



Studies on interactions of excited cadmium and mercury atoms with secondary and tertiary alkyl- and silylamines in gas phase

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ABSTRACT

The Cd(³P₁)- and Hg(³P₁)-photosensitized emissions of some secondary and tertiary alkyl- and silylamines have been investigated under conditions of steady illumination at 493 and 298 K, respectively. The emission bands were observed at around 440 nm in the cadmium-photosensitized reactions of these amines. In contrast, no appreciable emission bands were observed in the mercury-photosensitized reactions of these amines. However, upon addition of tert-butyl alcohol to the amine-mercury system, an emission band evolved at around 350 nm in the mercury-photosensitization. The peak-wavelengths for secondary and tertiary alkyl- and silylamines are slightly shorter than the values predicted from the correlations between the peak wavelength and the first ionization energy obtained in the cadmium- and mercury-photosensitized luminescence of ammonia and primary amines. The quenching efficiencies of the cadmium and mercury resonance lines by secondary alkyl- and silylamines are higher than those by tertiary alkyl- and silylamines. These observations suggest that the steric hindrance by the alkyl and silyl groups to the approach of the nitrogen atom in the amines to excited cadmium and mercury atoms seems to be an important factor for the stabilization of the exciplexes and the quenching of the resonance lines. The behavior of silylamines is similar to that of alkylamines in cadmium- and mercury-photosensitized reactions.

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1. Introduction

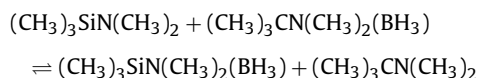
It is generally accepted that a silylamine is a much weaker base than the corresponding alkylamine. This property of silylamines is mainly related to the planar geometry of the nitrogen atom in the silylamine. However, a review of the literature of Lewis acid-base chemistry of silylamines provided a large number of scattered contributions to this problem [1].

In general, pK_a value of amines reflect electron donating ability of their nitrogen atom. However, it is not possible to measure the pK_a values for general silylamines because they readily undergo hydrolysis in aqueous solutions making evaluations of electron donor ability difficult. The pK_a values only for highly hindered silylamines are currently available [2,3]. Therefore, it is difficult to understand the significance of the pK_a values, which is related to electron donor ability, for silylamines because relevant data are not available in comparison with analogous alkylamines.

Gas-phase proton affinity data were obtained for some silylamines by measuring the equilibrium constants of the proton-transfer reactions between the silylamines and the

reference bases using pulse ion cyclotron resonance mass spectrometry [4].

Huber and Schmidbaur [5] investigated the following equilibrium reactions by ¹H NMR spectroscopy:



They obtained the equilibrium constant ($K=0.35$ in C₆D₆ and 0.50 in CDCl₃) for the equilibrium reaction; this shows that both the silylamine and the C/Si-analogous alkylamine have very similar donor properties. They also reported that small amines such as (CH₃)₃N are much more powerful donors than the silylamine (CH₃)₃SiN(CH₃)₂, whereas bulky amines are less efficient donors. Their results indicate that steric effects play an important role in the donor ability of amines. They also compared the donor ability of (CH₃)₃SiN(CH₃)₂ with those of many alkylamines, but not with those of other silylamines.

It is well known that the fluorescence of aromatic compounds shows quenching and exciplex emission in the presence of aliphatic tertiary amines in inter- and intramolecular systems [6–10]. Some silylamines are expected to exhibit these abovementioned phenomena. Preliminary experiments, however, show that

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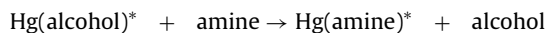
the fluorescences of naphthalene and anthracene were quenched by some silylamines, while the exciplex emissions between the excited states of naphthalene and anthracene and these silylamines were not observed.

In the cadmium- and mercury-photosensitized reactions of saturated amines, it was reported that the amines quenched the cadmium resonance line at 326.1 nm [11] and mercury resonance line at 253.7 nm [12,13] and that primary amines showed exciplex emissions in both cadmium- and mercury-photosensitized reactions. In addition, secondary and tertiary amines have been known to show exciplex emissions in the cadmium-photosensitized reactions, but not in the mercury-photosensitized reactions.

Newman et al. [14] obtained a good correlation between the peak wavelength and the first ionization energy of substrates for both the water-alcohol series and ammonia-amine series in mercury photosensitization; this result indicates that the emission bands are of the charge-transfer type. Yamamoto and Sato [15] also observed good relationships between the structure of the amines and the peak wavelength of the cadmium exciplex band, the quantum yield of exciplex emission and the relative rate constant for the exciplex formation. These findings show that the cadmium exciplexes are also of the charge-transfer type.

In these two photosensitized reactions, it was noted that the reactivity of amines, alcohols, and ethers is governed not only by the electron-donating ability of the nitrogen and oxygen atoms but also by, in part, other factors that suppress the exciplex formation in secondary and tertiary amines and in ethers [14,15].

The emission intensity of mercury-amine exciplexes was reported to increase with the addition of alcohols in the gas phase [16,17]. The increase in the emission intensity of the mercury-amine exciplex upon the addition of alcohols was explained by the following ligand-exchange reaction:



However in the liquid-phase, the formation of a mixed ligand exciplex was observed in the mercury-photosensitized luminescence of amine-alcohol mixtures [18]. In a previous study [19], we briefly investigated the cadmium-photosensitized reactions of some silylamines and observed the exciplex emission between excited cadmium atoms and silylamine molecules.

In the present investigation, we studied the $\text{Cd}(^3\text{P}_1)$ - and $\text{Hg}(^3\text{P}_1)$ -photosensitized reactions of some secondary and tertiary alkyl- and silylamines and we examined the influence of the structural properties of alkyl- and silylamines on these photosensitized reactions. We also studied the mercury-photosensitized luminescence of amine-alcohol mixtures.

2. Experimental

2.1. Materials

The cadmium used was of high-purity grade (99.9999%, Osaka Asahi Metal Co.). The silylamines and secondary and tertiary alkylamines used were *N*-trimethylsilyldimethylamine (TMSDMA), *N*-trimethylsilylpyrrolidine (TMSPY), 1,1,3,3-tetramethyldisilazane (TEMDS), 1,1,1,3,3,3-hexamethyldisilazane (HXMDS), 2,2,5,5-tetramethyl-2,5-disila-azacyclopentane (TEMDSAC), 1,1,1,2,3,3,3-heptamethyl disilazane (HPMDS), *N,N*-diethylamine (DEA), dipropylamine (DPA), piperidine (P), triethylamine (TEA), *N*-methylpyrrolidine (MPY), and *N*-methylpiperidine (MP). They were obtained from commercial sources, and they were used after drying with potassium hydroxide and a trap-to-trap distillation. The structures of the alkyl- and silylamines are shown in Scheme 1. *tert*-Butyl alcohol (*tert*-BAL) was obtained from a commercial source and was used as supplied.

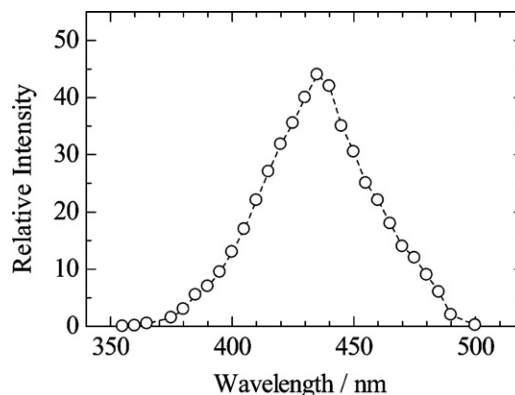


Fig. 1. Emission band obtained in the $\text{Cd}(^3\text{P}_1)$ -photosensitized reaction of TMSDMA at 493 K and at 200 Pa.

2.2. Measurements

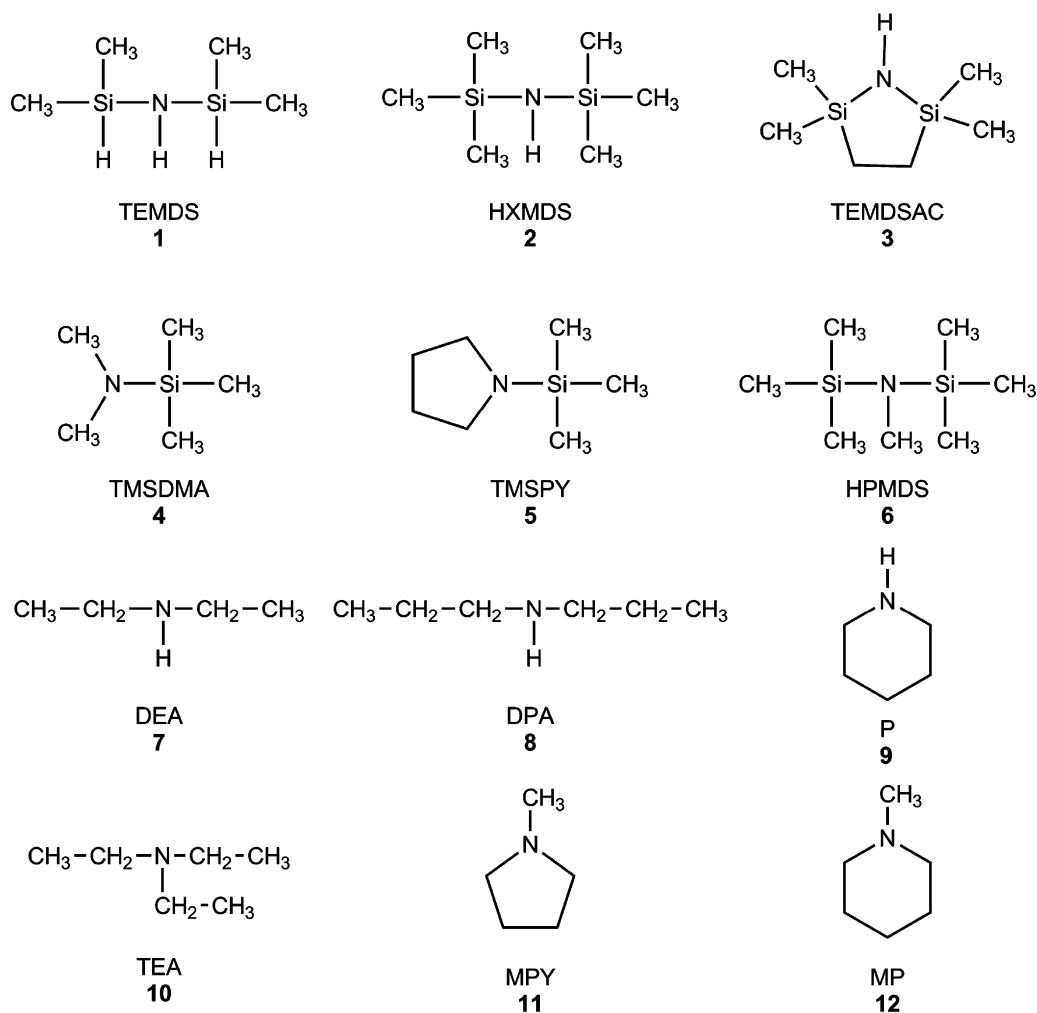
The apparatus used in the cadmium-photosensitized luminescence has been previously described [20–22]. The light source used here was a home-made spiral cadmium discharge lamp made of fused quartz. A cylindrical quartz cell (10 cm in length and 2 cm in diameter) including a piece of cadmium metal and the lamp, were kept in an electric furnace at 493 K. The 228.8-nm resonance line and the lines longer than 400 nm were filtered by a Pyrex plate and a Toshiba glass filter, UV-D33S. The 326.1-nm resonance line was used to produce $\text{Cd}(^3\text{P}_1)$ atoms from cadmium vapor. The light from the cell was detected by a Hitachi spectrophotometer, Model 139. The pressures of the gases were measured by a pressure gauge (W&T model 62-075, Nagano Keiki Co.). The low pressures of the gases were measured by using a set of calibrated expansion volumes. All the measurements of the cadmium-photosensitized luminescence were done at 493 K.

The emission spectra in the mercury-photosensitized luminescence were obtained with a Hitachi 203 fluorescence spectrophotometer equipped with a Hamamatsu R-446 photomultiplier. The reaction cell was a 10 mm quartz cuvette with four transparent planes. The cell, containing a mercury droplet, was connected to a vacuum system. A low-pressure mercury germicidal lamp (Toshiba Electric Co.) was used. The 253.7 nm resonance radiation, which was isolated with a monochromator produced $\text{Hg}(^3\text{P}_1)$ atoms from mercury vapor. Amine-alcohol mixtures of various proportions were prepared by circulating the gas mixtures of known amounts of amine and alcohols with a magnetically operated fan around a closed loop. The pressures of the gases were measured using a Baratron pressure gauge. Sample gases were introduced into the cell and permitted to stand for a few minutes before measuring the luminescence. All the experiments for the mercury-photosensitized reactions were performed at 298 K.

3. Results

Fig. 1 shows the emission band obtained for the cadmium-photosensitized reaction of TMSDMA. Similar emission bands were obtained for the other silylamines and the secondary and tertiary alkylamines used in this study. The quantum yields of the silyl- and alkylamine emissions were determined by comparing the integrated intensities of the emission bands of these amines with that of ammonia whose quantum yield is known (0.67 [15]), under the same conditions. These quantum yield values are listed in Table 1 along with the peak wavelengths of the emission band.

In order to estimate the quenching efficiency of the 326.1-nm resonance line, its intensity was measured as a function of the substrate pressure. The Stern–Volmer plots for TMSDMA,



TEMDSAC, TMSPY are shown in Fig. 2, where I_0 and I denote the intensities of the resonance line in the absence and presence of substrates, respectively. The half-quenching pressures obtained from the slopes of the straight lines in Fig. 2 are listed in Table 1. The half-quenching pressure is defined as the pressure at which the intensity of the resonance line is reduced to one-half of the intensity in the absence of quenchers.

The mercury-photosensitized reactions of silylamines and alkylamines were subsequently investigated. The quenching of the resonance line of mercury was observed; however, the

exciplex emission was not observed for these amines (a previous study reported that secondary and tertiary amines do not exhibit mercury-photosensitized luminescence [14]). The Stern-Volmer plots for the quenching of the mercury resonance line by TMSDMA, TEMDS, HPMDS, and TMSPY are shown in Fig. 3. The half-quenching pressures obtained from the slopes of the straight lines are listed in Table 2.

Previously, we studied the mercury-photosensitized luminescence of some alcohol-amine mixtures. We observed that the

Table 1

Peak wavelengths and quantum yields of the cadmium-photosensitized luminescence of amines and half-quenching pressure values for the quenching of the cadmium resonance line.

	Amines	λ_{\max} (nm)	ϕ	$P_{1/2}$ (326.1 nm) (Pa)
1	TEMDS	–	–	111
2	HXMDS	440	0.007	222
3	TEMDSAC	435	0.006	156
4	TMSDMA	435	0.057	238
5	TMSPY	445	0.039	345
6	HPMDS	440	0.039	667
7	DEA	455	0.10	17.2
8	DPA	445	0.035	20.5
9	P	440	0.030	41.8
10	TEA	444	0.12	81.3
11	MPY	435	0.29	35.1
12	MP	435	0.093	49.3

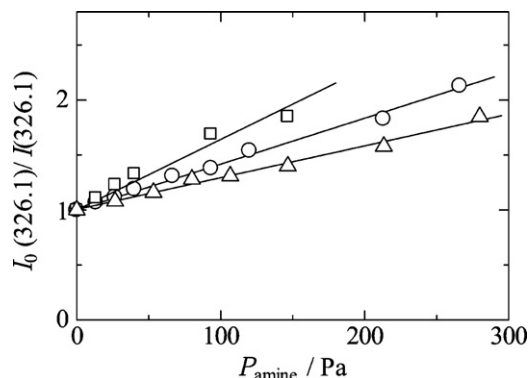


Fig. 2. Stern-Volmer plots for the quenching of the cadmium resonance line at 326.1 nm by TMSDMA (○), TEMDSAC (□), and TMSPY (△).

Table 2

Peak wavelengths and quantum yields of the mercury-photosensitized luminescence of tert-butyl alcohol–amine mixtures and half-quenching pressure values for the quenchings of the mercury resonance line and the emission from the Hg-tert-butyl alcohol exciplex.

	Amines	λ_{\max} (nm)	Φ	$P_{1/2}$ (253.7 nm) (Pa)	$P_{1/2}$ (300 nm) (Pa)
1	TEMDS HXMDS	345	0.052	38.2	20.4
2	TEMDSAC	350	0.18	53.5	23.3
3	TMSDMA	345	0.28	58.1	8.20
4	TMSPY	355	0.13	66.7	2.40
5	HPMDS	355	0.03	95.2	3.48
6	DEA	355	0.15	80.6	4.84
7	DPA	365	0.047	74.1	3.32
8	TEA	360	0.082	57.8	4.04
10	MPY	350	0.21	98.2	35.8
11	MP	350	0.14	111	23.4
12		355	0.24	83.3	40.4

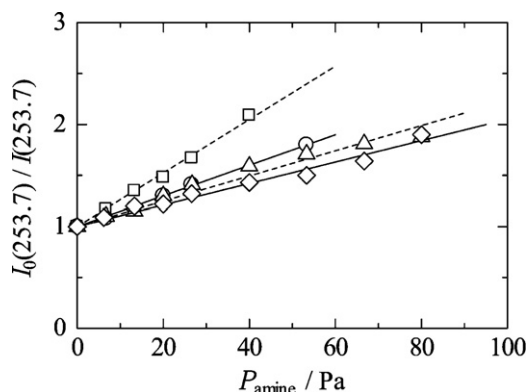
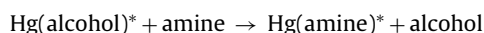


Fig. 3. Stern–Volmer plots for the quenching of the mercury resonance line at 253.7 nm by TMSDMA (○), TEMDS (□), HPMDS (△), and TMSPY (◇).

intensity of the sensitized luminescence for alcohols decreased drastically with an increase in the amine/alcohol ratio, while the emission intensity for amines increased with the alcohol pressures [17,18]. In order to explain these results, we proposed that the following ligand-exchange reaction;



was effective in this system. Fig. 4 shows the emission spectrum obtained for the mercury-photosensitized reaction of a tert-BAL-TMSDMA (13.3:1) mixture at 860 Pa. As shown in Fig. 4, two peaks are observed (at 300 nm and at 355 nm). The short wavelength band is attributed to the Hg-tert-BAL exciplex emission, and the long wavelength band is attributed to the Hg-TMSDMA exciplex produced by the ligand-exchange reaction. Similar emission spectra were obtained in mixtures of tert-BAL with other silylamines and

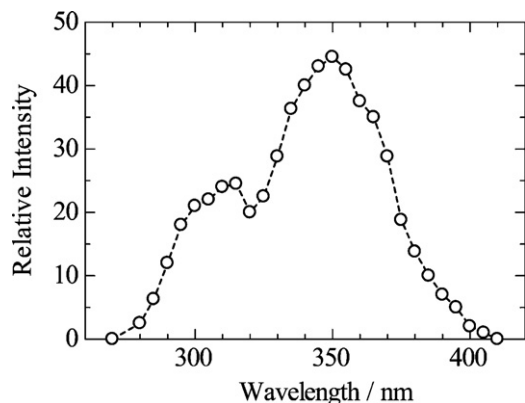


Fig. 4. Emission bands obtained in the Hg(3P_1)-photosensitized reaction of a TMSDMA(60 Pa)-tert-BAL(800 Pa) mixture at 298 K.

alkylamines. In these mixtures, it was also observed that the emission intensity of the alcohol exciplex decreased with an increase in the amine/alcohol ratio, when amines were added to a constant amount of tert-BAL. On the other hand, the emission intensity of the amine exciplexes increased with an increase in the tert-BAL pressure, when tert-BAL was added to constant amounts of amines. The peak wavelengths of the emission band and the quantum yields of the emissions from the amine exciplexes obtained in the presence of tert-BAL are listed in Table 2. The quantum yields of the silyl- and alkylamine emissions were determined by comparing the integrated intensities of the emission bands of these amines with that of ammonia (whose quantum yield is known [0.70 [23]]), under the same conditions.

Fig. 5 shows the Stern–Volmer plots of the quenching of the emission from the Hg-tert-BAL exciplex by some silylamines. The half-quenching pressures for the quenching of the alcohol exciplex emission by silylamines and secondary and tertiary alkylamines are obtained from the slopes of the straight lines and are listed in Table 2.

From the variation of luminescence efficiency with the structures of amines and the results of product analysis for the mercury-photosensitized reaction of amines, Newman et al. concluded that the main process competing with luminescence is the abstraction of an α -hydrogen atom of amines [14]. They also pointed out that, for secondary and tertiary amines, the absence of sensitized luminescence is due to the abundance of α -hydrogen or steric effect during collision with excited mercury atoms. Indeed, no significant sensitized emission band was observed in the mercury-photosensitized reactions of secondary and tertiary alkyl- and silylamines. As mentioned above, however, upon addition of tert-butyl alcohol to the amines, we observed emission bands at around 350 nm (Fig. 4). For the cadmium-photosensitized reaction of amines, the abstraction of an α -hydrogen atom is only a minor

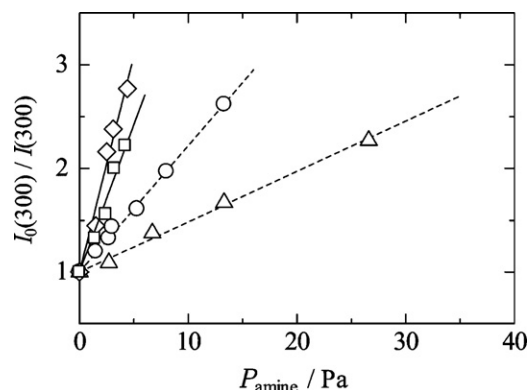


Fig. 5. Stern–Volmer plots for the quenching of the mercury exciplex emission of tert-BAL at 300 nm by TEMDSAC (○), HXMDS (◇), TMSDMA (△), and TMSPY (□).

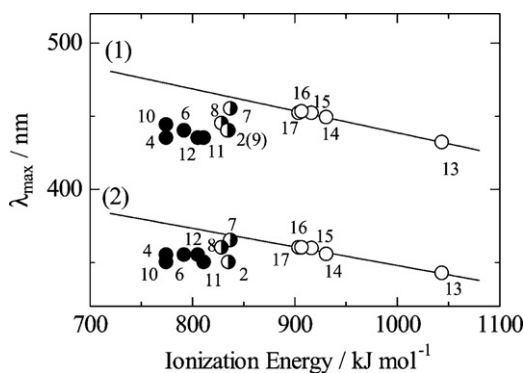


Fig. 6. Plots of the wavelength of the peak intensity of the emission band obtained in (1) cadmium- and (2) mercury-photosensitized reactions against the first ionization energy of amines and silylamines (the values of peak wavelength for ammonia and primary amines are quoted from Refs. [11,14] and the values of the ionization energy are cited from Refs. [25–27]). Ammonia and primary amines (○), secondary alkyl- and silylamines (◐), and tertiary alkyl- and silylamines (●). The lines show correlations between the peak wavelengths and the ionization potentials of ammonia and primary amines. The numbers up to 12 correspond to the compounds listed in Tables 1 and 2; 13, NH₃; 14, MeNH₂; 15, EtNH₂; 16, PrNH₂; and 17, BuNH₂.

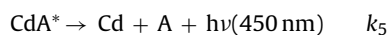
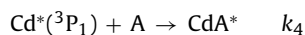
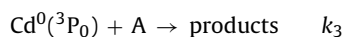
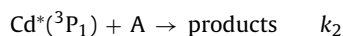
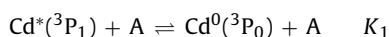
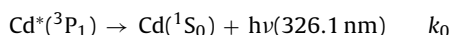
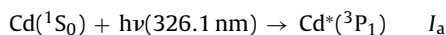
process, and emission bands were observed for pure secondary and tertiary alkyl- and silylamines.

4. Discussion

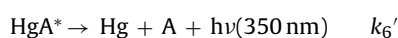
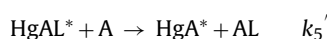
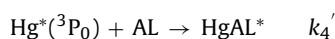
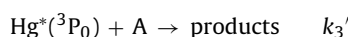
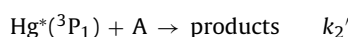
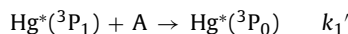
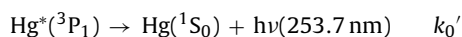
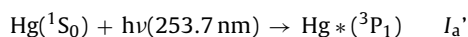
First, we consider the effect of the structure of the substrates on the position of the emission band. As shown in previous papers, the peak wavelengths of the mercury- and cadmium-photosensitized luminescence of primary amines decrease with an increase in the first ionization energies of the amines [11,14]. In Fig. 6, the peak wavelengths of the cadmium- and mercury-photosensitized luminescence of alkyl and silylamines are plotted as a function of first ionization energies of the amines. As reported previously [14], there is a good correlation between the peak wavelength and the ionization energy for the mercury-photosensitized luminescence of ammonia and primary amines (shown by the straight lines in Fig. 6). Similar correlation is also seen for the cadmium-photosensitized luminescence of ammonia and primary amines. As pointed out previously [11,14], the exciplexes formed between the excited mercury and cadmium atoms, and the amines are of the charge-transfer type. However, the peak wavelengths for secondary and tertiary alkyl- and silylamines are somewhat smaller than the values predicted from the correlations mentioned above. The wavelengths for tertiary amines deviated downward to a greater degree from the straight correlation lines than those for secondary amines in both the cadmium- and mercury-photosensitized emissions. The stability of the cadmium and mercury exciplexes of amines is governed not only by the electron-donating ability of the nitrogen atom of amines but also, partly, by other factor(s). This additional factor would suppress the stability of the exciplexes in the cases of secondary and tertiary amines.

With respect to the quenching of the resonance line and the exciplex emission in the cadmium- and mercury-photosensitized reactions, the following reaction schemes may be considered in a manner similar to those described in the previous papers [14,17]:

For cadmium:



For mercury:



For the cadmium-photosensitized reaction, a steady-state treatment under the assumption that an equilibrium between Cd*(³P₁) and Cd⁰(³P₀) is established at 493 K leads to the following equation:

$$\frac{I_0}{I} = 1 + \frac{k_q}{k_0} [\text{A}] \quad (1)$$

Here, I_0 and I denote the intensities of the resonance line at 326.1 nm (defined as $k_0[\text{Cd}^*(^3\text{P}_1)]$) in the absence and in the presence of amines, respectively; k_q is the composite quenching rate constant given by $k_q = k_2 + K_1 k_3 + k_4$. The Stern–Volmer plots shown in Fig. 2 are expressed by Eq. (1).

The half-quenching pressure ($P_{1/2}$) is expressed as follows:

$$P_{1/2} = \frac{k_0}{k_q} \quad (2)$$

For the mercury-photosensitized reactions, the following equation is obtained by a steady-state treatment:

$$\frac{I_0'}{I'} = 1 + \frac{k_q'}{k_0'} [\text{A}] \quad (3)$$

Here, I_0' and I' denote the intensities of the resonance line at 253.7 nm (defined as $k_0'[\text{Hg}^*(^3\text{P}_1)]$) in the absence and in the presence of amines, respectively; k_q' is the composite quenching rate constant given by $k_q' = k_1' + k_2'$. The Stern–Volmer plots shown in Fig. 3 are expressed by Eq. (3). The half-quenching pressure ($P_{1/2}$) is expressed as follows:

$$P_{1/2} = \frac{k_0'}{k_q'} \quad (4)$$

Figs. 7 and 8 show the values of $P_{1/2}$ for the quenching of the cadmium and mercury resonance lines by alkyl- and silylamines. The quenching efficiencies of monosilylamines (compound numbers 4 and 5) are similar to those of disilylamines (compound numbers 1, 2, 3, and 6). As shown in Figs. 7 and 8, the values of $P_{1/2}$ for secondary alkyl- and silylamines are generally lower than those for tertiary alkyl- and silylamines in the cadmium- and mercury-photosensitized reactions. This indicates that the quenching efficiencies for secondary alkyl- and silylamines are higher than those for tertiary alkyl- and silylamines.

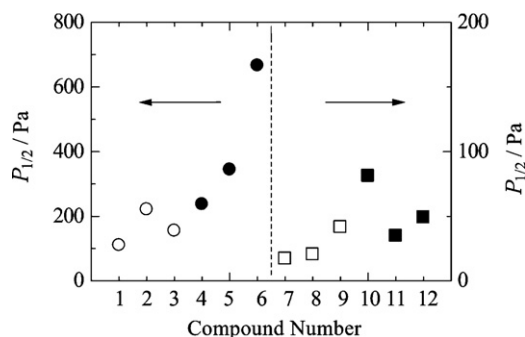


Fig. 7. Plots of the values of $P_{1/2}$ for the quenching of the cadmium resonance line by alkyl- and silylamines against the compound number listed in Table 1. Secondary alkylamines (\square), tertiary alkylamines (\blacksquare), secondary silylamines (\circ), tertiary silylamines (\bullet).

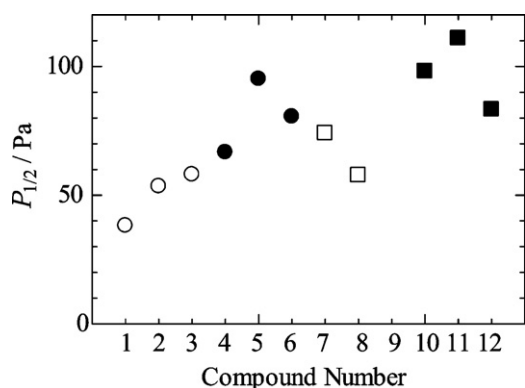


Fig. 8. Plots of the values of $P_{1/2}$ for the quenching of the mercury resonance line by alkyl- and silylamines against the compound number listed in Table 2. Secondary alkylamines (\square), tertiary alkylamines (\blacksquare), secondary silylamines (\circ), tertiary silylamines (\bullet).

The relationship between the relative rate constant for the formation of the exciplex (k_4 in this study) and the ionization energy of the substrates in the cadmium-photosensitized reaction was discussed in the previous papers [11,24]. In general, the rate constant was found to increase with a decrease in the ionization energy. It was also found that there is a good correlation between the rate constants and the ionization energies for primary amines and alcohols; however, the rate constants for secondary and tertiary amines and ethers were relatively lower than the values predicted from the abovementioned correlation (the rate constants for tertiary amines deviated downward to a greater degree from the straight line than those for secondary amines). As indicated in the previous paper, the reactivity of amines is not only mainly governed by the electron-donating ability of the nitrogen atom but also partly governed by another factor. This additional factor suppressed the formation of the exciplex in secondary and tertiary amines. The origin of this additional factor was not identified in the previous paper. As already mentioned above, the quenching efficiencies of the cadmium and mercury resonance lines by tertiary alkyl- and silylamines are lower than those by secondary ones; this additional factor seems to provide steric hindrance to the approach of the nitrogen atom in alkyl- and silylamines to the excited cadmium and mercury atoms.

Similar tendencies were observed for the quenching of the mercury-exciplex emission of tert-BAL by amines, with an exception for tertiary silylamines.

5. Conclusion

The mercury-photosensitized luminescence of silylamines was observed for tert-butyl alcohol-silylamine mixtures for the first time. The peak wavelengths for secondary and tertiary alkyl- and silylamines are slightly shorter than the values predicted from the correlations between the peak wavelength and the first ionization energy in both the cadmium- and mercury-photosensitized emissions of ammonia and primary amines. The wavelengths for tertiary amines deviated downward to a greater degree from the straight lines than those for secondary amines (Fig. 6). The quenching efficiencies of the cadmium and mercury resonance lines by secondary alkyl- and silylamines were found to be higher than those by tertiary alkyl- and silylamines (Figs. 7 and 8). These results indicate that the stabilities of the cadmium- and mercury-exciplexes and the quenching efficiencies of the cadmium and mercury resonance lines are affected by crowding around the nitrogen atom in alkyl- and silylamines. Silylamines exhibit a behavior similar to that of alkylamines in the cadmium- and mercury-photosensitized reactions. The wavelength shifts of the cadmium- and mercury-photosensitized emissions would provide information on electron-donating ability and steric effects of series of alkyl- and silylamines, thus, would be useful to evaluate the nature of the silylamines in comparison with the corresponding alkylamines.

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