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Photochemical formation of ammonium/thiolate complexes from quaternary ammonium thiocyanates and its use in crosslinking of polymers

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Abstract

1-Phenacyl-(1-azania-4-azabicyclo[2.2.2]octane) thiocyanate (PnDbSCN) and *N*-phenacyl-4-(*N*′,*N*′-dimethylamino)pyridinium thiocyanate (PnDpSCN) were prepared as novel photobase generators. These quaternary ammonium salts (QASs) have higher melting points and decomposition temperatures than corresponding QASs having dithiocarbamate anions. From ^{1}H NMR and UV spectral analyses, it was found that PnDbSCN turned to 1,4-diazabicyclo[2.2.2]octane (DABCO) and phenacylthiocyanate (PnSCN) on irradiation at 254 nm, and the resulting PnSCN was further transformed into 5-phenyloxazole-2-thione (POT) in the presence of DABCO to form ammonium/thiolate complexes. PnDpSCN also showed similar ^{1}H NMR spectral changes on irradiation. Poly(glycidyl methacrylate) (PGMA) films containing amines in the presence of POT crosslinked at lower temperature than those in the absence of POT, and PGMA films containing QASs became insoluble on irradiation followed by post-exposure baking.

Keywords: Quaternary ammonium salt; Photobase generator; Tertiary amine; Thiocyanate anion; Crosslinking

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1. Introduction

Photobase generator (PBG) is attractive as a novel family of catalyst for polymerization or crosslinking of polymeric materials as well as photoacid generators [1]. However, there are not so many reports concerning PBGs. Although tertiary amines have higher activity as a base catalyst than primary and secondary amines, the example of PBGs that photochemically release tertiary amines is rare. Some quaternary ammonium salts (QASs) were proposed as photochemical precursors of the tertiary amines by Jensen and Hanson [2] and Neckers and co-workers [3–10].

We have already reported that QASs having dithiocarbamate anions were highly photoreactive PBGs and generated tertiary amines on irradiation as shown in Scheme 1 [11]. The amines worked as a catalyst of crosslinking of polymers and oligomers bearing epoxy groups. However, these QASs were unstable in solutions and films. Recently, we have found that QASs having thiocyanate anions showed higher stability than those having dithiocarbamates [12,13].

In this article, we report the photochemical reactions and thermal properties of QASs having thiocyanate anion along with theiability as crosslinking reagents of polymers bearing epoxy groups. Two QASs, 1-phenacyl-(1-azania-4-azabicyclo[2.2.2]octane) thiocyanate (PnDbSCN) and *N*-phenacyl-4-(*N*′,*N*′-dimethylamino)pyridinium thiocyanate (PnDpSCN) were examined as photochemical precursors of strong bases such as 1,4-diazabicyclo[2.2.2]octane (DABCO) and *N*,*N*-dimethylaminopyridine (DMAP).

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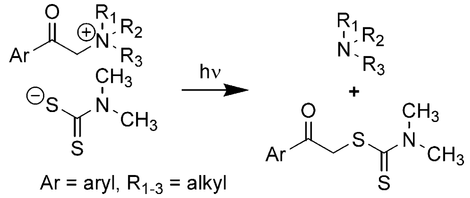


Scheme 1. Photoreaction of QASs having dithiocarbamate anions.

2. Experimental details

2.1. Apparatus

Melting points for QASs were obtained by using a Rigaku 8230B differential scanning calorimeter. Melting points for other compounds were measured by a Yanako MT-3S micro melting point meter and uncorrected. IR, UV, and NMR spectral measurements were performed using Jasco FTIR410, Shimadzu 2400PC, and Jeol JNM-GX270 (270 MHz) spectrometers, respectively. Molecular weights of polymers were determined by size exclusion chromatography (SEC) on equipment consisting of a Jasco PU-980 pump, a Jasco RI-930 detector, and a Shodex KF-806M column with polystyrene standards and tetrahydrofuran (THF) as an eluent. Thermogravimetric analysis (TGA) was carried out using a Shimadzu TGA50 thermogravimetric analyzer with a heating rate of 10 K/min under N_2. Onset temperatures from TGA curves were adopted as thermal decomposition temperature (T_d). Light intensity was measured by an Orc UV-M02 illuminometer. Film thickness was measured by a Nanometrics Nanospec/AFT M3000. Post-exposure bake (PEB) was carried out on a Koike HM-15G Hotplate.

2.2. Materials

DMAP and DABCO were obtained from Wako Chemical (Osaka, Japan).

Both phenacylthiocyanate (PnSCN) and 5-phenyloxazole-2-thione (POT) were prepared from phenacyl bromide and sodium thiocyanate at 0 °C in methanol and at 150 °C in *N*,*N*-dimethylformamide, respectively. After recrystallization, melting points of PnSCN and POT were 70.5–71.5 °C (literature [14], 74.5–75 °C) and 231–234 °C (literature [15], 234–235 °C), respectively. PnDbSCN was prepared as reported previously [11].

N-Phenacyl-4-(*N*′,*N*′-dimethylamino)pyridinium bromide (PnDpBr) was obtained by mixing methanol solutions of phenacyl bromide and DMAP in a ice-cold bath followed by evaporation and recrystallization from ethanol: yellowish powder in 87.4% yield. mp = 223–223.5 °C. IR (KBr): 1690 cm^{-1} (C=O). ^{1}H NMR (D_2O): $\delta = 3.08$ (6H, s, NCH_3), 5.70 (2H, d, $J = 5.1$ Hz, CH_2), 6.78 (2H, d, $J = 7.5$ Hz, 3- and 5-H in pyridine ring), 7.45–8.89 (7H, m, aromatic).

N-Phenacyl-4-(*N*′,*N*′-dimethylamino)pyridinium *N*″,*N*″-dimethyldithiocarbamate (PnDpDt) was obtained by mixing methanol solutions of 2.0 g (6.2 mmol) of PnDpBr and 0.93 g (6.5 mmol) of sodium *N*,*N*-dimethyldithiocarbamate followed by evaporation and recrystallization from ethanol: 1.03 g (45.8%) of yellow needles. IR (KBr): 1705 (C=O), 975 cm^{-1} (NC=S). ^{1}H NMR (CD_3CN): $\delta = 3.20$ (6H, s, $ArNCH_3$), 3.45 (6H, s, S=C–N–CH_3), 5.86 (2H, s, CH_2), 6.89 (2H, d, $J = 8.1$ Hz, 3- and 5-H in pyridine ring), 7.58–8.00 (7H, m, aromatic). Calcd. for $C_{18}H_{23}N_3OS_2$—C: 59.80, H: 6.41, N: 11.62; found C: 59.93, H: 6.40, N: 11.56.

N-Phenacyl-4-(*N*′,*N*′-dimethylamino)pyridinium thiocyanate (PnDpSCN) was obtained by a similar procedure to PnDpDt: colorless solid in 40.5% yield. IR (KBr): 2040 (SCN), 1700 (C=O) cm^{-1}. ^{1}H NMR (CD_3CN): $\delta = 3.22$ (6H, s, CH_3), 5.74 (2H, s, CH_2), 6.91 (2H, d, $J = 7.5$ Hz, 3- and 5-H in pyridine ring), 7.61–8.02 (7H, m, aromatic). ^{13}C-NMR (CD_3CN): $\delta = 40.73$, 63.54, 108.37, 118.29, 129.11, 130.10, 134.94, 135.54, 144.00, 157.56, 192.86. Calcd. for $C_{16}H_{17}N_3OS$—C: 64.19, H: 5.72, N: 14.04; found C: 64.23, H: 5.73, N: 13.98.

Poly(glycidyl methacrylate) (PGMA) and poly(methyl methacrylate) (PMMA) were prepared by radical polymerization using α,α′-azobisisobutylonitrile as an initiator. After reprecipitation, number average molecular weights of PGMA and PMMA were 138,000 and 107,000, respectively.

The stability of QASs in solution was monitored by UV spectral changes.

2.3. Equilibrium constant

Equilibrium constant of ammonium/POT complex formation (K) in the presence of excess amine is represented as Eq. (1),

$$K = \frac{[\mathrm{C}]}{([\mathrm{POT}] - [\mathrm{C}])([\mathrm{A}] - [\mathrm{C}])} \approx \frac{[\mathrm{C}]}{([\mathrm{POT}] - [\mathrm{C}])[\mathrm{A}]} \qquad (1)$$

where [C], [POT], and [A] are the concentrations of the complex, POT, and the amine, respectively. From Lambert–Beer equation,

$$\frac{A}{l} = \varepsilon'_{\mathrm{POT}}[\mathrm{POT}] = \varepsilon_{\mathrm{POT}}([\mathrm{POT}] - [\mathrm{C}]) + \varepsilon[\mathrm{C}] \qquad (2)$$

where $\varepsilon_{\mathrm{POT}}$ and ε are molar absorption coefficients of POT and the complex at 350 nm, respectively, l is 1 cm as path length of the cell, and A is the apparent absorbance of sample at 350 nm. $\varepsilon'_{\mathrm{POT}}$ is the apparent molar absorption coefficient of POT in the mixture. From Eqs. (1) and (2), $1/(\varepsilon'_{\mathrm{POT}} - \varepsilon_{\mathrm{POT}})$ can be related to 1/[A] in Eq. (3) as Ketelaar introduced previously [16],

$$\frac{1}{\varepsilon'_{\mathrm{POT}} - \varepsilon_{\mathrm{POT}}} = \frac{1}{K(\varepsilon - \varepsilon_{\mathrm{POT}})[\mathrm{A}]} + \frac{1}{\varepsilon - \varepsilon_{\mathrm{POT}}} \qquad (3)$$

2.4. Irradiation and crosslinking

UV spectral changes for QASs in solutions were measured in a tube bearing 1 cm × 1 cm quartz cuvette. Before irradiation, the solution was degassed by freeze–pump–thaw cycles in the tube. Irradiation was performed in the cuvette with an Ushio ULO-6DQ low-pressure mercury lamp (6 W). The light intensity at 254 nm was 1.0 mW/cm^2.

PMMA and PnDbSCN (15 mol% against MMA unit) were dissolved in a mixture of THF:ethanol = 6:1 (v/v), and the resulting solution was cast into a Petri dish followed by slow evaporation. Thickness of the films was ca. 50 μm.

PGMA films containing POT, amine, and QAS were obtained by spin-coating on silicone wafers from cyclohexanone/acetonitrile (5/2, v/v) solutions. Film thickness was ca. 0.5 μm.

Insoluble fraction of PGMA films was obtained from the ratio of thickness before and after soaking the films in THF for 10 min.

2.5. ^{1}H NMR measurement and calculation of quantum yields

In a quartz tube (o.d. = 13 mm Ø), 10 ml of QAS solution (1.5 mM in acetonitrile) and a stirrer piece were put and degassed by bubbling argon gas for 20 min. The tube was cocked, and irradiated with the ULO-6DQ with stirring by a magnetic stirrer. The light intensity was measured by potassium trioxalate actinometer to be 2.5 mW at 254 nm for this amount of solution. After irradiation for a given time, solvent was removed by evaporation. To the residue, 0.80 ml of CD_3CN containing 15 or 40 μmol of triethylsilane as a standard was added, and ^{1}H NMR spectra were measured.

Quantum yields of photolysis of QASs (Φ_{dec}) at 254 nm was obtained from the following equation:

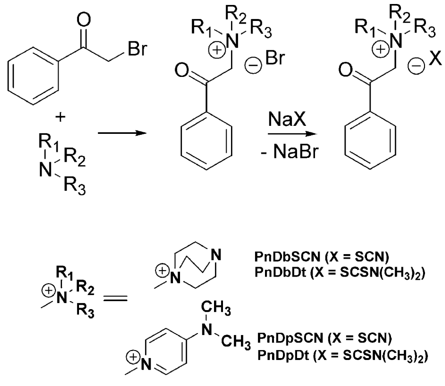
$$\Phi_{\mathrm{dec}} = \frac{\Delta M_{\mathrm{QAS}}}{0.025t} \qquad (4)$$

where t is irradiation time and ΔM_{QAS} is the amount of reacted QASs based on the area in the NMR spectra.

3. Results and discussion

3.1. Synthesis and physical properties of QASs having thiocyanate anion

PnDpSCN and PnDpDt were successfully prepared from phenacyl bromide and DMAP followed by anion exchange by a method similar to PnDbSCN [11] as shown in Scheme 2. Physical properties of QASs are listed in Table 1. T_d's of PnDbSCN and PnDpSCN were higher than PnDbDt and PnDpDt, respectively. Furthermore, no changes in UV spectra of PnDbSCN and PnDpSCN in methanol or acetonitrile were observed after standing for 24 h at room temperature. The molar absorption coefficients of QASs having thiocyanate at 254 nm were about half of corresponding QASs having dithiocarbamate.



Scheme 2. Synthesis of QASs.

3.2. Photochemical reaction of QASs having thiocyanate anion

Fig. 1 shows UV spectral changes for PnDbSCN and PnDpSCN on irradiation at 254 nm in acetonitrile. For both QASs, peaks around 250 nm due to phenacyl moiety decreased, which indicated the proceeding of photoreaction.

Expected photoproducts from QASs having thiocyanates are amines and phenacylthiocyanate as shown in Scheme 3, when a similar reaction shown in Scheme 1 occurs. However, the formation of amines could not be detected from irradiated solutions of PnDbSCN and PnDpSCN by colorimetric method using Phenol red, while the amine from PnDbDt in water was detected as reported previously [11].

It has been known that PnSCN photochemically isomerizes to phenacylthioisocyanate (PnNCS) [17], and furthermore, the thioisocyanate can cyclize to form POT in the presence of base [14,15]. Therefore, the amine seems to be trapped with POT to form a complex as shown in Scheme 4. In fact, on irradiation of PnDbSCN in PMMA matrix, shoulders appeared at 310 and 325 nm in UV spectra as shown in Fig. 2. The former shoulder was consistent with the result of irradiation of PnSCN in PMMA film as reported previously [13], where strong peak appeared at 308 nm on irradiation followed by NH_3-gas treatment in UV spectrum. The latter peak can be assigned to that of ammonium/POT complex. Fig. 3 shows bathochromic shift of POT in the presence of excess amount of DABCO.

Table 1
Physical properties of QASs

QAS	mp[a] (°C)	T_d[b] (°C)	λ_{max}[c] (nm)	ε[c] (l/(mol cm))	
				At λ_{max}	At 254 nm
PnDbSCN	172	224	245	11800	9900
PnDpSCN	114	251	290	30900	11000
PnDbDt	118	125[d]	295[d]	11900[d]	21200[d,e]
PnDpDt	185	174	293	46200	25000

[a] Measured by DSC.
[b] Measured by TGA.
[c] In acetonitrile.
[d] Data from literature [11].
[e] At 251 nm.

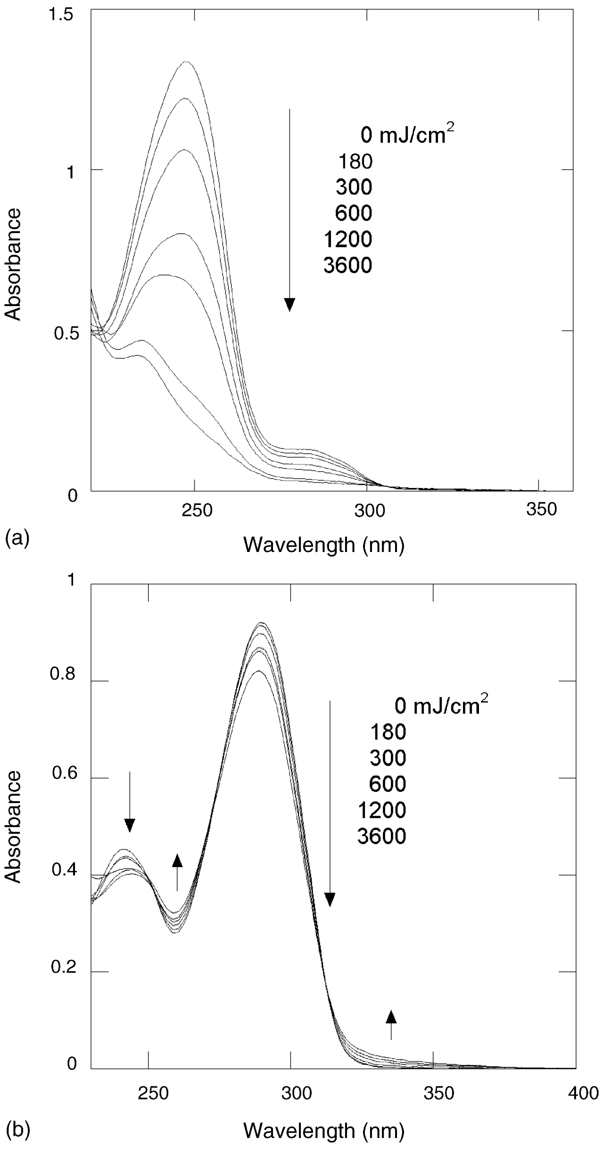
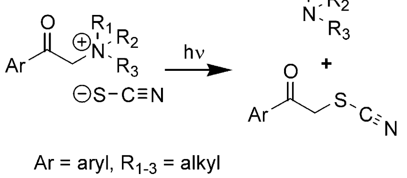
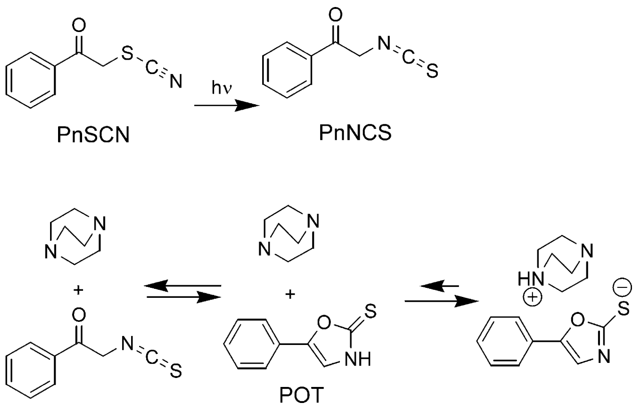


Fig. 1. UV spectral changes of (a) 1.1×10^{-4} M acetonitrile solution of PnDbSCN and (b) 2.3×10^{-5} M acetonitrile solution of PnDpSCN on irradiation under N_2. Numbers in the figure show irradiation energy.

By using Ketelaar relation [16], equilibrium constant (*K*) of the formation of DABCO/POT complex in the presence of excess DABCO can be obtained from the slope and intercept of the linear relation between $1/(\varepsilon'_{\mathrm{POT}} - \varepsilon_{\mathrm{POT}})$ and 1/[A] to be 1000 l/mol. Similar experiment for DMAP afforded *K* for DMAP/POT complex to be 1300 l/mol. Although the ratio of the complex formation depends on the concentration of POT and amines, these values of *K* indicate that some of POT exists as a complexed form in the presence of DABCO or DMAP because the concentration can be expected to be high in polymer matrix.



Scheme 3. Expected photoreaction of QASs having thiocyanate anions.



Scheme 4. Photoreaction of PnDbSCN.

Fig. 4 shows ^{1}H NMR spectral changes of PnDbSCN on irradiation in acetonitrile. Peaks c–e due to three kinds of methylene protons in PnDbSCN decreased with irradiation time. On irradiation at 96×10^{-6} einstein (Fig. 4d), peaks a–e disappeared and the new peaks appeared at 3.01 ppm. Methylene protons in DABCO at 2.65 ppm (Fig. 4f) shifted downfield to peak f by adding authentically prepared POT (Fig. 4e). When DABCO and POT (40 mol% against starting PnDbSCN) was added to the photolysate of PnDbSCN at 76×10^{-6} einstein, the peak intensity at 3.01 ppm increased with an upfield shift of 0.09 ppm. This result indicated that the new peak at 3.01 ppm in Fig. 4d was due to the methylene protons in DABCO in the presence of POT. The appearance

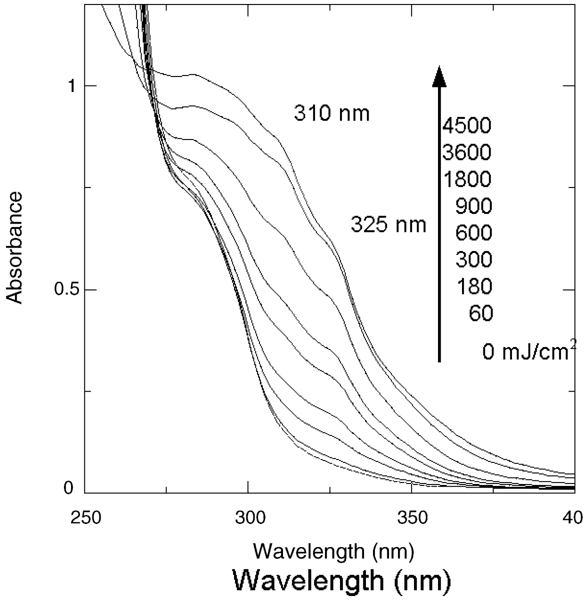


Fig. 2. UV spectral changes of PnDpSCN on irradiation in PMMA films. Film thickness: 50 μm. Numbers in the figure show irradiation energy.

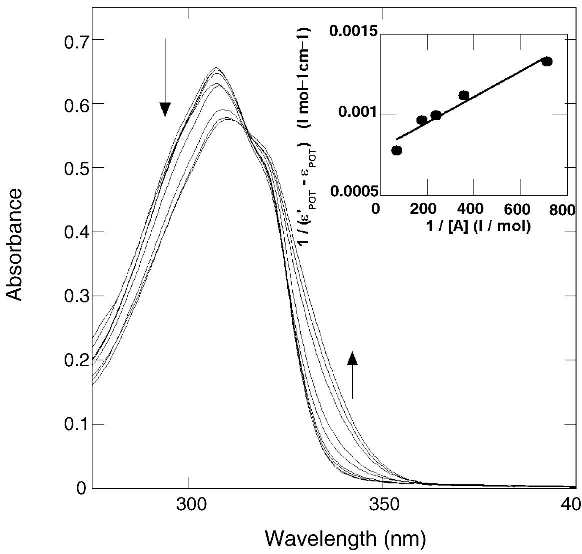


Fig. 3. UV spectra of POT in the presence of DABCO in methanol. [POT] = 2.80×10^{-5} M. [DABCO] × $10^5 = 0$ (top at 308 nm), 1.4, 14, 28, 140, 280, 420, 560, and 1400 (bottom) M. Inset shows a plot of $1/(\varepsilon'_{\mathrm{POT}} - \varepsilon_{\mathrm{POT}})$ as a function of 1/[A].

of another new peaks in Fig. 4d at 7.2–7.6 ppm in aromatic region suggests the formation of POT.

A similar spectral change was observed for the irradiation of PnDpSCN as shown in Fig. 5. Peaks k, l, j, and m due to DMAP moiety decreased on irradiation, and new peaks appeared at 3.13 and 6.7 ppm, suggesting the formation of DMAP/POT complexes. The assignment of the former peak was also confirmed by the increase in peak intensity at 3.13 ppm with an upfield shift of 0.07 ppm by adding DMAP and POT (10 mol% against starting PnDpSCN) to the photolysate of PnDpSCN at 24×10^{-6} einstein.

Table 2
Quantum yields of photolysis of QASs in acetonitrile

QAS	Φ_{dec}
PnDbSCN	0.11
PnDpSCN	0.043

Based on the area of peaks e and m, the degree of photoreaction of PnDbSCN and PnDpSCN are plotted as a function of absorbed photons in Fig. 6, and the slopes gave quantum yields of photolysis of QASs (Φ_{dec}) as listed in Table 2. These values would be lower than that of PnDbDt based on the UV spectral changes. It is difficult to measure Φ_{dec} of PnDbDt because of its instability in solutions. Φ_{dec} of PnDbSCN was higher than that of PnDpSCN. Although the reason of the difference is unclear at present, it is indicated that the photoreactivity of QASs was much influenced by the structure of amines.

3.3. Crosslinking of polymers bearing epoxy groups

Amines work as a catalyst for crosslinking of polymers bearing epoxy groups as shown in Scheme 5. Thiols are known as a good accelerator for curing of epoxy/amine systems due to the formation of reactive thiolate anions in the presence of amines [18]. Fig. 7 shows the effect of POT on the thermal crosslinking of PGMA films containing amine.

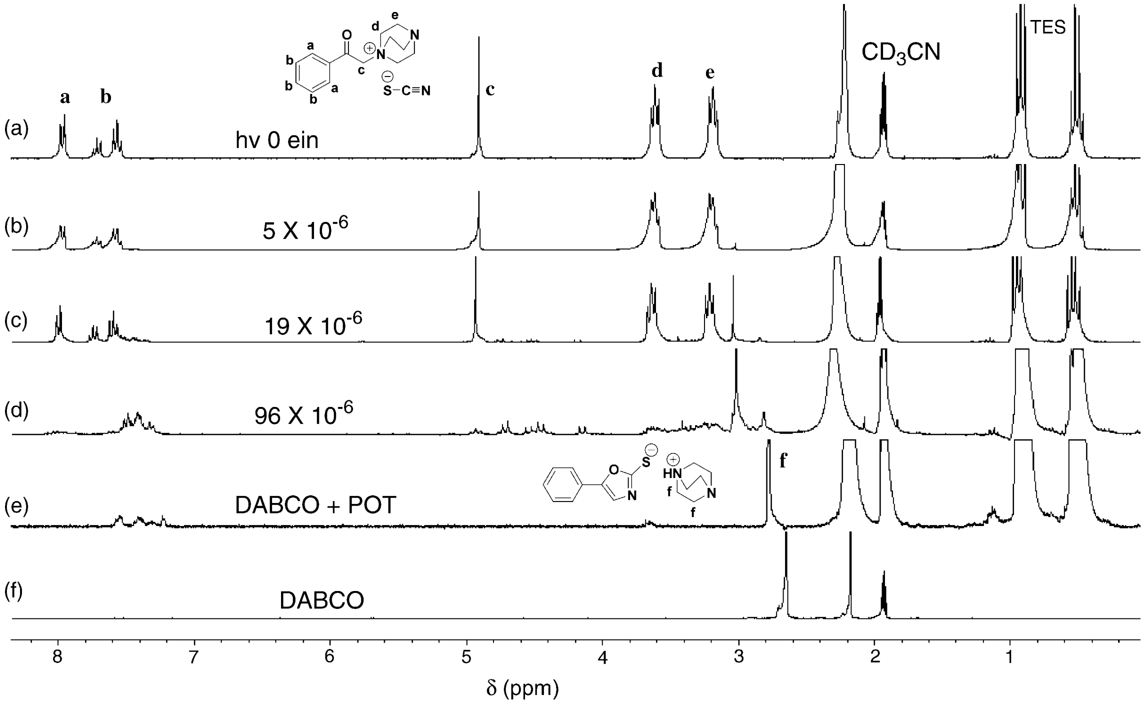


Fig. 4. ^{1}H NMR spectra (CD_3CN) of PnDbSCN on irradiation at (a) 0, (b) 5×10^{-6}, (c) 19×10^{-6}, (d) 96×10^{-6} einstein in acetonitrile, (e) DABCO:POT = 1:1 mixture, and (f) DABCO only. Tetraethylsilane was involved as an internal standard.

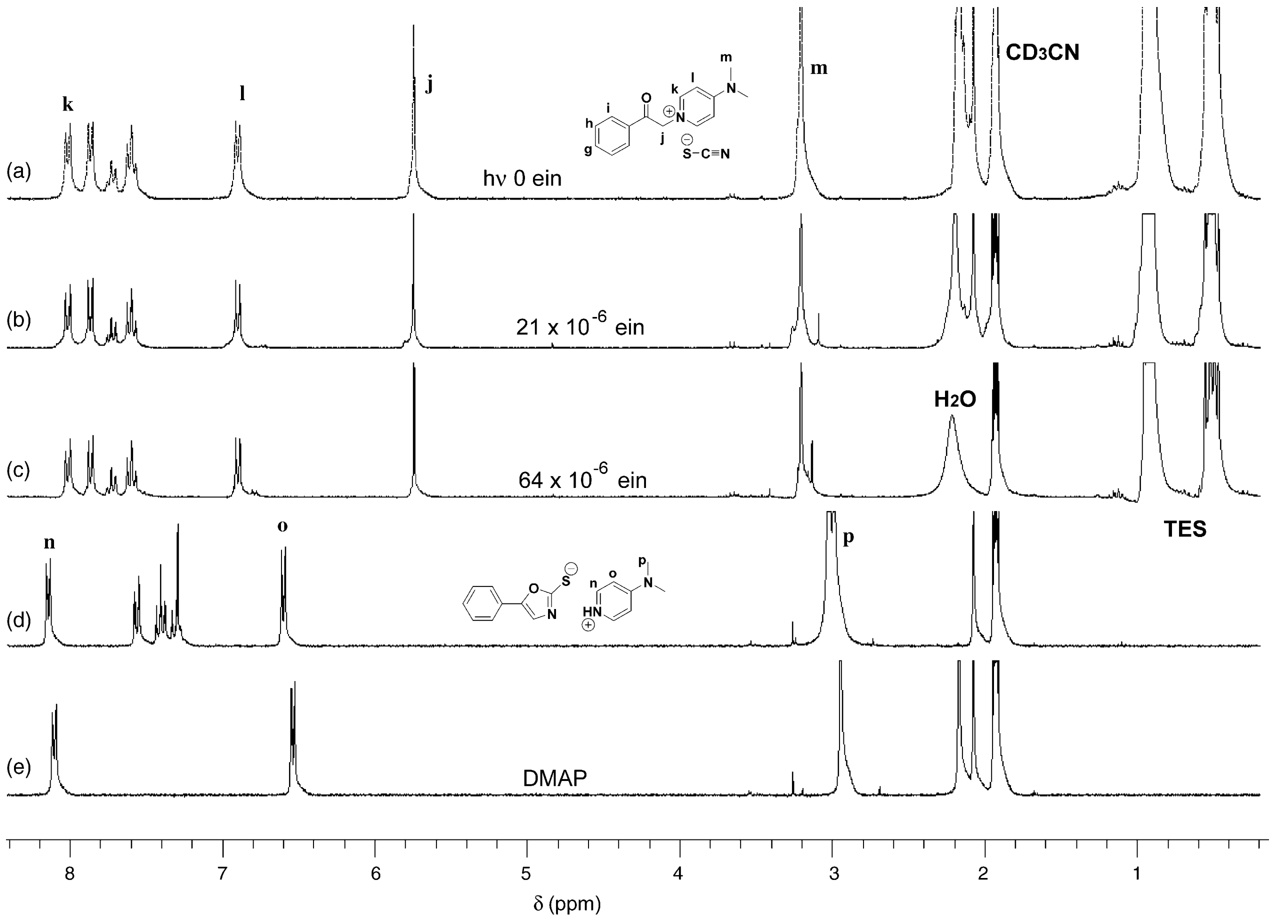


Fig. 5. ^{1}H NMR spectra (CD_3CN) of PnDpSCN on irradiation at (a) 0, (b) 21×10^{-6}, (c) 64×10^{-6} einstein in acetonitrile, (d) DMAP:POT = 1:1 mixture, and (e) DMAP only.

Films became insoluble in THF when baked at 70–80 °C in the presence of DABCO or DMAP. Addition of POT effectively decreased the onset temperature for insolubilization, even though simple POT did not cause the crosslinking. This effect was also observed for the insolubilization of epoxy-*o*-cresol novolac resin/tris(2-hydroxyethyl)isocyanurate tris(3-

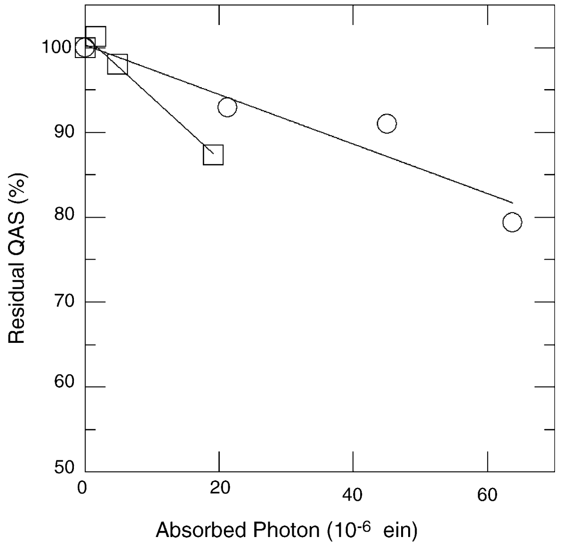
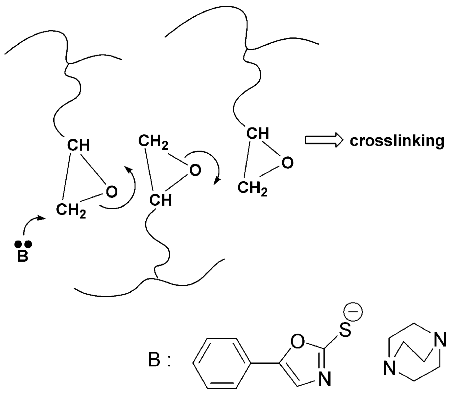


Fig. 6. Changes of residual (□) PnDbSCN and (○) PnDpSCN on irradiation in acetonitrile based on the area changes of peaks e and m in Figs. 4 and 5, respectively.



Scheme 5. Crosslinking of polymers bearing epoxy groups catalyzed by amine or thiolate.

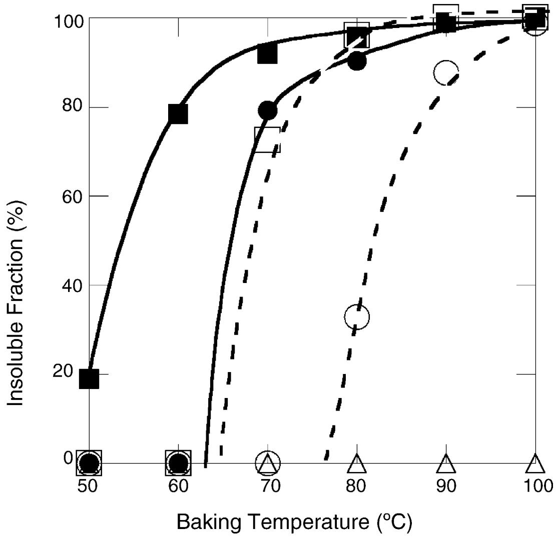


Fig. 7. Insolubilization of PGMA films containing additives after baking for 3 min. (■) DABCO + POT, (□) DABCO, (●) DMAP + POT, (○) DMAP, and (△) POT only. [GMA unit]:[amine]:[POT] = 100:5:5 (mol/mol).

mercaptopropionate) system [19]. These results indicate that ammonium-thiolate complex formation was advantageous for crosslinking polymers bearing epoxy groups.

Photochemical crosslinking of PGMA films containing PnDbSCN and PnDpSCN was attempted. Fig. 8 shows the insolubilization of PGMA films containing QASs as a function of irradiation time. Films containing PnDbDt became insoluble on short irradiation due to high photoreactivity and greater absorbance at 254 nm. Both films containing PnDbSCN and PnDpSCN were essentially soluble only on irradiation in spite that some amounts of ammonium/POT complexes were generated. However, films became insoluble after post-exposure bake (PEB) due to crosslinking as shown in Scheme 5. The difference of irradiation time required to the insolubilization between PnDbSCN and PnDpSCN was due to the difference of Φ_{dec} as shown in Table 2. We could find an optimum process condition for the selective insolubilization at irradiated areas with the assist of PEB at 60 °C for 3 min, which enable us to apply this system to imaging materials.

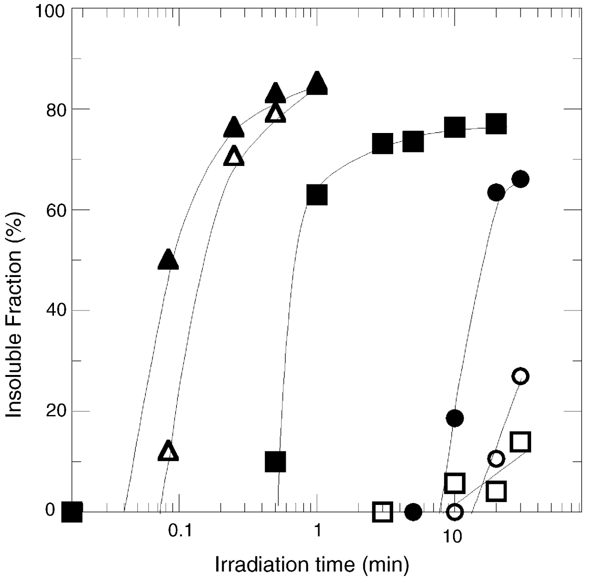


Fig. 8. Insolubilization of PGMA films containing (▲, △) PnDbDt, (□, ■) PnDbSCN, and (○, ●) PnDpSCN on irradiation followed by PEB. Open symbols: irradiation only. Solid symbols: irradiation followed by baking at 60 °C for 3 min. [GMA unit]:[amine]:[POT] = 100:5:5 (mol/mol).

4. Conclusion

QASs having thiocyanate anions were prepared, and their photochemical behavior was investigated. UV and ^{1}H NMR spectral changes of PnDbSCN on irradiation indicated the formation of ammonium/POT complex. Similar ^{1}H NMR changes were observed for PnDpSCN. Thermal insolubilization of PGMA films containing amines was enhanced by the addition of POT. Both PnDbSCN and PnDpSCN showed their catalytic abilities in crosslinking of PGMA films on irradiation followed by PEB.

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References

[1] K. Dietliker, in: G. Bradley (Ed.), Photoinitiators for Free Radical Cationic & Anionic Photopolymerization, second ed., John Wiley & Sons, Chichester, England, 1998 (Chapter IV).
[2] K.H. Jensen, J.E. Hanson, Chem. Mater. 14 (2002) 918–923.
[3] S. Hassoon, A.M. Sarker, M.A.J. Rodgers, D.C. Neckers, J. Am. Chem. Soc. 117 (1995) 11369–11370.
[4] A. Mejiritski, A.Y. Polykarpov, A.M. Sarker, D.C. Neckers, Chem. Mater. 8 (1996) 1360–1362.
[5] S. Hassoon, A.M. Sarker, A.Y. Polykarpov, M.A.J. Rodgers, D.C. Neckers, J. Phys. Chem. 100 (1996) 12386–12393.
[6] M. Sarker, A. Lungu, D.C. Neckers, Macromolecules 29 (1996) 8047–8052.
[7] M. Sarker, A. Mejiritski, B.R. Wheaton, D.C. Neckers, Macromolecules 30 (1997) 2268–2273.
[8] Y. Kaneko, A.M. Sarker, D.C. Neckers, Chem. Mater. 11 (1999) 170–176.
[9] W. Zhang, K.F. Xiaosong, D. Martin, D.C. Neckers, J. Org. Chem. 64 (1999) 458–463.
[10] M. Sarker, Y. Kaneko, D.C. Neckers, Chem. Mater. 13 (2001) 3949–3953.
[11] H. Tachi, M. Tsunooka, J. Polym. Sci. Part A: Polym. Chem. 39 (2001) 1329–1341.
[12] M. Tsunooka, Y. Kurokawa, T. Yamamoto, K. Suyama, M. Shirai, J. Photopolym. Sci. Technol. 15 (2002) 47–50.
[13] K. Suyama, K. Fuke, M. Tsunooka, M. Shirai, J. Photopolym. Sci. Technol. 16 (2003) 83–86.

[14] M. Tsunooka, Y. Cha, M. Tanaka, Makromol. Chem., Rapid Commun. 2 (1981) 491–493.

[15] M. Tsunooka, Y. Cha, M. Tanaka, F. Konishi, J. Polym. Sci. Polym. Chem. Ed. 22 (1984) 739–747.

[16] J.A.A. Ketelaar, C. van de Stope, A. Goudsmit, W. Dzacubas, Rec. Trav. Chim. 71 (1952) 1104.

[17] W. Kern, K. Hummel, D. Koller, N. Sadaghiani, Makromol. Chem. 194 (1993) 2759–2769.

[18] S. Katogi, M. Yusa, M. Shirai, M. Tsunooka, Chem. Lett. 32 (2003) 418–419.

[19] M. Tsunooka, K. Suyama, K. Fuke, M. Shirai, Proc. RadTech Eur., Berlin, Germany, 2003, pp. 687–692.