oven. The amounts of the antiozonants that migrated to the surface in the NR vulcanizate were determined by the difference between the amounts of the antiozonants that remained in the rubber vulcanizates before and after the migration. The amounts of the antiozonants remaining in the rubber vulcanizates were analyzed using a gas chromatography after extraction of the antiozonants with THF. Experiments were carried out three times and averaged.

References

1. Cox, W. L. *Rubber Chem. Technol.* **1959**, *32*, 364.
2. Layer, R. W. *Rubber Chem. Technol.* **1966**, *39*, 1584.
3. Erickson, E. R.; Bernstein, R. A.; Hill, E. L.; Kusy, P. *Rubber Chem. Technol.* **1959**, *32*, 1062.
4. Razumovskii, S. D.; Batashova, L. S. *Rubber Chem. Technol.* **1970**, *43*, 1340.
5. Braden, M.; Gent, A. N. *Rubber Chem. Technol.* **1962**, *35*, 200.
6. Layer, R. W.; Lattimer, R. P. *Rubber Chem. Technol.* **1990**, *63*, 426.
7. Lattimer, R. P.; Layer, R. W.; Hooser, E. R.; Rhee, C. K. *Rubber Chem. Technol.* **1991**, *64*, 780.
8. Choi, S.-S. *J. Appl. Poly. Sci.* **1997**, *65*, 117.
9. Choi, S.-S. *J. Appl. Polym. Sci.* **1998**, *68*, 1821.
10. Choi, S.-S. *Bull. Kor. Chem. Soc.* **1998**, *19*, 170.
11. Dimauro, P. J.; Paris, H. L.; Fath, M. A. *Rubber Chem. Technol.* **1979**, *52*, 973.
12. Lederer, D. A.; Fath, M. A. *Rubber Chem. Technol.* **1981**, *54*, 415.
13. Gardiner, J. B. *Rubber Chem. Technol.* **1968**, *41*, 1312.

Synthesis of Oxomolybdenum(V) Complexes with Trifunctional ONO-Donor Ligands

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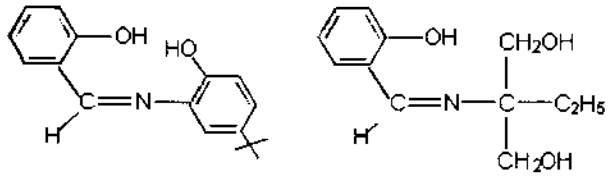
The chemistry of oxomolybdenum complexes with Schiff base ligands has received intense attention because of its relevance to the active sites of molybdoenzymes.[1] It is well-known[2] that the biological redox process of the molybdoenzymes involves oxidation states, IV-VI. In addition, extended X-ray absorption fine structure (EXAFS) spectroscopic studies have implicated the presence of oxygen and nitrogen atoms at the active sites of oxo-transfer molybdoenzymes.[3]

In this context we have taken up N-salicylidene-2-amino-4-*t*-butylphenol ($SabpH_2$) and N-salicylidene-2-amino-2-ethyl-1,3-propanediol ($SapdH_2$) as trifunctional (ONO) dianionic Schiff base ligands. These ligands are of particular interest because chelation may occur either from



$sabpH_2$ $sapdH_2$

ON or ONO sites, in principle. It was also reported[4] that functionalities present in the ligands play a vital role in controlling whether the oxomolybdenum complexes are monomers or polymeric species. Thus, with the aim of simulating many characteristic properties for the active sites of molybdoenzymes, we have synthesized the type of complex $(PyH)[MoO(X)_2(L)]$ (X=Cl, NCS : L=Sabp, Sapd). In this paper, we report the synthesis, spectral properties, and the electrochemical behaviors of the complexes.

Experimental

Materials. Most chemicals used in synthesis were of reagent grade and used without further purification. The complexes $(PyH)[MoOCl_5]$ and $(PyH)[MoO(NCS)_5]$ as starting materials were prepared by literature methods.[5] All the Schiff bases were prepared by the same method as was published previously.[6] All solvents were dried by standard procedures and distilled before use.

Physical Measurements. Elemental analyses for C, H, N were carried out by the Instrumental Center of Kyungpook National University. The melting point measurements were performed by using a Hakke melting point apparatus. The molar conductivities of the complexes were measured by a YSI conductivity bridge (model 31). The IR spectra of solid samples in KBr were recorded on a Mattson Polaris FT-IR. The ^1H NMR spectra in DNSO-d_6 were recorded on a Varian Gemini 200 spectrometer and referenced to TMS (internal). Electronic spectra were obtained on a Shimadzu 160A UV-visible spectrophotometer. Cyclic voltammograms were recorded on an Electrochemical Research System 270/6/0 (EG&G) consisting of PAR 263 Potentiostat/Galvanostat and an Electrochemical Analysis Software 270. The electrochemical studies were conducted in an oxygen-free DMSO solution containing 0.1 M TEAP (tetraethylammoniumperchlorate) as a supporting electrolyte. We employed a three-electrode cell configuration consisting of a platinum

working, a platinium counter, and an Ag/Ag^+ reference electrode.

Preparation of the Complexes (PyH)[MoO(X)$_2$(L)] (X=Cl, NCS : L=Sabp, Sapd). A similar procedure was followed for the preparation of all the complexes. 1 mmol (0.13 g) of salicylaldehyde was added to a methanol solution (5 mL) of 1 mmol of *t*-butyl-2-aminophenol (0.17 g)/2-amino-2-ethyl-1,3-propanediol (0.13 g) and refluxed for 1.5h, resulting in a pale yellow solution. After the solution was cooled to room temperature, 1 mmol of relevant (PyH)[$MoOCl_5$](0.45 g)/(PyH)[$MoO(NCS)_5$](0.56 g) was added with stirring. After complete dissolution of the salt, pyridine 2 mmol (0.16 g) in 2 mL of methanol was slowly added to give a precipitate of the desired compound. After the mixture was allowed to stand for 24h in a refrigerator, the orange-brown compound was filtered, and washed with methanol and diethyl ether. The compound was dried in a vacuum oven (25 mmHg, 25 °C) for 1 day.

Analytical data for the complexes prepared are as follow;

(PyH)[MoOCl$_2$(Sabp)](1). Yield: 0.27 g (51%); mp 224 °C. Anal. Calcd for $C_{22}H_{23}N_2O_3Cl_2Mo$: C, 49.83; H, 4.37; N, 5.28. Found: C, 49.65; H, 4.35; N, 5.42. ΛM (Mho cm^2 mol^{-1}, DMSO): 32. UV/vis (nm. logε, dm^3 mol^{-1} cm^{-1}): 805 (0.90), 461 (2.50), 312 (3.77), 292 (3.67), 266 (3.98). Significant infrared bands (cm^{-1}): 1611 ($\nu_{C=N}$), 971 ($\nu_{Mo=O}$). ^{1}H NMR (200 MHz, DMSO-d$_6$): δ 7.79 (t, 2H, *m*-Py), 8.26 (t, 1H, *p*-Py), 8.82 (d, 2H, *o*-Py).

(PyH)[MoO(NCS)$_2$(Sabp)](2). Yield: 0.33 g (57%); mp 229 °C. Anal. Calcd for $C_{24}H_{23}N_4O_3S_2Mo$: C, 50.09; H, 4.03; N, 9.73; S, 11.14. Found: C, 49.83; H, 4.05; N, 9.94; S, 11.31. ΛM (Mho cm^2 mol^{-1}, DMSO): 42. UV/vis (nm. logε, dm^3 mol^{-1} cm^{-1}): 825 (1.98), 307 (4.16), 288 (4.11), 267 (4.17). Significant infrared bands (cm^{-1}): 2030 ($\nu_{N\equiv C,NCS}$), 1598 ($\nu_{C=N}$), 942 ($\nu_{Mo=O}$), 847 (ν_{C-S}). ^{1}H NMR (200 MHz, DMSO-d$_6$): δ 1.32 (s, 9H, *t*-Bu), 6.72-7.81 (m, 7H, ArH), 8.02 (t, 2H, *m*-Py), 8.53 (t, 1H, *p*-Py), 8.90 (d, 2H, *o*-Py), 9.31 (s, 1H, HC=N).

(PyH)[MoOCl$_2$(Sapd)](3). Yield: 0.25 g (52%); mp 209 °C. Anal. Calcd for $C_{17}H_{21}N_2O_4Cl_2Mo$: C, 42.17; H, 4.37; N, 5.79. Found: C, 42.32; H, 4.21; N, 5.69. ΛM (Mho cm^2 mol^{-1}, DMSO): 31. UV/vis (nm. logε, dm^3 mol^{-1} cm^{-1}): 808 (0.4), 370 (3.05), 266 (3.89). Significant infrared bands (cm^{-1}): 1611 ($\nu_{C=N}$), 969 ($\nu_{Mo=O}$). ^{1}H NMR (200 MHz, DMSO-d$_6$): δ 7.42 (t, 2H, *m*-Py), 7.83 (t, 1H, *p*-Py), 8.61 (d, 2H, *o*-Py).

(PyH)[MoO(NCS)$_2$(Sapd)](4). Yield: 0.31 g (59%); mp 187 °C. Anal. Calcd for $C_{19}H_{21}N_4O_4S_2Mo$: C, 43.10; H, 4.00; N, 10.58; S, 12.11. Found: C, 42.95; H, 4.15; N, 10.47; S, 12.01. ΛM (Mho cm^2 mol^{-1}, DMSO): 40. UV/vis (nm. logε, dm^3 mol^{-1} cm^{-1}): 757 (1.24), 503 (2.00), 414 (2.40), 310 (3.52), 265 (3.95). Significant infrared bands (cm^{-1}): 2066 ($\nu_{N\equiv C,NCS}$), 1601 ($\nu_{C=N}$), 954 ($\nu_{Mo=O}$), 864 (ν_{C-S}). ^{1}H NMR (200 MHz, DMSO-d$_6$): δ 7.98 (t, 2H, *m*-Py), 8.49 (t, 1H, *p*-Py), 8.88 (d, 2H, *o*-Py).

Results and Discussion

The oxomolybdenum(V) complexes (PyH)[MoO(X)$_2$L] are synthesized by the reaction of (PyH)[$MoOCl_5$]/(PyH)[$MoO(NCS)_5$] with the appropriate Schiff base ligands in a methanol solution. All complexes are air stable in the solid state and have poor solubilities in common organic solvents except DMF and DMSO.

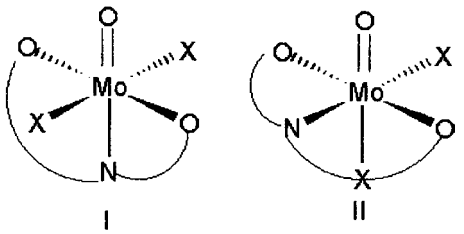
Molar conductivities for the complexes determined at a concentration of *ca.* 1×10^{-3} mol dm^{-3} in DMSO fall in the range of 31 to 42 mho cm^2 mol^{-1}, indicating that the complexes behave as 1 : 1 electrolytes in DMSO.[7]

The complexes are formulated on the basis of elemental analysis and spectral data of IR and NMR. Infrared spectra for the complexes showed a single intense absorption peak in the range of 942-971 cm^{-1}. These absorptions are assigned to the molybdenum-terminal oxygen vibration band ($\nu_{Mo=Ot}$), by comparision with the data previously reported.[8] IR spectra of the complexes (PyH)[$MoOCl_2L$] (L= Sabp and Sapd) show $\nu_{Mo=Ot}$ at *ca.* 970 cm^{-1}, which is greater than that of (PyH)[$MoO(NCS)_2L$] at *ca.* 948 cm^{-1}. This strong Mo=O$_t$ bond in the chloro complexes may be responsible for the weaker *trans* influence than is the NCS ligand. The ν_{OH} band at *ca.* 3430 cm^{-1} of the free ligands disappeared on complexation, except complexes (3) and (4), in which the observed ν_{OH} is attributed to the aliphatic OH. The intense bands at *ca.* 1625 cm^{-1} associated with the C=N stretching frequency of the free ligands are shifted to *ca.* 1600 cm^{-1} in the corresponding complexes. These data indicate that the charged phenolic oxygen atom and the azomethine nitrogen atom of Schiff base ligands are coordinated to the metal ion. These results are in accordance with the structural assignment for the complex of the N-salicylideneimines reported previously.[9] That is, when the Schiff base is coordinated as an anionic ligand through both oxygen and nitrogen atoms, the $\nu_{C=N}$ vibration shifts to a lower frequency by *ca.* 20 cm^{-1}. Meanwhile, the coordination through only the nitrogen atom as a neutral species shifts this band to a higher frequency than that of a free ligand. In regard to the NCS ligands, their characteristic absorption frequencies clearly indicate coordination through the nitrogen atom. The strong band resulting from the C≡N stretching mode is observed in the range 2066 to 2030 cm^{-1}, much lower than the value (*ca.* 2100 cm^{-1}) observed for the SCN ligand in the thiocyanato complex.[10] This frequency lowering is characteristic for the N-bonded NCS ligand. Also, the C-S stretching vibration for the complexes (2) and (4) are observed at relatively high frequencies, 847 and 864 cm^{-1}, respectively-a typical feature for isothiocyanates.[11]

Each of the Schiff base ligands possesses one or two phenolic groups and an azomethine group. The ^{1}H NMR spectrum of the ligand $SabpH_2$ in DMSO-d$_6$ showed the aromatic protons as multiplet in the range of 6.75-7.65 ppm and OH protons of the two phenolic groups at 9.50 and 13.89 ppm. For the $SapdH_2$ ligand, phenolic and 1,3-propanediol OH protons showed resonance peaks at 14.46 and 4.80 ppm, respectively. The azomethine protons for $SabpH_2$ and $SapdH_2$ each appeared as a sharp singlet at 8.99 and 8.55 ppm. For the complex (2), the OH proton resonances are not observed and the resonances for the azomethine proton of the free ligand $SabpH_2$ are shifted to the downfield of 9.31 ppm on complexation. These results suggest that the ligands coordinate to the molybdenum atom through charged phenolic oxygen atoms and a nitrogen atom of an azomethine group from the IR data. However, in all of the complexes the resonances for the protons of

aromatic and other groups showed very weak peak intensities or NMR silence when compared with PyH^+. This is ascribed to the paramagnetic complex due to the $Mo^V(d^1)$ state. The aromatic protons of the pyridinium ion as counter ions are easily distinguished from those of Schiff base ligands by comparison of the spectra for the corresponding free ligands and the complexes containing Me_4N^+, Et_4N^+ instead of the pyridinium ion.[8a] The ortho-, meta- and para-proton resonances appeared in the range 8.61-8.90, 7.42-8.02, and 7.84-8.53 ppm, respectively, with a relative intensity ratio of 2 : 2 : 1.

On the basis of the above results, the possible geometries of the complex synthesized are as follows. The dianionic tridentate Schiff base ligand can be spanned by two modes-



X=Cl, NCS

fac and *mer*- but isomeric facial spanning is sterically precluded by the ligand planarity.[12] For the *mer*-spanning, the two configurations are also possible. As shown in structure I, if the two Cl/NCS occupy the *cis*-position to the terminal oxygen, the $\nu_{Mo=Ot}$ band would appear at lower frequencies than each of the bands 970 and 950 cm^{-1} for the complexes $[MoOCl_5]^2$ and $[MoO(NCS)_5]^{2-}$ containing *trans*-Cl or -NCS to $Mo=O_t$.[11,13] The complexes prepared in the present work showed $\nu_{Mo=Ot}$ stretching at *ca.* 970 and 948 cm^{-1}. This result indicates that Cl or NCS occupies the *trans*-position to $Mo=O_t$. Thus the coordination environment around the molybdenum ion can be described as 6-coordinate octahedral configuration (structure II), the equatorial positions being occupied by the ONO-donor atoms of the tridentate Schiff base ligand and one of two Cl (or NCS), and the apicial positions by the terminal oxygen and remaining one of Cl (or NCS).

The electronic spectral data for the complexes are assigned on the basis of the energy levels reported by Sabat[5] for the tetragonal oxo-complex of d^1 molybdenum(V) ion (C_{4v}). One low energy band in the range 757-825 nm (logε= 0.4-1.98) is assigned to the first crystal field transition $^2B_2 \rightarrow {}^2E$ of the three spin-allowed *d-d* transitions ($^2B_2 \rightarrow {}^2E$, $^2B_2 \rightarrow {}^2B_1$, and $^2B_2 \rightarrow {}^2A_1$). The appearance of this low-energy band is a characteristic of mononuclear oxomolybdenum(V) complexes and essentially independent on a ligand.[1a,5] Other transitions are obscured by the intense charge transfer band. 1a,14 On the other hand, the bands observed in the range 265-503 nm (logε=~4.00) are probably due to L→M or intraligand transitions.[15]

The electrochemical behaviors of the complexes in DMSO/0.1 M TEAP have been studied by cyclic voltammetry at a platinum working electrode, and the data in the potential range 0.0 to −2.0 V (*vs.* Ag/Ag^+) are summarized in Table 1.

The cyclic voltammograms for the complexes (1) and (2) are shown in Figure 1. All complexes displayed two

Table 1. Cyclic Voltammetric Results[a] for the Complexes in 0.1 M TEAP-DMSO at 25 °C

Complex	E_{pc}[b], V (*vs.* SCE)	
1	-0.94(-0.65)	-1.75(-1.45)
2	-0.92	-1.48(-1.36)
3	-1.02(-0.64)	-1.75(-1.70)
4	-0.96(-0.79)	-1.77(-1.50)

[a] Solute concentration ~10^{-3} M; working electrode platinum; reference electrode Ag/Ag^+; counter electrode platinum. [b] Values in parentheses are coupled oxidation peaks observed with a complete CV cycle.

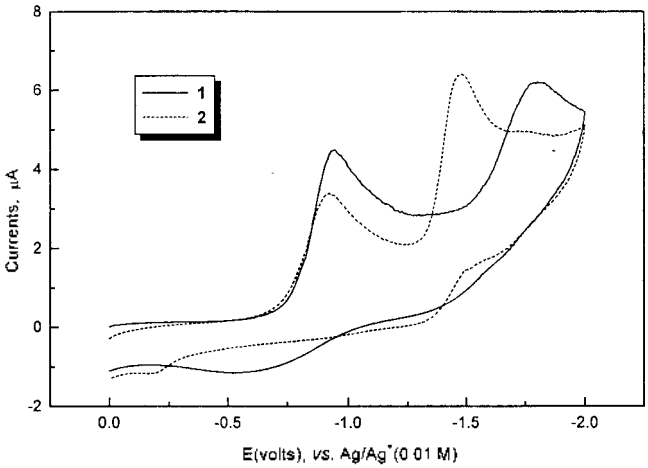


Figure 1. Cyclic voltammograms for ~1×10^{-3} M (PyH)[$MoOCl_2$(Sabp)](1) and (PyH)[$MoO(NCS)_2$(Sabp)](2) in 0.1 M TEAP-DMSO (scan rate 100 mVs^{-1}).

successive cathodic responses in the range −0.92~−1.02 and −1.48~−1.77 V, respectively, possessing almost equal current heights (Figure 1). The reduction waves are found to be coupled to weak and broad anodic peaks in the range −0.64~−0.79 and −1.36~−1.70 V, respectively. A comparison of the current heights at E_{pc}'s with those of the authentic one-electron reductions observed in the cases of other monomeric oxomolybdenum(V) complexes[1a,8] suggests that each of the reductions involves a metal-centered one-electron process. Interestingly, the reduction potentials (E_{pc}) and the $\nu_{(Mo=Ot)}$ vibration showed a correlation with the monodentate ligand of *trans*-position to $Mo=O_t$. For the complexes (1) and (2) or (3) and (4), the $\nu_{(Mo=Ot)}$ vibration in the solid state increased in the order, NCS < Cl. The values of E_{pc} for the above complexes also shifted in a more cathodic direction in the same order. This demonstrates the sensitivity of the molybdenum electron density on the *trans*-position ligand to $Mo=O_t$. Also, the E_{pc} for the complexes containing the Sapd ligand showed a more negative value than that in Sabp complexes, comparing each of the complexes (1)-(3) and (2)-(4).

References

1. (a) Purohit, S.; Koley, A. P.; Prasad, L. S.; Manoharan, P. T.; Ghosh, S. *Inorg. Chem.* **1989**, *28*, 3735. (b) Mondal, J. U.; Schultz, F. A.; Brennan, T. D.; Scheidt, W. R. *Inorg. Chem.* **1988**, *27*, 3950. (c) Rajan, O. A.;

Chakravorty, A. *Inorg. Chem.* **1981**, *20*, 660. (d) Bhattacharjee, S.; Bhattacharyya, R. *J. Chem. Soc., Dalton Trans.* **1992**, 1357. (e) Topich, J.; Lyon, J. T. *Inorg. Chem.* **1984**, *23*, 3202.
2. (a) Bray, R. C. In *Molybdenum Chemistry of Biological Significance*; Plenum: New York, 1980; p 117. (b) Bray, R. C. *Enzymes* **1975**, *12*, 299.
3. Cramer, S. P.; Wahl, R.; Rajagoplan, K. V. *J. Am. Chem. Soc.* **1981**, *103*, 7721.
4. (a) Bhattacharjee, S.; Bhattachryya, R. *J. Chem. Soc., Dalton, Trans.* **1993**, 1151. (b) Berg, J. M.; Holm, R. H. *Inorg. Chem.* **1983**, *22*, 1768.
5. Sabat, H.; Rudolf, M. F.; Jezowska-Trzebltowska, B. *Inorg. Chim. Acta* **1973**, *7*, 365.
6. Kessissoglou, D. P.; Kirk, M. L.; Lah, M. S.; Li, X.; Raptopoulou, C.; Hatfield, W. E.; Pecoraro, V. L. *Inorg. Chem.* **1992**, *31*, 5424.
7. (a) Geary, W. *J. Coord. Chem. Rev.* **1971**, *7*, 81. (b) Greenwood, N. N.; Straughan, B. P.; Wilson, A. E. *J. Chem. Soc. A.* **1968**, 2209.
8. (a) Kim, J. S.; Koo, B. K. *Bull. Korean Chem. Soc.* **1992**, *13*, 507. (b) Kim, H. J.; Koo, B. K. *ibid.* **1994**, *15*, 766.
9. (a) Hill, W. E.; Atabay, N.; McAuliffe, C. A.; McCullough, F. P.; Razzolci, S. M. *Inorg. Chim. Acta* **1979**, *35*, 35. (b) Chandra, S.; Sharma, K. K. *Transition Met. Chem.* **1984**, *9*, 1.
10. Nakamoto, K. *Infrared Spectra of Inorganic and Coordination Compounds*, 2nd ed., John Wiley and Sons: New York, 1970, p 187.
11. Sabatini, A.; Bertini, I. *Inorg. Chem.* **1965**, *4*, 959.
12. Marchi, A.; Duatti, A.; Rossi, R.; Magon, L.; Pasqualini, R.; Bertolasi, V.; Ferretti, V.; Gilli, G. *J. Chem. Soc., Dalton Trans.* **1988**, 1743.
13. Lippard, S. J. *Progress in Inorganic Chemistry*, 1st ed., John Wiley and Sons: New York, U.S.A., 1977, Vol. 22, p 57.
14. (a) Allen, E. A.; Brisdon, B. J.; Edwards, D. A.; Fowles, G. W. A.; Wiliams, R. G. *J. Chem. Soc.* **1963**, 4649. (b) Lippard, S. J. *Progress in Inorganic Chemistry*, 1st ed., John Wiley and Sons: New York, U. S.A., 1977, Vol. 22, p 59.
15. Chaudhury, M. *J. Chem. Soc., Dalton Trans.* **1984**, 115.

Pyrolysis of 1,2-Diethyltetrachlorodisilane

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Silylenes are cleanly generated from α-elimination of simple disilanes through shift of a hydrogen, halogen, or alkoxy group attached to silicon.[1] Competition between hydrogen and chlorine shifts in the pyrolysis of 1,2-dichloro-1,2-dimethyldisilane had been examined.[1k] The results from this pyrolysis showed hydrogen shift was considerably more favorable than chlorine shift. From thermolysis of 1,2-dimethoxytetramethyldisilane, a silane and a silylene was formed *via* shift of the methoxy group but not the methyl group.[1b] A few results of alkyl group shift in the pyrolyses of disilanes have been reported.[1g,2,3] Davidson and coworkers reported that a methyl group shift in the pyrolysis of hexamethyldisilane requires a higher activation energy.[2] Evidences have been found for hydrogen and methyl shift in the conversion of disilirane ($Me_2\underline{SiSiHMeC}H_2$) to β-silylsilylenes.[1g,3] It was reported that a methyl shift in the disilirane ring opening had a *ca.* 20 kcal/mol higher barrier than a hydrogen shift.[4]

We recently reported that vacuum pyrolysis of 1,2-diethyltetraalkoxydisilane in the presence of diene trapping agent was found to proceed through migration of alkoxy group and no migration of ethyl group attached to silicon.[5] Here, we wish to report silylene generation *via* alkyl group shift in the pyrolysis of 1,2-dialkyltetrachlorodisilane (alkyl= ethyl, methyl). It will be discussed the characteristics of chemical behavior of alkylchlorosilylenes produced in this report, comparing with that of previously reported ethylmethoxysilylene. In an attempt to elucidate the energetics of thermal decomposition of 1,2-diethyltetrachlorodisilane into ethylchlorosilylene, chlorosilylene, and dichlorosilylene, the temperature dependence of the product distribution was examined and the AM 1 semi-empirical calculations[6] were performed to estimate the heats of formation for the molecules containing silicon.

Experimental

General. All pyrolyses were carried out in a seasoned hot zone consisting of a 10 mm i.d. $\times$30 cm quartz tube wrapped with nichrome ribbon and covered with asbestos tape. This hot zone was seasoned with hexamethyldisilazane before use. Both residence and pressure were controlled by a 0.8 mm aperture placed at the end of vertical quartz tube which was connected to a vacuum line. The progress of pyrolysis was followed by gas chromatography with a flame ionization detector (FID) using a Hewlett-Packard 5890 instrument on a HP-1 capillary column (cross-linked 5% methylphenylsilicone, 25 m). Products yields were determined by gas chromatography with cyclohexane as an internal standard on the basis of the quantity of 1,2-dialkyltetrachlorodisilane decomposed. Seperation of the products was performed on