hydrides do not have their metal atoms in the same positions as in the parent metal (although some hydrides have metal atom positions corresponding to a high-temperature modification of the metal). Some structural data for metals and hydrides are given in Table II. Other facts which must be explained by any theory of metallic hydrides are the following:

(a) In several of the hydrides, the bonded hydrogen is very mobile, and has only a small activation energy of diffusion.

(b) The magnetic susceptibility of palladium falls as hydrogen is added, and the material becomes diamagnetic at a composition of about $PdH_{0.6}$.

(c) If a potential is applied accross a filament of a hydride such as one of palladium, titanium, or tantalum, the hydrogen migrates toward the negative electrode.

Table II

Structures of Metals and their Hydrides

Metals	Limiting Formulas and Structures* of the Hydrides				
Sc	hcp	ScH_2	ccp		
Y	hcp	YH_2	ccp	YH_3	hcp
La	hcp with double c axis (ccp)	LaH_2	ccp	LaH_2	ccp
Ce	ccp (hcp)	CaH_2	ccp	CeH_3	ccp
Pr,Nd	hcp with double c axis	MH_2	ccp	MH_3	hcp
Sm	rhombohedral	SmH_2	ccp	SmH_3	hcp
Eu	bcc	EuH_2	ortho-rhombic		
Gd,Tb,Dy,Ho, Er,Tm,Lu	hcp	MH_2	ccp	MH_3	hcp
Yb	ccp	YbH_2	ortho-rhombic	YbH_3	ccp
Ac	ccp	AcH_2	ccp		
Th	ccp (bcc)	ThH_2	ccp	Th_4H_{15}	bcc
Pa	bct	PaH_3	β-W		
U	complex	UH_3	β-W		
Np,Pu		MH_2	ccp	MH_3	hcp
Ti,Zr,Hf	hcp (bcc)	MH_2	ccp		
V,Nb,Ta	bcc	MH	bcc		
Cr	bcc	CrH	hcp	CrH_2	bcc
Pd	ccp	PdH	ccp		

(continued)

(note to Table II)

* hcp = hexagonal close packed, ccp = cubic close packed, bcc = bocy-centered cubic, bct = body-centered tetragonal. These structures correspond to the positions of the metal atoms in the hydrides; the actual structures may be somewhat expanded from the close packed form.

In one theory which is used to describe the metallic hydrides, hydrogen atoms are assumed to have lost their electrons to the conduction band of the metal structure and to be present in the lattice as protons. Thus the compound is like an alloy. In another theory of metallic hydrides, the hydrogen atoms are assumed to have acquired electrons from the conduction band and to be present as hydride ions. The partially depleted conduction band gives residual metallic bonding in the compound. Thus the compound can be described as a saline hydride with metallic properties. The various properties of metallic hydrides have been explained on the basis of both theories.

The titanium group metals, and probably vanadium and niobium, form hydrides with stoichiometries approaching MH_2. The vanadium group metals form hydrides with stoichiometries approaching MH. The latter hydrides are relatively stable and have body-centered cubic structures, or structures slightly distorted from those. The MH_2 hydrides are less stable and are presumably cubic, with the fluorite structure. Chromium forms CrH, with the nickel arsenide structure, and perhaps a dihydride. Palladium forms $PdH_{0.6}$ at room temperature; the hypothetical PdH would have the sodium chloride structure. Copper forms CuH, prepared by the reaction of aqueous Cu^{2+} with hypophosphorous acid, which decomposes irreversibly and has the wurtzite structure.

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All the lanthanide elements and scandium and yttrium form hydrides by direct exothermic reaction with hydrogen. They all form dihydrides and trihydrides, except scandium and europium (which form only dihydrides) and ytterbium (which forms a dihydride and an unstable $YbH_{2.5}$). All the lanthanide hydrides (except EuH_2 and YbH_2, which are like CaH_2) are less dense than the metal, in contrast to saline hydrides. The typical dihydrides have magnetic and electrical properties characteristic of metals, whereas the trihydrides are more like saline hydrides.

The hydrides of the actinides reflect the general chemistry of the actinides; that is, the chemistry is unique at the beginning of the series and resembles the lanthanides after about uranium. The well characterized phases are ThH_2, Th_4H_{15}, PaH_3, UH_3, NpH_2, NpH_3, PuH_2, PuH_3, and AmH_2.

Transition-Metal Hydride Complexes.- Until the late 1950's, very few hydrides were known in which hydrogen atoms were covalently bonded directly to transition metal atoms. Since then, numerous compounds of this type have been characterized. Most of them have the general formula MH_xL_y, where M is a transition metal and L is a ligand capable of acting both as a σ donor and a π acceptor. Examples are $K_3[CoH(CN)_5]$ (a homogeneous hydrogenation catalyst for the

conversion of alkynes to alkenes), trans-$PtHCl[P(C_2H_5)_3]_2$ (a compound so stable that it can be sublimed in a vacuum), $OsHCl_2[P(C_4H_9)_2C_6H_5]_3$ (a complex with an odd number of electrons and therefore paramagnetic), and K_2ReH_9 (remarkable because the transition metal is bonded to an unusually large number of hydrogen atoms, and no other ligands).

Some transition metal ions and complexes react directly with molecular hydrogen to form complexes containing metal-hydrogen bonds. Three distinct mechanisms for this type of reaction have been recognized, which are illustrated by the following reactions:

Heterolytic splitting of H_2:

$$RuCl_6^{3-} + H_2 \rightleftharpoons RuHCl_5^{3-} + H^+ + Cl^-$$

Homolytic splitting of H_2:

$$2Co(CN)_5^{3-} + H_2 \rightleftharpoons 2CoH(CN)_5^{3-}$$

Insertion of H_2 (dihydride formation):

$$IrCl(CO)[P(C_6H_5)_3]_2 + H_2 \rightleftharpoons IrH_2Cl(CO)[P(C_6H_5)_3]_2$$

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Heterolytic splitting is a substitution reaction (in which a ligand is replaced by a hydride ion) without change in the formal oxidation state of the metal. In homolytic splitting and insertion, hydride formation is accompanied by the formal oxidation of the metal. These types of reactions are believed to be involved in metal-catalyzed reactions of hydrogen with olefins, etc. One of the most active hydrogenation catalysts known is the complex $RhCl[P(C_6H_5)_3]_3$. An unsaturated organic substance, S, can displace one of the $P(C_6H_5)_3$ groups, forming $RhCl(S)-[P(C_6H_5)_3]_2$. The latter can add hydrogen to form $RhH_2Cl(S)[P(C_6H_5)_2]_2$, in which the organic substance and the hydrogen can interact and yield the hydrogenated product.

Transition metal hydride complexes are generally prepared by the reduction of transition metal compounds in the presence of π-bonding ligands. Besides the obvious reducing agents BH_4^- and $LiAlH_4$, reagents such as alcoholic KOH, hydrazine, and hypophosphorous acid have been used. A typical procedure is to treat the anhydrous halide with $NaBH_4$ or $LiAlH_4$ in an ether solvent. For example,

$$C_5H_5Fe(CO)_2Cl \xrightarrow[\text{tetrahydrofuran}]{NaBH_4} C_5H_5Fe(CO)_2H$$

$$\underline{\text{trans}}\text{-Pt(PR}_3\text{)}_2\text{Cl}_2 \xrightarrow[\text{tetrahydrofuran}]{\text{LiAlH}_4} \underline{\text{trans}}\text{-Pt(PR}_3\text{)}_2\text{(H)Cl}$$

Many hydrides and carbonyl hydrides can be prepared by the treatment of anhydrous halides with triphenylphosphine or triphenylarsine in alcohol. For example,

$$(NH_4)_2OsCl_6 \xrightarrow[\substack{\text{diethyleneglycol}\\ \text{monomethyl ether}}]{PPh_3;\ 165^\circ} HOsCl(CO)(PPh_3)_3$$

In early research, many of the transition metal hydride complexes were believed to involve low oxidation states of the metals, and the presence of hydrogen was not suspected. We now know that the hydrogen in such compounds can be identified by an infrared absorption, due to the M-H stretch, in the range 1600-2250 cm^{-1} and a proton magnetic resonance signal at very high field, corresponding to τ values in the range 15-50.

Structural studies show that the hydrogen atom usually acts as an ordinary monatomic uninegative ligand, i.e., as H^-. In some metal cluster hydride complexes, the hydrogen atoms are shared between adjacent metal atoms. For example, it is believed that, in the compound $[ReH(CO)_4]_3$, the hydrogen atoms occupy bridging positions along the edges of an Re_3 triangle.

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Covalent Hydrides.- These hydrides contain hydrogen atoms covalently bonded to elements such as the elements of main groups 3, 4, 5, 6 and 7 of the periodic table. The hydrides of the more electropositive of these elements, e.g., boron, react as if the hydrogen atoms had "hydridic" character (that is, as if they were H^- ions). Thus these hydrides react with water to form hydrogen: $B_2H_6 + 6H_2O \rightarrow 6H_2 + 2H_3BO_3$. Hydrides of the more electronegative elements such as those of groups 6 and 7 react as if the hydrogen atoms had protonic (H^+) character. Thus these react with bases to form salts or adducts: $HCl + C_5H_5N \rightarrow (C_5H_5NH)Cl$. Hydrides of the elements of intermediate electronegativity, such as those of group 4, have no marked hydridic or protonic character. These hydrides may show, depending on the reagent, either hydridic or protonic character. Thus germane, GeH_4, shows hydridic character in the reaction with hydrogen bromide, $GeH_4 + HBr \rightarrow GeH_3Br + H_2$, and shows protonic character in its reaction with sodium amide, $GeH_4 + NaNH_2 \rightarrow NaGeH_3 + NH_3$.

The study of the hydrides of carbon and their derivatives constitutes the enormous discipline of organic chemistry. Carbon atoms are remarkable for their ability to catenate, i.e., to form chains, as in the aliphatic hydrocarbons, or alkanes. Other elements show this ability

to a much lesser extent. Silicon and germanium hydrides analogous to the organic alkanes have been characterized up to $Si_{10}H_{22}$ and $Ge_{10}H_{22}$, i.e., up to silicon and germanium chain lengths of 10. The longest molecular chain hydrides which have been definitely characterized for other elements are Sn_2H_6, HN_3, P_3H_5, As_3H_5, Sb_2H_4, H_2O_3, and H_2S_6.

In some covalent hydrides, a hydrogen atom is bound simultaneously to two separate electronegative atoms. The two atoms are said to be hydrogen bonded. The strongest hydrogen bonds are formed between the small, highly electronegative atoms of fluorine, oxygen, and nitrogen. In the bifluoride ion, HF_2^-, the hydrogen atom bridges two fluorine atoms. In ice, each oxygen atom is surrounded by four other oxygen atoms, and hydrogen atoms are located between the oxygen atoms. When ice melts, some of the hydrogen bonds are broken, allowing the structure to collapse and effecting an increase in density. Hydrogen bonding is very important in biology because it plays a major role in determining the configurations of molecules. Thus the helical (spiral) configuration of polypeptide chains is "knitted" together by N-H···O hydrogen bonds. The boiling points of HF, H_2O and NH_3 are much higher than those of their heavier analogs, HCl, H_2S and PH_3. This

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apparent discrepancy can be explained by the extensive hydrogen bonding in the liquid states of the former compounds. In order to provide the thermal energy to break the hydrogen bonds and to permit vaporization, a high temperature is required.

When a strongly acidic compound such as HCl or HNO_3 dissolves in water, the proton (H^+) separates completely from the anion (e.g., Cl^- or NO_3^-) and interacts strongly with the water molecules. The proton is very strongly hydrated by one water molecule to form the oxonium ion, H_3O^+, and this in turn is hydrogen-bonded to other water molecules, forming species such as $H_9O_4^+$. This oxidation potential of the hydrogen electrode couple, $\frac{1}{2}H_2 \rightarrow H^+ + e^-$, is taken by convention to be zero. All metals with positive oxidation potentials can in principle displace hydrogen from a strong acid solution: $M + nH^+ \rightarrow M^{n+} + \frac{n}{2}H$. Metals with negative oxidation potentials are inert toward the aqueous hydrogen ion.

The hydrides of beryllium, aluminum, gallium, zinc, cadmium, and mercury are relatively difficult to prepare and handle. They have been obtained only in the form of relatively nonvolatile polymers or solvates. Beryllium hydride has been prepared in fairly high purity (99.5%) by removing BH_3 from $Be(BH_4)_2$ with triphenylphosphine:

$$2Ph_3P + Be(BH_4)_2 \xrightarrow[180^\circ]{\text{sealed tube}} 2Ph_3PBH_3 + BeH_2$$

Aluminum hydride is formed by the reaction of $LiAlH_4$ with aluminum chloride:

$$2LiAlH_4 + AlCl_3 \xrightarrow{\text{ether}} 3LiCl + 4AlH_3$$

The polymeric hydride holds ether and other donor solvents tenaciously. Gallium hydride has been prepared by the reaction of $(CH_3)_3NGaH_3$ (prepared from $LiGaH_4$ and $(CH_3)_3NHCl$) with BF_3:

$$(CH_3)_3NGaH_3(g) + BF_3(g) \xrightarrow{-15^\circ} GaH_3(\ell) + (CH_3)_3NBF_3(g)$$

The GaH_3 (probably polymeric) melts at about -20° and decomposes above -15°. The preparation of etherates of ZnH_2, CdH_2, and HgH_2 has been reported by the low-temperature reactions of the iodides or methyl compounds with $LiAlH_4$. Decomposition temperatures of 25, -20, and -125° have been reported.

The hydrides of boron have always fascinated chemists because their structures cannot be explained by classical bonding theory. For example, the simplest boron hydride, diborane (B_2H_6), has only twelve valence electrons to hold

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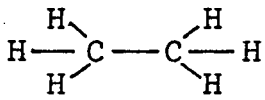
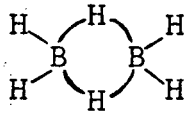
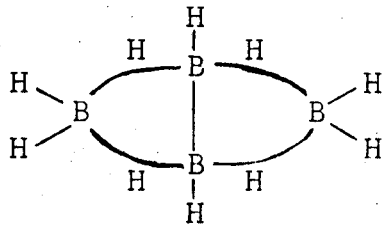
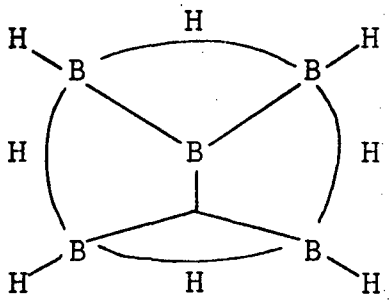
its eight atoms together, whereas the hydrocarbon ethane, C_2H_6, has fourteen valence electrons. Fourteen valence electrons provide seven two-atom electron-pair bonds for the structure  , but only six such bonds could be formed for B_2H_6. Indeed B_2H_6 does not have an ethane-like structure, but rather a structure in which two of the hydrogen atoms act as bridges between the boron atoms:  . By assigning a pair of electrons to each of the three-atom B-H-B bridge bonds and a pair to each of the two-atom B-H bonds, we can explain the structure in terms of only twelve valence electrons. By using similar combinations of two-atom and three-atom bonds, one can rationalize the structures of the other boron hydrides, e.g., B_4H_{10}, B_5H_9, B_5H_{11}, B_6H_{10}, $B_{10}H_{14}$, etc. For example, the bonding in tetraborane, B_4H_{10}, is explained in terms of four B-H bonds, one B-B bond, and four B-H-B bonds:



Similarly, the bonding in pentaborane-9, B_5H_9, can be described in terms of five B-H bonds, four B-H-B bonds and a resonance hybrid of two B-B bonds and one B-B-B bond. One structure which can be written is:



Similar descriptions of the bonding in highly symmetrical species such as the icosahedral $B_{12}H_{12}^{2-}$ ion can be given, but in such cases molecular orbital theory is probably more satisfactory.

Carboranes are "electron deficient" hydrides containing both boron and carbon atoms, usually in a polyhedral framework. They have generally been prepared by reactions of acetylene or substituted acetylenes with boron hydrides, as in the following reactions:

$$B_5H_9 + RC{\equiv}CR + \text{base} \rightarrow B_4H_6C_2R_2 + \text{base:}BH_3$$

$$B_5H_9 + C_2H_6 \xrightarrow[\text{discharge}]{\text{silent electric}} B_3C_2H_5, B_4C_2H_6, B_5C_2H_7, \text{ etc.}$$

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$$B_{10}H_{12}\cdot 2(C_2H_5)_2S + HC{\equiv}CH \rightarrow B_{10}C_2H_{12} + 2(C_2H_5)_2S + H_2$$

The most thoroughly studied carborane is $B_{10}C_2H_{12}$, in which the boron and carbon atoms form the corners of an icosahedron, analogous to the B_{12} framework in $B_{12}H_{12}^{2-}$. It is an extremely stable molecule, with the framework resisting attack by strong acids, alkalies and oxidizing agents.

Some properties of some of the volatile hydrides are given in Table III.

Table III

Properties of Some Volatile Hydrides

Formula	Melting Point °C	Boiling Point °C	ΔH° of formation of gas kcal/mole
B_2H_6	-165	- 90	7.5
B_4H_{10}	-120	16.1	13.8
B_5H_9	- 46.1	58.4	15.0
B_5H_{11}	-123.3	63	22.2
B_6H_{10}	- 63.2	108	19.6
$B_{10}H_{14}$	100	--	4.4
SiH_4	-185	-111.2	7.3
Si_2H_6	-129.3	- 14.2	17.1
Si_3H_8	-114.8	53.0	25.9
GeH_4	-165.9	- 88.51	21.6
Ge_2H_6	-109	29	38.7
Ge_3H_8	-101.8	110.5	53.6
SiH_3GeH_3	-119.7	7.0	27.8
SnH_4	-150	- 51.8	38.9
PbH_4	--	ca.- 13	60

(continued)

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(Table III, continued)

Formula	Melting Point °C	Boiling Point °C	ΔH° of formation of gas kcal/mole
PH_3	-133.8	- 87.74	1.3
P_2H_4	- 99	63.5	5.0
SiH_3PH_2	< -135	12.7	2
AsH_3	-116.9	- 62.5	15.9
SbH_3	- 88	- 18.4	34.7
BiH_3	--	ca. 17	66

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