polysilane high polymers. Ziegler et al.44 have reported that poly(phenylmethylsilane) is a good photoconductor in which only holes are mobile. The electronic transition which creates the carriers was reported to be a $\pi^* - \sigma^*$ transition from the excited singlet state of the aromatic to the silicon backbone σ -bonds. The hole was reported to move by a hopping mechanism between phenyl groups. A speculative explanation for the formation of the holes based on the current work would involve excitation of the π^* singlet state of the phenyl group followed by ET from the Si-Si bond framework to the lowest energy singly occupied molecular orbital (SOMO) of the excited phenyl group. Recent work⁴⁵ provides evidence for hole migration in the silicon framework. Not only would this mechanism be in keeping with the direction of CT observed in the quenching of the excited singlet states of aromatics by hexamethyldisilane, but it would also be analogous to the mechanism for generation of semiconductor

(44) Kepler, R. G.; Ziegler, J.; Harrah, L. A.; Kurtz, S. R. Bull. Am. Phys. Soc. **1983**, 28, 362.

properties in polysilanes by doping with strong electron acceptors. In contrast, σ - σ * states are most likely involved in the photoconduction mechanism in alkyl-substituted polysilanes.⁴⁵

Conclusion

We have shown that the quenching of the excited singlet states of electron-deficient benzenes by hexamethyldisilane proceeds by an ET or CT process and can proceed at essentially the diffusion limit in cases where the exothermicity of the ET step is large and negative. This ET reaction is one member of a unique class in which the ground-state donor is an electron-rich, σ -bonded species. An investigation of the photochemical reactions of polysilanes currently extant in the literature suggests that these σ to π^* ET processes may be widespread.

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Registry No. Polysilane, 32028-95-8; hexamethyldisilane, 1450-14-2; benzene, 71-43-2; α , α , α -trifluorotoluene, 98-08-8; benzonitrile, 100-47-0; hexafluorobenzene, 392-56-3; 1,4-dicyanobenzene, 623-26-7.

Resonance-Enhanced Multiphoton Ionization Spectra of the SiCI Radical between 430 and 520 nm

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The resonance-enhanced multiphoton ionization spectra of the SiCl radical observed between 430 and 520 nm are reported. The spectral structure arises from two-photon transitions to the C ${}^{2}\Pi_{r}$, D ${}^{2}\Sigma^{+}$, and E states. Absorption of a third laser photon formed the cation. No subsequent fragmentation of cations was observed. The spectra terminating on the C ${}^{2}\Pi_{r}(v'=3-7)$ levels and the Si³⁷Cl spectrum of the C ${}^{2}\Pi_{r}$ state are reported for the first time. The Si³⁵Cl C ${}^{2}\Pi_{r}$ state spectroscopic constants of $\omega_{e} = 682.7 \pm 3.8 \text{ cm}^{-1}$ and $\omega_{e}x_{e} = 3.8 \pm 0.5 \text{ cm}^{-1}$ are reported.

I. Introduction

This paper demonstrates the first detection of SiCl radical by resonance-enhanced multiphoton ionization (REMPI) spectroscopy. Throughout the spectral region of this study very strong SiCl⁺ ion signals were observed which suggests that REMPI spectroscopy can be used to sensitively detect relatively small concentrations of SiCl radicals. SiCl appears as one of the intermediates in the plasma reduction of SiCl₄ to produce semiconductor Si crystals.¹ A sensitive method for detecting SiCl radicals, which REMPI provides, may further the understanding of such Si growth mechanisms.

Resonance enhancement originates from two-photon preparation of the C ${}^{2}\Pi_{r}$, D ${}^{2}\Sigma^{+}$, and E states. Part of the present work confirms bands previously observed with the traditional emission and absorption spectroscopies, but the REMPI spectrum revealed many more, previously unreported vibrational bands. The sensitivity and mass selectivity of REMPI also enabled the first measurement of the band positions of the Si³⁷Cl isotopic radical. This isotopic information ascertained the vibrational numbering assignments of the REMPI bands. On the basis of this larger assigned data set, we report spectroscopic constants, ω_{e} and $\omega_{e}x_{e}$, for the C ${}^{2}\Pi_{r}$ state.

II. Experimental Methods

The apparatus is similar to one previously described.² Briefly, it consists of a flow reactor in which Cl atoms reacted with silane

(SiH₄) to produce SiCl radicals. A portion of the flow reactor effluent leaked through a skimmer and into the ion optics of a time-of-flight mass spectrometer. The SiCl radicals were ionized by the focused light from an excimer laser pumped tunable dye laser. The ions were extracted into the time-of-flight mass spectrometer, and the mass-resolved m/z 63 or 65 (corresponding to Si³⁵Cl or Si³⁷Cl) ion currents were recorded. The vacuum chamber which enclosed the mass spectrometer was operated at a pressure of ~10⁻⁵ Torr.

Chlorine atoms were produced by passing ~2 Torr of a 10% $Cl_2/90\%$ He mixture through a microwave discharge. Downstream from the microwave discharge near the sampling skimmer, an injector tube introduced ~10 mTorr of a 15% SiH₄/85% He mixture. Maximum signal was obtained when the injector was very close (~3 mm) to the skimmer. Under these very Cl atom rich conditions SiCl was produced. Presumably, other radical species were also generated under these reaction conditions and the SiCl radical may not have been the predominant product. As expected of such a multistep reaction, the magnitude of the SiCl signal was very sensitive to the amount of SiH₄ introduced. On the basis of our calculations, which assume 100% conversion of SiH₄ into SiCl, we can detect at least 10⁹ radicals/cm³ in the ionization region of the mass spectrometer with a single laser shot.

The tunable dye laser generated 5-20-mJ pulses which were focused into the ionization region by a 250-mm lens. The spectra presented are *not* corrected for variations in laser power; however,

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⁽²⁾ Dulcey, C. S.; Hudgens, J. W. J. Phys. Chem. 1983, 87, 2296.

the figures are composite spectra constructed from overlapping regions of several laser dyes chosen over regions in which the laser output changed slowly. The laser dyes used were coumarin 440 (430-450 nm), coumarin 460 (450-470 nm), coumarin 480 (470-490 nm), coumarin 500 (490-520 nm), and coumarin 540A (520-550 nm). In conformity with custom laser wavelengths are listed in reference to air medium.

III. Results and Discussion

REMPI spectra of SiCl carried by m/z 63 and m/z 65 were observed over the laser wavelength range of 430–520 nm (Figures 1 and 2). All the major peaks are assigned, though for clarity not all of the bands are indexed in the figures. Congruous with the relative ${}^{35}Cl/{}^{37}Cl$ isotopic abundance, the intensity of the m/z63 signal (Si ${}^{35}Cl$) was 3 times greater than the m/z 65 signal (Si ${}^{37}Cl$). As observed in most mass spectra of resonantly ionized free radicals,³ the laser-produced SiCl⁺ ions showed no evidence of fragmentation.

In addition to the ion signals that originated from SiCl radicals, m/z 28 ion signals corresponding to transitions to known states of atomic silicon were also observed.

1. $C {}^{2}\Pi \leftrightarrow X {}^{2}\Pi_{r}$ Bands. a. Doublet Splitting. Long progressions are observed in the region of 430-520 nm (Figures 1 and 2). The band wavelengths, energies, and assignments are listed in Table I. Between 445 and 490 nm (Figure 1) one progression of five doublets shows peak intensity ratios of ~2:1. These doublet intensity ratios arise from the difference in populations between the X ${}^{2}\Pi_{3/2}$ and X ${}^{2}\Pi_{1/2}$ spin-orbit states. The more intense members originate from the lower energy X ${}^{2}\Pi_{1/2}$ state.

Between 40 000 and 43 000 cm⁻¹ Jevons⁴ has reported the UV emission spectrum of the C ${}^{2}\Pi_{r}(v'=0,1,2) \rightarrow X {}^{2}\Pi_{r}$ bands. In a two-photon spectrum these bands will lie between 465 and 490 nm. In the REMPI spectrum the average two-photon laser frequency separation between each pair of doublets is ~200 cm⁻¹. This separation is the same as is expected of a two-photon transition from the ground state (A'' = 206.6 cm⁻¹) to the C ${}^{2}\Pi_{r}$ state (A' = 10.9 cm⁻¹),⁵ i.e., A''-A' = 195.7 cm⁻¹. The doublet intervals measured from the positions of the O-branch peaks of each pair of vibrational bands are listed in Table II.

b. Vibrational Spacing. Jevons⁴ observed $(v' = 0, 1, 2) \rightarrow (v'' = 0)$ vibrational bands which exhibited an interval of ~665 cm⁻¹. Our present two-photon resonant REMPI spectrum shows these same bands as well as members up to v' = 6 from v'' = 0 and v' = 7 from v'' = 1. These higher vibrational bands overlap with bands from the D ${}^{2}\Sigma^{+} \longleftarrow {}^{2}\Pi_{r}$ system. The fact that these new bands belong to the C-state was determined by the vibrational spacing and the isotope shifts. This paper is the first to report the C ${}^{2}\Pi_{r}(v'=3-7)$ vibrational bands.

The Deslandres tables (Tables III and IV) list the band maxima and their differences. Table III lists bands originating from X ${}^{2}\Pi_{1/2}$, and Table IV lists bands originating from X ${}^{2}\Pi_{3/2}$. A least-squares fit of the 33 upper state vibrational spacing differences (using v' = 0 through v' = 7) listed in Tables III and IV yielded C ${}^{2}\Pi_{r}$ state spectroscopic constants of $\omega_{e} = 682.7 \pm$ 3.8 cm⁻¹ and $\omega_{e}x_{e} = 3.8 \pm 0.5$ cm⁻¹. These can be compared with the values reported by Jevons⁴ of $\omega_{e} = 674.2$ cm⁻¹ and $\omega_{e}x_{e} = 2.2$ cm⁻¹, who used v' = 0 through v' = 2.

c. Isotope Shifts. The REMPI spectrum of the Si³⁷Cl radical was measured by recording the m/z 65 signal. Table II lists the observed ³⁷Cl-³⁵Cl isotope shifts of the C ²II_r $\leftrightarrow X$ ²II_r vibrational bands. Calculated shifts are also listed which are based on ground-state Si³⁵Cl vibrational constants⁵ of $\omega_e = 535.586$ cm⁻¹, $\omega_e x_e = 2.1748$ cm⁻¹, $\omega_e y_e = 0.00598$ cm⁻¹, and our C-state vibrational constants. For transitions involving v' = 0-4 very precise isotopic shifts were measured from positions of the sharp O-

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TABLE I: Positions and Assignments of Vibrational B	ands Observed
in the REMPI Spectrum of the X ² II, Si ³⁵ Cl Radical be	etween 430 and
520 nm	

λ _{laser} , nm (air)	two-photon energy, cm ⁻¹ (vac)	upper state	Ω‴	vib band $(v' \leftarrow v')$
517.48 515.36 513.40 510.62	38 638 38 797 38 946 39 157	С С С С С	1/2 1/2 3/2 1/2	0-5 1-6 0-4 0-4
508.65 506.25 503.87 501.96 500.05	39 309 39 495 39 682 39 833 39 985	С С С С С	1/2 3/2 1/2 1/2 3/2	1-5 0-3 0-3 1-4 • 0-2
497.38 495.31 493.67 491.06	40 199 40 367 40 501 40 717	C C C C	1/2 1/2 3/2 1/2	0-2 1-3 0-1 0-1
489.07 487.13 485.44 484.74 482.96 481.49	40 883 41 045 41 189 41 248 41 400 41 526	С С С С С С С С С	1/2 3/2 3/2 1/2 1/2 1/2	1-2 0-0 1-1 0-0 1-1 2-2
479.26 477.78 476.96 475.26 473.98 471.74 470.32	41 720 41 849 41 921 42 070 42 184 42 385 42 513	с с с с с с с с с с с с с с	3/2 3/2 1/2 1/2 1/2 1/2 3/2 3/2	1-0 2-1 1-0 2-1 3-2 2-0 3-1
469.54 468.13 466.73 464.54 463.18 462.37 460.94	42 583 42 711 42 840 43 041 43 168 43 244 43 377	С С С С С С С С С С С С С С С С С С С	1/2 1/2 3/2 3/2 1/2 1/2	2-0 3-1 4-2 3-0 4-1 3-0 4-1
459.80 457.62 456.40 455.42 454.39 453.16 450.82	43 485 43 693 43 809 43 903 44 003 44 122 44 351	с с с с с с с с с с с с с с	1/2 3/2 3/2 1/2 1/2 1/2 3/2	5-2 4-0 5-1 4-0 5-1 6-2 5-0
449.59 448.85 447.75 446.33 444.25 443.03 442.58 441.71 440.63	44 472 44 546 44 655 44 797 45 007 45 131 45 177 45 266 45 377	C C C D D C C E	3/2 1/2 1/2 3/2 1/2 1/2 1/2 1/2 1/2	$ \begin{array}{c} 6-1 \\ 5-0 \\ 6-1 \\ 0-0 \\ 1-1 \\ 6-0 \\ 7-1 \\ 0-0 \end{array} $
439.98 438.53 437.96 436.72 434.69 433.74 431.75	45 444 45 594 45 654 45 783 45 997 46 098 46 310	D D D E D D	3/2 3/2 1/2 1/2 1/2 3/2 1/2	0-0 2-1 1-0 2-1 1-0 2-0 2-0

branches. Spectral congestion thwarted O-branch measurements of the REMPI bands involving transitions to v' > 4 or v'' > 0. For these bands the isotopic shifts were measured from band maxima. The agreement between measured shifts and the calculated shifts is good. This agreement confirms the vibrational numbering of the bands and also confirms the specific C- and D-state band assignments for the spectral region in which the two states overlap.

All evidence (vibrational intervals, electronic origin, and doublet splitting) supports the assignment of the majority of the REMPI spectrum to two-photon C ${}^{2}\Pi_{r} \leftarrow X {}^{2}\Pi_{r}$ transitions.

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⁽⁴⁾ Jevons, W. Proc. Phys. Soc., London 1936, 48, 563.

TABLE II: Positions and Assignments of Band Maxim	Observed in REMPI Spectra of the C ² II	I, ← X ² Π,	, Bands of Si ³⁵ Cl and Si ³⁷ Cl Radicals
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				isotope s	hift, cm ⁻¹		
vib band	Ω''	chlorine isotope	O-branch max, cm ⁻¹	obsd	calcd	vib spacing, cm ⁻¹	A'' - A', cm ⁻¹
			Based upon O-Bran	ch Maxima ^a			
0-0	1/2	35	41 227.86				
	1/2	37	41 229.65	-1.8	0.9		
	3/2	35	41 023.30				204.56
	3/2	37	41 029.79	-6.5			199.86
1-0	1/2	35	41 896.57			668.71	
	1/2	37	41 887.95	8.6	8.9		
	3/2	35	41 698.75				197.82
	3/2	37	41 689.55	9.2			198.40
2-0	1/2	35	42 562.28			665.71	
	1/2	37	42 546.44	15.8	16.8		
	3/2	35	42 363.94				198.34
	3/2	37	42 347.43	16.5			199.01
3-0	1/2	35	43 218.95			656.67	
	1/2	37	43 195.19	23.8	24.5		
	3/2	35	43 021.43				197.52
	3/2	37	43 005.28	16.2			189.91
4-0	1/2	35	43 876.02			657.07	
	1/2	37			32.1		
	3/2	35	43 681.24				194.78
	3/2	37	43 649.70	31.5			
			Based upon Band	Maxima ^b			
5-0	1/2	35	44 546				195
	1/2	37	44 506	40	39		202
	3/2	35	44 351				
	3/2	37	44 304	47			
6-0	1/2	35	45 177			631	
	1/2	37	(45 1 3 2) ^c	45	47		

^a Measurement precision is 0.08 cm⁻¹. ^b Measurement precision is 1 cm⁻¹. ^c Blended.



Figure 1. The composite spectrum of the Si³⁵Cl radical from 430 to 490 nm showing the two-photon transitions from the X ${}^{2}\Pi_{r}$ ground state to the C ${}^{2}\Pi_{r}$, D ${}^{2}\Sigma^{+}$, and E states. The many evident hot bands are not indexed.



Figure 2. The composite spectrum of the Si³⁵Cl radical (m/z 63) from 480 to 520 nm. Only the hot bands terminating at the upper state v' = 0 level are indexed.

d. Ionization Potential and REMPI Mechanism. To determine the overall REMPI mechanism that produces spectrum, the ionization potential of the SiCl radical must be known. The SiCl ionization potential is not known but can be estimated. The related radical, SiF, possesses an ionization potential of 7.28 eV.⁶ For the similar species, AIF (IP = 9.73 eV) and AlCl (IP = 9.4 eV), the ionization potential difference is ~0.3 eV.⁶ Thus, we estimate that the ionization potential of SiCl lies near 7.0–7.2 eV.

The C ${}^{2}\Pi_{r}$ state possesses an outer-shell electron configuration⁵ of the type

$$(z\sigma)^{2}(y\sigma)^{2}(w\pi)^{4}(x\sigma)^{2}...(4p\pi)$$

From this configuration the lowest energy ionization process will

⁽⁶⁾ Lias, S. G.; Bartmess, J. E.; Holmes, J. L.; Levin, R. D.; Liebman, J. F. J. Phys. Chem. Ref. Data, in press.

TABLE III: The Deslandres Table of the Si³⁵Cl Radical C ${}^{2}\Pi_{r} \leftarrow X {}^{2}\Pi_{1/2}$ Bands^a

v'	v'' = 0		v'' = 1		v'' = 2		v'' = 3		v'' = 4		v'' = 5		<i>v''</i> = 6	
0	41 248 673	531	40 717 682	518	40 199 683	517	39 682 685	525	39157 676	520	38 638 671			
1	41 921 662	521	41 400 671	517	40 883 643	516	40 367	534	39 833	524	39 309	512	38 797	
2	42 583 661	512	42 070 641	544	41 526 658									
3	43 244 659	532	42 71 1 666	527	42 184 655									
4	43 903 643	526	43 377 625	537	42 840 645									
5	44 546 631	543	44 003 652	518	43 485 638									
6	45 177	523	44 655 611	533	44 1 2 2									
7			45 265											

^a Band maximum positions and vibrational intervals between pairs of vibrational levels are listed in cm^{-1} (vac).

TABLE IV: The Deslandres Table of the Si³⁵Cl Radical C $^2\Pi_r$ $\overleftarrow{}$ X $^2\Pi_{3/2}$ Bands^a

v'	$v^{\prime\prime} = 0$		v'' = 1		$v^{\prime\prime} = 2$		v'' = 3		v'' = 4
0	41 0 45	544	40 501	516	39 985	490	39 495	550	38 946
	675		687						
1	41 720	531	41 189						
	665		660						
2	42 385	536	41 849						
	657		664						
3	43 041	529	42 512						
	651		655						
4	43 692	525	43 168						
	658		642						
5	44 3 5 1	542	43 809						
			663						
6			44 472						

^{*a*} Band maximum positions and vibrational intervals between pairs of vibrational levels are listed in cm^{-1} (vac).

form the ground-state ion. For an SiCl ionization potential of 7.2 eV, laser ionization is possible from the two-photon-prepared C ${}^{2}\Pi_{r}$ state by absorption of one photon more energetic than 517 nm, i.e., a 2 + 1 REMPI mechanism.

e. Hot Bands. Hot bands that display a spacing of ~ 535 cm⁻¹ are observed throughout the spectrum. This interval corresponds to the known ground-state vibrational spacing. Sequences are built on most of the C-state bands, e.g., just to the blue of the 4–0 band lie the 5–1 and 6–2 bands. From 485 to 520 nm hot bands from the ground-state vibrational levels of up to v'' = 7 are seen (see Table I). The presence of v'' = 7 hot bands indicates that SiCl radicals are produced with at least 20 kcal/mol of vibrational energy.

f. Spectrum from 485 to 550 nm. In the present REMPI spectrum the SiCl signal becomes progressively weaker from the origin at 485 nm out to 520 nm (Figure 2). No REMPI bands were observed between 520 and 550 nm. Two reasons can account for the decrease in signal intensity and absence of signal to the red of 517 nm: (1) the vibrational levels observed may reflect a SiCl formation process which only populates levels up to v'' = 6 or 7, and (2) the intensity falloff is symptomatic of a change in the REMPI mechanism. For an IP = 7.2 eV, four photons (instead of three) are required to ionize SiCl when the laser is tuned to the red of 517 nm.

2. $D^{2}\Sigma^{+} \longleftarrow {}^{2}\Pi_{r}$ Bands. Between 431 and 447 nm the REMPI spectrum (Figure 1) shows a progression of two-photon $D^{2}\Sigma^{+}(v'=0,1,2) \longleftrightarrow X^{2}\Pi_{r}(v''=0)$ bands which agree with those previously reported.^{4,7} The positions of these lines are listed in Table I. The vibrational interval in the $D^{2}\Sigma^{+}$ state is ~650 cm⁻¹. Hot bands originating from $D^{2}\Sigma^{+}(v'=0,1,2) \longleftrightarrow X^{2}\Pi_{r}(v''=1)$ transitions are also seen. Bands originating from the $D^{2}\Sigma^{+} \longleftrightarrow X^{2}\Pi_{r}$ system that lie to the red of the origin at 447 nm (i.e., $v' = 0 \longleftrightarrow v'' > 0$) are obscured by the strong $C^{2}\Pi_{r} \longleftrightarrow {}^{2}\Pi_{r}$ signals.

The D-state electron configuration is⁸

$$(z\sigma)^{2}(y\sigma)^{2}(w\pi)^{4}(x\sigma)^{2}...(4p\sigma)^{4}(x\sigma)^{2}$$

From this configuration the lowest energy ionization process will form the ground-state ion. Laser ionization can proceed through a 2 + 1 REMPI mechanism.

3. E State $\leftarrow {}^{2}\Pi_{r}$ Bands. The E-state(v'=0) $\leftarrow X$ ${}^{2}\Pi_{1/2}(v''=0)$ band which was previously observed⁵ by UV absorption at 45375 cm⁻¹ appears in the REMPI spectrum as a weak two-photon band at 440.63 nm. The E-state(v'=0) $\leftarrow X$ ${}^{2}\Pi_{3/2}(v''=0)$ band is not distinguished, presumably because it is obscured by the C ${}^{2}\Pi_{1/2}(v'=6) \leftarrow X {}^{2}\Pi_{1/2}(v''=0)$ band at 442.58 nm. The band at 434.64 nm (45997 cm⁻¹) is tentatively assigned as the E-state(v'=1) $\leftarrow X {}^{2}\Pi_{1/2}(v''=0)$ band. Such an assignment gives a (v'=1)-(v'=0) vibrational interval of 620 cm⁻¹. Transitions to v' > 0 have not been previously reported.

The vibrational interval of 620 cm⁻¹ in the E-state is similar to the vibrational intervals observed in the C ${}^{2}\Pi_{r}$ and D ${}^{2}\Sigma^{+}$ states and the ~680-cm⁻¹ interval in the X ${}^{1}\Sigma^{+}$ cation.⁹ These similarities favor an assignment of the E-state as a Rydberg state which possesses a ground-state cation core. The most likely candidate assignment is to one of the 4d Rydberg states.

Previous UV absorption studies by Oldershaw and Robinson⁷ observed bands assigned to the F-state $\leftarrow X \,{}^{2}\Pi_{r}$ system at 217.475 and 216.482 nm. These bands, expected to appear at 435.0 and 433.0 nm in the two-photon resonant REMPI spectrum, were not observed.

Registry No. SiCl, 13966-57-9; Si³⁵Cl, 96498-26-9; ³⁵Cl, 13981-72-1; ³⁷Cl, 13981-73-2.

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⁽⁹⁾ Barrow, R. F.; Drummond, G.; Walker, S. Proc. Phys. Soc. London, Sect. A 1954, 67, 186.