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The Mass Spectra of Some Volatile Hydrides^{*1}

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Abstract

The hydrides of group VB and VB elements, except CH_4 and NH_3 , have been prepared and, except for PbH_4 and BiH_3 , their mass spectra studied. Fragmentation patterns and appearance potentials of the positive ions from these hydrides are reported. The ions have been tested for excess kinetic energy and this energy was found to be negligible. The average M-H bond energies and the ΔH_f° obtained from appearance potential data agree well with values obtained by other methods. Evidence is cited for the existence of distibine.

Introduction

Thermochemical studies of the hydrides of the group IVB and VB elements have been made employing mass spectrometric techniques. From appearance potential data the heat of formation and average bond energies have been calculated, and these thermodynamic properties are compared and correlated for the various hydrides within their respective groups.

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Experimental

Silane was prepared by the reduction of silicon tetrachloride with lithium aluminum hydride according to the method of Finholt, et al.² Mass spectral analysis showed no higher homologs present.

Germane, digermane and trigermane were prepared by the method of Jolly and Drake.³ Their report furnished some missing details to a similar study that was being conducted independently in these laboratories.

Phosphine was prepared by the method of Gunn and Green.⁴ No diphosphine was observed mass spectrometrically in this preparation after the product had been purified by conventional techniques.⁵

Diphosphine was prepared as a by-product of the disproportionation of phosphorous acid. According to Remy⁶ the principle products are phosphoric acid and phosphine. However, fractional distillation under high vacuum produced a residue which was diphosphine in pure form according to mass spectral assays.

The reduction of an acidic solution of arsenic trichloride with an aqueous sodium borohydride solution was used to prepare arsine. Diarsine, As_2H_4 , was formed as a by-product in this reaction but was easily separated from the arsine by conventional techniques.⁵

The preparation of stibine was essentially the same as that for arsine except that antimony trichloride was used in place of arsenic trichloride. An early preparation analyzed in a 180° mass spectrometer confirmed the presence of stibine in the first fraction collected but also showed the presence of distibine, Sb_2H_4 , in a second fraction from which

the stibine had been removed by fractional distillation. However, subsequent assays of other preparations in the 60° instrument described below in which the sample system and ion source geometry differed from that of the 180° instrument, failed to positively corroborate the early observation. However, a small ion peak in the distibine mass range, presumably due to Sb_2^+ , was observed. This can be explained since the amount of distibine in the first observation was very small and the source efficiency of the 60° instrument less than that of the 180° instrument. None-the-less, this peak was definitely a part of the sample being introduced and the failure to observe other ion currents was attributed to a low signal-to-noise ratio during the time of observation. Further evidence for the existence of distibine was obtained by the following procedure. After the stibine had been collected, the vacuum line, in which the preparation was carried out, was thoroughly evacuated at a temperature where stibine is a gas, and then isolated. The vacuum line was then allowed to warm to room temperature. A black metallic antimony mirror was formed in the system. The antimony could not have been from stibine since this hydride was removed by the evacuation. Also, the rate of formation of this mirror was much faster than any previously observed rate of decomposition of stibine.

Attempts to prepare bismuthine by reducing an acidic solution of bismuth trichloride with sodium borohydride and to introduce it into the mass spectrometer failed. A metallic mirror was always obtained when the sample was transferred from the vacuum line to the sample bulb.

These mirrors gave positive spot tests for bismuth. The bismuth mirrors also formed when the vacuum system had been lined with a paraffin coating. This rules out the possibility that the bismuthine decomposition depends upon the presence of glass and indicates that bismuthine is decomposed thermally at room temperature.

All attempts to introduce distannane and plumbane into the present mass spectrometer failed due to the instability of these hydrides at the ion source temperature in the mass spectrometer.

The mass spectrometer used is a sector single deflection instrument of 15 cm radius using a vibrating reed electrometer as an ion current detector. The analyzer tube and inlet are all-metal systems. Provisions have been made so that the repeller potential in the ion source may be varied continuously by means of helical potentiometer. This enables semi-quantitative information to be obtained about the excess kinetic energy of the fragment ions by Taubert's method.⁷ The ionizing electrons have a Maxwellian thermal energy distribution. A more complete description of the instrument is given elsewhere.⁸ All spectra were made with the following conditions prevailing in the ion source: ionizing current 95 μ a; ion accelerating voltage 1625 v.; repeller voltage +1.0 v.; electron accelerating voltage, (E. A. V.), 60 v. The appearance potentials were made under the same conditions except the E. A. V. was varied in 0.1 v. steps. The methods of obtaining and plotting the ionization efficiency curves have been described.⁹ Extrapolation of straight line portions of the ionization efficiency curves was the method¹⁰ used for

obtaining the appearance potentials. While this method has certain disadvantages, experimental problems with these compounds made it adequate; therefore, no attempt was made to obtain A.P.'s by more refined techniques.

Results and Discussion

Silane

Fragmentation patterns. - The positive ion fragmentation pattern of silane is given in column two of Table I. These results disagree

TABLE I

FRAGMENTATION PATTERNS OF SOME VOLATILE HYDRIDES^a

Ion	Si	Ge	Sn	P	As	Sb
MH_4^+	1	0.5	0.1	-	-	-
MH_3^+	78.1	93.7	100.0	68.0	66.5	73.9
MH_2^+	100.0	100.0	53.6	25.4	22.3	31.8
MH^+	20.3	28.3	21.5	100.0	100.0	100.0
M^+	20.7	47.5	70.6	40.2	40.4	63.8

^a E. A. V. = 60 v.

with those reported by Neuert and Clasen¹¹ in the observation of a negligible ion current for the parent ion, SiH_4^+ , and an appreciable current for the Si^+ fragment. This discrepancy can be explained on the basis of an error in mass assignment by Neuert and Clasen. The possibility of similar errors in this work was eliminated by calibrating the mass scale with N_2 and O_2 , according to a procedure fully described

elsewhere.¹² All fragmentation patterns given in this report have been corrected for variations in gas flow and pressure.

Appearance potentials (A. P.). - The ionization efficiency curves were obtained by the method discussed by Flesch and Svec⁹ using argon as an internal standard. The curves are reproduced in detail in reference 8. The values of the A. P.'s of the various processes occurring in the ion source are shown in column three of Table II.

TABLE II

A. P. AND PROCESSES OF THE GROUP IVB HYDRIDES

Ion	Process	Si	Ge	Sn
MH_4^+	$\text{MH}_4 \rightarrow \text{MH}_4^+ + e$	11.4 ^a ev	10.5 ^a ev	11.7 ^a ev
MH_3^+	$\text{MH}_4 \rightarrow \text{MH}_3^+ + \text{H} + e$	11.8 $\pm$ 0.2	10.8 $\pm$ 0.3	11.9 $\pm$ 0.2
MH_2^+	$\text{MH}_4 \rightarrow \text{MH}_2^+ + \text{H}_2 + e$	12.1 $\pm$ 0.2	11.0 $\pm$ 0.2	12.1 $\pm$ 0.2
	$\text{MH}_4 \rightarrow \text{MH}_2^+ + 2\text{H} + e$	16.5 $\pm$ 0.3	15.4 $\pm$ 0.3	16.4 $\pm$ 0.3
MH^+	$\text{MH}_4 \rightarrow \text{MH}^+ + \text{H}_2 + \text{H} + e$	16.1 $\pm$ 0.2	11.3 $\pm$ 0.2	13.3 $\pm$ 0.2
	$\text{MH}_4 \rightarrow \text{MH}^+ + 3\text{H} + e$	20.4 $\pm$ 0.5	16.8 $\pm$ 0.3	17.3 $\pm$ 0.5
M^+	$\text{MH}_4 \rightarrow \text{M}^+ + 2\text{H}_2 + e$	11.7 $\pm$ 0.2	10.7 $\pm$ 0.2	11.4 $\pm$ 0.2
	$\text{MH}_4 \rightarrow \text{M}^+ + \text{H}_2 + 2\text{H} + e$	16.4 $\pm$ 0.2	14.1 $\pm$ 0.5	15.5 $\pm$ 0.2
	$\text{MH}_4 \rightarrow \text{M}^+ + 4\text{H} + e$	20.8 $\pm$ 0.2	18.3 $\pm$ 0.3	19.5 $\pm$ 0.2

^aEstimated according to the method described by Saalfeld.¹²

The appearance potential of SiH_4^+ was estimated by the method described by Saalfeld and Svec.¹² All errors cited in this work are estimates of the absolute error and do not represent the reproducibility of the data. The fragment ions were checked for excess kinetic energy

by the method of Taubert⁷ and this energy was found to be negligible. Using the A.P. of the least energetic process leading to the formation of Si^+ and the ΔH_f° of Si^+ from Latimer,¹⁴ ΔH_f° and the average Si-H bond energy are found to be 8.5 Kcal/mole and 72.2 Kcal/mole, respectively. These values are in good agreement with the calorimetric values reported by Gunn and Green.⁴ Using the data cited in Table II the dissociation energies of the various ion fragments of SiH_4 were calculated and these are shown in column two of Table III.

TABLE III

ION FRAGMENT BOND DISSOCIATION ENERGIES VOLATILE HYDRIDES

Ion	Si	Ge	Sm	P	As	Sb
$\text{MH}_3^+ - \text{H}$	0.4 ev	0.3 ev	0.2 ev	-	-	-
$\text{MH}_2^+ - \text{H}$	4.7	4.6	4.5	2.9 ev	2.4 ev	1.9 ev
$\text{MH}^+ - \text{H}$	3.9	1.4	0.9	2.0	2.3	2.4
$\text{M}^+ - \text{H}$	0.4	1.5	2.2	4.4	2.6	2.5

The low value for the bond dissociation energy of $\text{SiH}_3^+ - \text{H}$ explains the observation of a negligible amount of parent ions. The large values for the $\text{SiH}_2^+ - \text{H}$ and $\text{SiH}^+ - \text{H}$ energies corroborate other observations in the fragmentation patterns.

Germane

Fragmentation patterns. - The fragmentation pattern of germane prepared from the separated isotope ^{74}Ge in the form of germanium IV oxide is shown in column three of Table I. These results agree well with the fragmentation pattern obtained from germane prepared from naturally

abundant germanium dioxide. Again the results disagree with those of Neuert and Clasen¹¹ presumably for the same reason as given above. The mass scale covering germane in this investigation was checked using chlorine as a calibrating gas.

The fragmentation patterns of digermane and trigermane are shown in Tables IV and V, respectively. For all compounds reported in this study the monoelemental types, i. e. (MH_4 and MH_3), have been completely separated from the dielemental types (M_2H_6 and M_2H_4) as shown by mass spectrometric analysis.

TABLE IV
FRAGMENTATION PATTERNS OF SOME DIELEMENTAL HYDRIDES

Ion	Ge	P	As	Sb
M_2H_6^+	43.0			
M_2H_5^+	25.2			
M_2H_4^+	68.6	6.2	3.0	0.1
M_2H_3^+	28.3	11.7	1.4	14.9
M_2H_2^+	100.0	32.6	7.3	41.0
M_2H^+	60.4	25.4	21.7	62.0
M_2^+	63.0	100.0	100.0	100.0
MH_4^+	5.4	-	-	-
MH_3^+	32.1	-	-	-
MH_2^+	34.5	-	-	-
M^+H	11.8	-	-	-
M^+	41.2	-	-	-

TABLE V

Ge ₃ H ₈ FRAGMENTATION PATTERNS					
Ion	Ge ₃ H ₈ ⁺	Ge ₃ H ₇ ⁺	Ge ₃ H ₆ ⁺	Ge ₃ H ₅ ⁺	Ge ₃ H ₄ ⁺
Relative Peak Height	26	16	3	3	12
Ion	Ge ₃ H ₃ ⁺	Ge ₃ H ₂ ⁺	Ge ₃ H ⁺	Ge ₃ ⁺	
Relative Peak Height	12	5	100	100	

In Table IV it should be noted that an appreciable amount of GeH₄⁺ was observed in the digermane spectrum while it was not so observed in that of germane. Since the magnitude of this peak varied linearly with the square root of the pressure, it is attributed to a secondary process occurring in the ion source. The Ge⁺ fragment, formed from Ge₂H₆, was found to have excess kinetic energy.

The spectrum of trigermane was measured on the instrument described by Flesch and Svec⁹ and was not prepared from the separated isotope preparation. None of the ion fragments from trigermane containing Ge₂ or Ge are reported since these ion currents were too small for sensible measurements.

The mass scale in the Ge₂H₆ region was calibrated with chromyl chloride, CrO₂Cl₂, the spectrum of which has been characterized by Flesch and Svec.⁹ The corresponding mass scale for the trigermane region was not calibrated.

Appearance potentials. - The ionization efficiency curves for germane are also given in detail in reference 8. The A.P. and their associated

processes are shown in column four of Table II. Krypton was used as an internal standard to calibrate the voltage scale.

The A. P. of GeH_4^+ was estimated in the same manner as the A. P. for SiH_4^+ . The A. P. of the least energetic process from which Ge^+ is formed when combined with the $\Delta H_f^\circ (\text{Ge}^+)^{14}$ gives $\Delta H_f^\circ (\text{GeH}_4)$ and the average Ge-H bond energy. These values, 20.8 Kcal/mole and 68.3 Kcal/moles, respectively, concur with the calorimetric values of Gunn and Green⁴ giving additional support to the fact that the kinetic energy observed by Taubert's method⁷ was negligible. The data presented in Table II also enable the calculation of the bond dissociation of the ion fragments from GeH_4 which are given in column three of Table III.

Stannane

The results of the SnH_4 study, conducted on another mass spectrometer, are reported elsewhere.¹³ The value for $\Delta H_f^\circ (\text{SnH}_4)$ calculated from these data is 23.0 Kcal/mole, which is lower than the value reported by Gunn and Green. This difference may be reasonably explained by the presence of excess kinetic energy in the Sn^+ fragment. With the instrument used in the SnH^+ study there was no way to detect this energy and it is suspected that the Sn^+ fragment might have contained excess energy in these earlier measurements.

Jolly¹⁵ has reported the existence of distannane, Sn_2H_6 , and while the existence of this hydride has been corroborated in these laboratories, all attempts to introduce preparations of it into the mass spectrometer have failed due to the instability of the compound at the temperature of the ion source.

Phosphine

Fragmentation pattern. - The fragmentation pattern of PH_3 is given in column five of Table I. The mass scale in this case was calibrated with the S^+ ion currents from sulfur dioxide. Column three of Table IV shows the fragmentation pattern of diphosphine. This mass scale was calibrated with the parent ion currents from sulfur dioxide. No values are listed in Table IV for the monophosphorous ions because their abundances were so small that the values for the relative peak heights could not be reproduced.

The fragmentation pattern of diphosphine suggests that the P-P bond is the strongest bond in diphosphine. This hypothesis could not be tested further since any A. P. measurement would be meaningless because of the low intensities of the monophosphorous ions and the numerous processes occurring in the ion source.

Appearance potentials. - The ionization efficiency curves for ions from phosphine are likewise given in detail in reference 3. Argon was used as an internal standard to calibrate the voltage scale. The A. P.'s and their processes are given in column three of Table VI.

Using $\Delta H_f^\circ (\text{P}^+)$ as given by Latimer¹⁴ and the least energetic process given for the formation of P^+ , values of 1.4 Kcal/mole and 76.8 Kcal/mole are obtained for ΔH_f° and the average P-H bond energy, respectively. The kinetic energy of fragment ions was found to be negligibly small. The mass spectrometric values agree well with the

TABLE VI

A. P. AND PROCESSES OF THE GROUP VB HYDRIDES

Ion	Process	P	As	Sb
MH_3^+	$\text{MH}_3 \rightarrow \text{MH}_3^+ + e$	$11.5 \pm 0.3 \text{ ev}$	$12.1 \pm 0.2 \text{ ev}$	$9.9 \pm 0.3 \text{ ev}$
MH_2^+	$\text{MH}_3 \rightarrow \text{MH}_2^+ + \text{H} + e$	14.4 ± 0.2	14.5 ± 0.2	11.8 ± 0.3
MH^+	$\text{MH}_3 \rightarrow \text{MH}^+ + \text{H}_2 + e$	12.4 ± 0.2	12.4 ± 0.2	9.9 ± 0.2
	$\text{MH}_3 \rightarrow \text{MH}^+ + 2\text{H} + e$	16.4 ± 0.3	16.8 ± 0.3	14.2 ± 0.3
M^+	$\text{MH}_3 \rightarrow \text{M}^+ + \text{H}_2 + \text{H} + e$	16.5 ± 0.2	14.8 ± 0.2	12.1 ± 0.2
	$\text{MH}_3 \rightarrow \text{M}^+ + 3\text{H} + e$	20.8 ± 0.3	19.4 ± 0.2	16.7 ± 0.3

results obtained by other methods.⁴ The bond dissociation energies in the ions are shown in column four of Table III. The large value for the $\text{P}^+ - \text{H}$ bond energy supports the observation that PH^+ is the largest ion fragment in the spectrum and the fact that $\text{PH}^+ - \text{H}$ bond energy is the weakest bond in phosphine substantiates PH_2^+ as the least abundant ion in the phosphine spectrum.

Arsine

Fragmentation pattern. - The fragmentation pattern of arsine is shown in column six of Table I. In this case the mass scale has been positively identified by comparison with the known peaks of germane. The fragmentation pattern of diarsine, As_2H_4 , is given in column four of Table IV. The mass scale in this case was calibrated by comparison with the known peaks of digermane. No monoarsenic ions or A.P. are given because of difficulties similar to those discussed for diphosphine. The As-As bond appears to be the strongest in the molecule which is similar to the observation for diphosphine.

Appearance potentials. - The ionization efficiency curves of positive ions from arsine are given in detail in reference 8. The A. P. and their associated processes are given in column four of Table VI. Using the value of $\Delta H_f^\circ (\text{As}^+)$ given by Latimer¹⁴ and the A. P. of the process forming $\text{As}^+ + \text{H}_2 + \text{H}_1$, a value of 15.0 Kcal/mole for $\Delta H_f^\circ (\text{AsH}_3)$ is found. This agrees well with that from Gunn and Green's measurement.⁴ The average As-H bond energy was observed to be 72.9 Kcal/mole. This is slightly higher than the value given by Gunn and Green and might be attributed to excess kinetic energy contained by the As^+ fragment. However, measurements here showed this energy to be negligible. The value is in good agreement with the theoretical value cited by Huggins.¹⁶

Using the data given in Table VI the bond dissociation energies for the positive fragment ions have been calculated and are given in column five of Table III.

Stibine

Fragmentation patterns. - The fragmentation pattern of stibine, SbH_3 , calculated from the known isotope abundances¹⁷ is given in column seven of Table I. Stannane was used to calibrate the mass scale. The fragmentation pattern of distibine, Sb_2H_4 , is given in Table IV. Once again the M-M bond, Sb-Sb in this case, appears to be the strongest in the molecule. The difficulties mentioned for diphosphine and diarsine were enhanced here because of the instability of distibine.

Appearance potentials. - The ionization efficiency curves of stibine are given in detail in reference 8. Column five of Table VI shows the A. P. and their associated processes. Argon was used as an internal

standard to calibrate the voltage scale. Combining Latimer's¹⁴ value for $\Delta H_f^\circ (\text{Sb}^+)$ with the A.P. equal to 12.1 ev, the $\Delta H_f^\circ (\text{SbH}_3)$ was found to be 34.6 Kcal/mole. This is in excellent agreement with Gunn and Green's value.⁴ The average Sb-H bond energy was 61.1 Kcal/mole, also in good agreement with other literature values.⁴ Using the data presented in Table VI the bond dissociation energies may be calculated and these are summarized in column six of Table III. The fact that the SbH^+ was observed as the most abundant ion in the stibine spectrum is supported by the fact that the Sb^+-H bond energy is the largest.

Summary and Conclusions

The fragmentation patterns of the hydrides discussed here may be compared if the ions are corrected for their difference in ionization cross sections. Absolute measurement of these cross sections is very difficult but a convenient method of calculating them is that of Otvos and Stevenson.¹⁸

The comparisons shown in Tables VII and VIII, respectively, are based on the cross sections calculated for methane and ammonia, whose fragmentation patterns have been taken from the compilation of the American Petroleum Institute. The fragmentation patterns of methane and ammonia have been included for completeness. The fragmentation pattern of stannane is taken from the work of Saalfeld and Svec.¹³

The fact that the parent ions MH_4^+ and MH_3^+ decrease regularly from C to Sn and N to Sb concurs with the information on the stability of these molecules obtained from values of the heats of formation. The increasing trend of the amount of the M^+ fragments from C to Sn and

TABLE VII

FRAGMENTATION PATTERNS OF GROUP IVB HYDRIDES ON THE
COMMON IONIZATION CROSS SECTION OF 8.16 FOR METHANE

	C	Si	Ge	Sn
MH_4^+	100.0	0.44	0.18	0.03
MH_3^+	85.7	34.6	34.1	27.4
MH_2^+	16.1	44.3	36.4	14.7
MH^+	8.1	9.0	10.3	5.9
M^+	2.8	9.2	17.3	19.4

TABLE VIII

FRAGMENTATION PATTERNS OF GROUP VB HYDRIDES ON THE
COMMON IONIZATION CROSS SECTION OF 6.84 FOR AMMONIA

	N	P	As	Sb
MH_3^+	100.0	27.7	20.9	17.4
MH_2^+	80.0	10.3	7.0	7.5
MH^+	7.5	40.7	31.5	23.5
M^+	2.2	11.4	12.7	15.0

N to Sb agrees with both the facts that the M-H bond energy decreases with atomic number and with the atomic ionization potential data of these atoms. The heat of formations and average bond energies obtained by various investigators are summarized in Tables IX and X, respectively.

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TABLE IX

HEAT OF FORMATION OF THE IVB AND VB HYDRIDES IN Kcal/mole

	Gunn and Green ⁴	Rossini <u>et al.</u> ¹⁹	This work
CH ₄	-17.9	-17.9	-
SiH ₄	7.3	-14.8	8.5
GeH ₄	21.6	-	21.6
SnH ₄	38.9	-	23.0
NH ₃	-11.0	-11.0	-
PH ₃	1.3	2.21	1.4
AsH ₃	15.9	41.0	15.0
SbH ₃	34.7	-	34.6

TABLE X

AVERAGE M-H BOND ENERGY FOR THE IVB AND
VB HYDRIDES IN Kcal/mole

	Gunn and Green ⁴	Huggins ⁷	Cottrell ²⁰	This work
C-H	99.3	-	98.7	-
Si-H	76.5	79.0	76.0	72.2
Ge-H	69.0	74.0	-	68.3
Sn-H	60.4	71.0	74.0	70.3
N-H	93.4	85	85	-
P-H	76.8	77	77	76.8
As-H	66.8	71	66	72.9
Sb-H	60.9	70	-	61.1

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