RADIATION CHEMISTRY AND MASS SPECTROMETRY OF
VOLATILE SILANES AND GERMANES

Progress Report

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June 1, 1974 - May 31, 1975

PREPARED FOR THE ENERGY RESEARCH AND DEVELOPMENT ADMINISTRATION

UNDER CONTRACT NO. E(11-1)-3416

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Preface

Our continuing study of the high-energy radiation chemistry of silane and germane systems may, as in the past, be subdivided into three types of investigation as follows:

- A. Gamma-ray radiolyses of silanes and germanes and of mixtures of these substances with organic compounds, free-radical scavengers and ion scavengers.
- B. Mass-spectrometric studies of the ion-molecule reactions occurring when systems corresponding to those of (A) are subjected to ionization by electron impact. This type of investigation affords an examination and of the purely ionic component of a chemical conversion initiated by ionizing radiation.
- C. Mass-spectrometric studies of the free-radical reactions characteristic of gamma-rays radiolyses of systems corresponding to those of (A). Initiation of reaction in these investigations is by photosensitization so that ionization of the systems does not occur and we may study the pertinent free-radical reactions independently of the ionic processes.

This annual progress report describes work done from June 1, 1974 to May 31, 1975. In any program of such a nature as this, it is necessary to shift emphasis from time-to-time between chemical systems and between types of investigation, depending upon personnel, equipment, and directions suggested by the results obtained. During the report year all work was done with volatile silane systems and while the emphasis was almost equally

divided between A, B, and C above, most progress was made in areas B and C.

This report consists of abstracts of the various topics in this program studied during the report-year. Reprints from the scientific literature that describe completed topics in this program, including earlier reprints that were not available at the time of the last progress report, accompany the progress report as a separately bound appendix.

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1. ION-MOLECULE REACTIONS IN SILANE AND ORGANOSILANE SYSTEMS (COO-3416-19)

Ion-molecule reactions in hydrocarbons and in hydrocarbon derivatives containing oxygen, nitrogen, sulfur, and halogens have been studied extensively.¹ The knowledge acquired from these investigations has been of considerable value, not only to physical chemists and chemical physicists interested in reactions and molecular properties in the gaseous phase, but also to organic chemists in providing some new avenues of thought concerning the condensed phase chemistry of strong acid solutions of alkanes.² An impressive body of knowledge relative to ion-molecule reactions of hydrocarbons and their derivatives containing atoms of Groups V - VII now exists.

However, from the point of view of the inorganic and organometallic chemist, comparatively little is known about the ion-molecule reactions characteristic of compounds in which one or more carbon atoms of a hydrocarbon or hydrocarbon derivative are replaced by another atom or atoms of Group IVa of the periodic table, namely by silicon, germanium, tin, or lead. While structurally similar to hydrocarbons and hydrocarbon derivatives, the chemistry of these inorganic and organometallic compounds is generally quite different.

Present knowledge of ion-molecule reactions of such compounds is restricted almost entirely to silanes and organosilanes. This paper is a review of this body of knowledge, making comparisons wherever pertinent to the ion-molecule reaction chemistry of hydrocarbons.

1. J. L. Franklin, Editor, Ion-Molecule Reactions, Plenum Press, New York 1972.
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2. LONG-LIVED INTERMEDIATE COMPLEXES IN THE REACTION OF Silyl Ions WITH ETHYLENE.

For more than forty years the Ramsberger-Rice-Kassel (later the Ramsberger-Rice-Kassel-Marcus) theory has been the foundation of our understanding of unimolecular reactions. In this theory molecules are energized and de-energized by collision, and produce reaction when they attain certain critical conformations, a process which may be depicted as in (1) and (2), where A^* is a

molecule with enough energy to react and $A^\ddagger$ is one of the critical conformations (transition state) through which reaction occurs. Experimental studies of (1) and (2) have necessarily involved the stable reaction products and their dependence on reaction conditions because the lifetime of the energized molecules A^* are so short.

Recently and for the first time an energized intermediate such as A^* and its decay products have been observed directly in Tandem mass-spectrometric of the gas-phase reactions of SiH_3^+ with C_2H_4 . The reactions occurring are

where E_{Tr} is the translational energy of SiH_3^+ k_a is the internal energy dependent rate constant and an asterisk signifies excess

internal energy. Recent experiments in our tandem mass spectrometer² have shown that in the limit of zero pressure the ion current ratio $I(\text{SiC}_2\text{H}_7^+)/I(\text{SiC}_2\text{H}_5^+)$ has finite values given by

$$\lim_{P \rightarrow 0} \frac{I(\text{SiC}_2\text{H}_7^+)}{I(\text{SiC}_2\text{H}_5^+)} = \frac{e^{-k_a(E)t}}{1 - e^{-k_a(E)t}}$$

Furthermore the limits of this ratio decrease as the translational energy of SiH_3^+ , and hence the internal energy of $\text{SiC}_2\text{H}_7^{+*}$, increases. Since t , the detection time of the instrument, is known $k_a(E)$ may be evaluated directly. The data thus yield the dissociation rate constant $k_a(E)$ for intermediate energized complexes that have not made any collisions.

For $E_{\text{TR}} = 0.5$ eV (center-of-mass) the value obtained is $k_a(E) = 1.3 \times 10^5 \text{ sec}^{-1}$. For $E_{\text{TR}} > 2$ eV the complex is not observed, showing that $k_a > 5 \times 10^6 \text{ sec}^{-1}$ for this collision energy.

These rate constants combined with thermochemical estimates of the energetics involved are in semi-quantitative accord with RRK theory for 24 vibrational degrees of freedom. It is thus suggested that here is a reaction proceeding through an intermediate complex in which all internal degrees of freedom are used.

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1. T. M. Mayer and F. W. Lampe, J. Phys. Chem. 78, 2433, (1974).
2. W. N. Allen and F. W. Lampe, Meeting of American Society of Mass Spectrometry, Houston, Texas, May 25-30, 1975.

3. THE BIMOLECULAR REACTIONS OF SILYL RADICALS

The mutual destruction reaction of silyl radicals has been studied by producing essentially equimolar concentrations of SiH_3 and SiD_3 radicals via the system

The reaction was carried out with mixtures of 95% D_2 at a total pressure of 30 torr in a photolysis cell coupled to a mass spectrometer. The isotopic composition of the disilane product was determined by measurement of the mass spectrum in the mass range of 62-68 as a function of ionizing energy. Ion-current ratios were then extrapolated to 10 eV to obtain a product mass spectrum free from the extensive fragmentation accompanying electron impact on disilane. The relative values for Si_2H_6 and Si_2D_6 were not accurate because of impurities present in the initial monosilanes. However, the distribution for disilane- d_1 through disilane- d_5 as shown in the second column of Table I is very conclusive. The calculated values in the third and fourth columns are obtained by assuming that the silyl radicals first attain a statistical isotopic equilibrium shown by (5).

Table I

Isomeric Distribution of Disilane Parent Ions from Equimolar
Amounts of SiH_3 and SiD_3

Isomer	Experimental	Mechanism I	Mechanism II
Si_2H_6^+	Impurity	33	2
$\text{Si}_2\text{H}_5\text{D}^+$	70	100	12
$\text{Si}_2\text{H}_4\text{D}_2^+$	24	33	60
$\text{Si}_2\text{H}_3\text{D}_3^+$	0	0	100
$\text{Si}_2\text{H}_2\text{D}_4^+$	100	33	60
Si_2HD_5^+	52	100	12
Si_2D_6^+	Impurity	33	2

Mechanism II gives the isotopic disilane yields if combination of the various silyl radicals is the mechanism. Mechanism I assumes that from the statistically equilibrated silyl radicals, silylene radicals, SiH_2 , SiHD , and SiD_2 , are formed in the statistical ratio 1:3:1, followed by insertion of these latter radicals into SiH_4 and SiD_4 , as shown by (6)- (11).

While isotope effects that have been ignored in the calculated distributions undoubtedly play a role, Mechanism I is so much closer to the experimental values than Mechanism II that we may conclude that disilane formation occurs exclusively by silylene insertion reactions at these pressures.

These findings confirm experimental results and calculations recently reported by others.^{1,2}

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4. FORMATION OF DISILANE IN THE D-ATOM INDUCED DECOMPOSITION OF MONOSILANE

The D-atom-induced decomposition of monosilane has been studied by irradiating mixtures of D_2 - SiH_4 -Hg with mercury resonance radiation in a photolysis cell coupled by a pinhole leak to a mass spectrometer. The only significant products observed in this flow system are disilane and hydrogen.

The form of the time dependence of the disilane yield on photolysis time is consistent with the expected mechanism¹ shown by (1)-(7) and the pertinent rate constants.²⁻⁴

Determination of the rate of photosensitized D-atom formation was carried out by replacing SiH_4 by C_2H_4 followed by determination of the rate of butane- d_2 formation through the well known reaction sequence (8)-(10). Quite surprisingly we find by this technique

that the rate of formation of disilane appears to be about three times the rate of formation of D-atoms. This implies some formation of disilane by a short chain reaction. This observation parallels that of previous workers⁵ who found quantum yields of disilane above unity in clean reaction cells. While these effects may be surface-dependent⁵, it is also possible that the following chain (11)-(12) is operating in addition to (1)-(7) above

The occurrence of (11)-(12) is consistent with the observations of Niki and Mains.

While the results are suggestive they are far from conclusive and experiments are in progress to examine this problem in more detail.

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5. THE REACTION OF SILYL RADICALS WITH ACETONE

When acetone is added to the D_2 - SiH_4 mixtures of section 4 and the mixture subjected to mercury photosensitization, the disilane yield is reduced and new mass spectrometric peaks above those of disilane are observed. The identification of these new peaks is not yet complete but we believe they are due primarily to isopropoxysilane and to the dimer and disproportionation products of siloxyisopropyl radicals. This subject is still under investigation and only the most preliminary results are now available. It is suggested that in addition to the reactions (1)-(7) of section 4, the following reactions occur when acetone is added to the system.

6. RADIOLYSIS OF MONOSILANE-NITRIC OXIDE MIXTURES

Several more sets of γ -radiolyses of SiH_4 -NO mixtures have confirmed the earlier conclusion that monosilane and nitric oxide are consumed in an approximately 1:1 ratio by a chain reaction. As shown in Table I the major products of this chain reaction are hydrogen, nitrous oxide and siloxanes represented by the general formula $\text{SiH}_3(\text{OSiH}_2)_n\text{OSiH}_3$, where values of n up to 4 have been observed. Appreciable yields of disilane are seen to persist even at NO concentrations of 20% although $G(\text{Si}_2\text{H}_6)$ is reduced somewhat from the value observed in pure monosilane. This is in direct contrast to the mercury photosensitized reaction of silyl radicals with nitric oxide, in which no disilane formation occurs.^{1,2}

Table I

100-eV Yields in γ -Radiolysis of SiH_4 -NO Mixtures

Dose Rate = 1.07×10^{16} es/cm ³ -hr	
$P^\circ_{\text{SiH}_4}$ = 100 torr	
P°_{NO} = 20 torr	
<u>Molecule</u>	<u>G $\frac{\text{molecules}}{100 \text{ eV}}$</u>
H_2	19.8
SiH_4	83
NO	54*
N_2O	16
Si_2H_6	2.3
$\text{SiH}_3\text{OSiH}_3$	4.4*
$\text{SiH}_3\text{OSiH}_2\text{OSiH}_3$	.8*

* Very uncertain

The results suggest the following radical^{1,2} and ionic³ mechanism subsequent to conversion of the primary products to SiH_3 and SiH_3^+ .

etc.

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ANNUAL LIST OF REPRINTS, THESES, LECTURES, ETC.A. Reprints (Appendix)

1. T. M. Mayer and F. W. Lampe, "Kinematics of Hydride-Ion and Hydrogen-Atom Transfer Reactions in Monosilane". J. Phys. Chem. 78, 2195 (1974).
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- F. W. Lampe, "Ion-Molecule Reactions in the Radiation Chemistry of Silanes". Hahn-Meitner Institut für Kernforschung, Berlin, Germany, June 7, 1974.
- F. W. Lampe, "Ion-Molekül Reaktionen und die Strahlenchemie von Silanen". Kernforschungsanlage, Jülich, Germany, June 10, 1974.
- F. W. Lampe, "Ion-Molecule Reactions in Silanes and Organosilanes". NATO Advanced Study Institute, Biarritz, France. July 3, 1974.
- F. W. Lampe, "Chemical Reactions of Ions in Mass Spectrometers", Department of Chemical Engineering, The Pennsylvania State University, February 18, 1975.
- F. W. Lampe, "Ionic Reactions in the Gas-Phase". Department of Chemistry, The Catholic University of America, Washington, D. C. February 21, 1975.
- F. W. Lampe, "Energy-Rich Complexes in the Reaction of Silyl Ions with Ethylene. 9th Organosilicon Symposium. Case Western Reserve University, Cleveland, Ohio, April 5, 1975.

B. Lectures (Continued)

F. W. Lampe, "Ion-Molecule Reactions in Volatile Silicon Compounds". Naval Research Laboratory, Washington, D. C. May 1, 1975.

W. N. Allen, "Energy-Rich Intermediate Complexes in Ion-Molecule Reactions". American Society of Mass Spectrometry, Houston, Texas, May 29, 1975.

C. Theses

T. M. Mayer, "Chemistry and Kinematics of Ion-Molecule Reactions in Silane Systems", Ph.D. Thesis, The Pennsylvania State University, May 1975.

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* Reprint in the Appendix.