3048 © 1994 The Chemical Society of Japan Bull. Chem. Soc. Jpn., 67, 3048—3052(1994) [Vol. 67, No. 11

# Silver(I) Ion-Mediated Desulfurization-Condensation of Thiocarbonyl Compounds with Several Nucleophiles

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(Received May 13, 1994)

The desulfurization-condensation reaction was investigated in the presence of silver(I) salt for several kinds of thiocarbonyl compounds with nucleophiles. Such thiocarbonyl compounds as 4,4′-bis(dimethylamino)thiobenzophenone, ethylene trithiocarbonate, and *N*-(thiobenzoyl)morpholine react with malononitrile or other active methylene compounds in the presence of silver trifluoroacetate to afford the corresponding olefinic compounds together with silver sulfide. This reaction of thiocarbonyl compounds with some other nucleophiles, such as aniline derivatives and ethylene glycol, has also afforded imines and 1,3-dioxolane derivatives.

It is well known that divalent sulfur compounds have strong affinity with the silver(I) ion. There have been many studies concerning the cleavage of the C–S bond$^{1-10)}$ or S–S bond$^{11,12)}$ by the silver(I) ion. In the field of photographic chemistry, it has been suggested that the interaction process of thiocarbonyl groups with silver halides plays a crucial role.$^{13,14)}$ However, few studies have been performed concering thiocarbonyl compounds regarding the relation of the silver(I) ion in the field of organic synthesis.

On the other hand, although thiocarbonyl groups have different reactivities toward nucleophiles$^{15,16)}$ compared with carbonyl groups, it has not been studied in detail because of a lack of stability.

Here, we report on some silver(I) ion-mediated desulfurization-condensation of thiocarbonyl compounds with such nucleophiles as active methylene compounds, aniline derivatives and ethylene glycol as an application of the sulfur–silver(I) interaction in the field of organic synthesis.

## Results and Discussion

**Active Methylene Compounds.** We attempted a reaction of 4,4′-bis(dimethylamino)thiobenzophenone (**1**) as a thiocarbonyl compound with methyl cyanoacetate in the presence of triethylamine and silver trifluoroacetate in acetonitrile, 1,1-diphenylethylene derivatives (**2**) were obtained in excellent yields. This reaction didn't occur in the absence of a heavy metal salt, such as silver(I), mercury(II) or copper(I), or their oxide (Table 1). Copper(II) chloride was not effective in this reaction (Entry 5). In this reaction the formation of a metal sulfide, such as silver sulfide, was observed.

Silver trifluoroacetate was the most favorable, in that case the reaction proceeded even at room temperature, mainly due to its solubility in acetonitrile.

The same reaction of **1** with other active methylene compounds gave the corresponding condensation products in good yields (Table 2, Entries 1—4).

**Thiocarbonyl Compounds.** Attempts were also made to react other thiocarbonyl compounds with active methylene compounds. Ethylene trithiocarbonate (**7**) also reacts with some active methylene compounds to afford several products (**8**—**11**) in good yields (Table 2, Entries 5—8)(Chart 1).

Although *N*,*N*-dimethylthioformamide (**12**) and *N*-(thiobenzoyl)morpholine (**15**) react with malononitrile to afford methylenemalononitrile derivatives (**13**,**16**), **15** doesn't react with other active methylene compounds, such as benzoylacetonitrile (Entry 10), though another thioamide, such as *N*-(2-thienylthiocarbonyl)morpholine (**18**), reacts with malononitrile and benzoylacetonitrile to afford the expected compounds.

Dithiocarbamate derivatives **26**, **27** or *N*,*N*,*N*′,*N*′-tetramethylthiourea (**28**) did not afford any condensation products (Chart 2).

**Nucleophiles.** Other nucleophilic reagents, such as aniline derivatives and ethylene glycol, were examined concerning a reaction of the thiocarbonyl moiety in the presence of silver trifluoroacetate in acetonitrile. Compounds **1** reacted with aniline or ethylene glycol to afford imine (**21**) or acetal (**24**) (Tables 3 and 4). Thio-

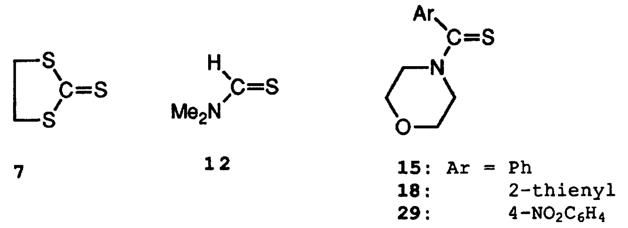


Chart 1.

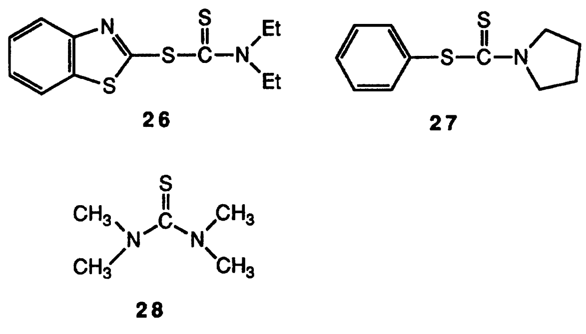
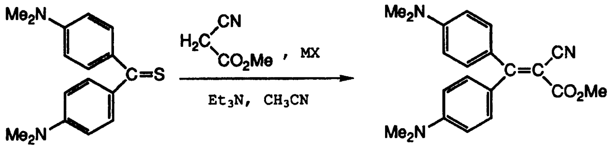


Chart 2.

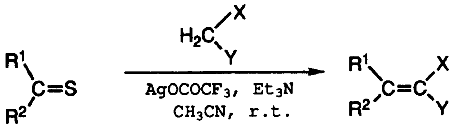
Table 1. Effect of Metal Ions on the Reaction of **1** with Methyl Cyanoacetate[a)]



| Run | MX | Time/h | Temp | Yield/%[b)] |
|---|---|---|---|---|
| 1 | $AgOCOCF_3$(×2.5 equiv) | 1 | Room temp | 87 |
| 2 | $AgOCOCH_3$(×2.5 equiv) | 1 | Reflux | 84 |
| 3 | $Ag_2O$ (×1.25 equiv) | 1 | Reflux | 90 |
| 4 | $Cu_2Cl_2$ (×1.25 equiv) | 1 | Reflux | 40 |
| 5 | $CuCl_2$(×1.25 equiv) | 3 | Reflux | 0 |
| 6 | HgO(×1.25 equiv) | 1 | Reflux | 97 |
| 7 | None | 3 | Reflux | 0 |

a) Molar ratio of **1** : Methyl Cyanoacetate : $Et_3N$ was 1 : 1.2 : 3.6. b) Isolated yields based on **1**.

Table 2. Reaction of Thiocarbonyl Compounds with Active Methylene Compounds in the Presence of Silver Trifluoroacetate[a)]



| Entry | $R^1$ | $R^2$ | X | Y | Product | Yield/%[b)] |
|---|---|---|---|---|---|---|
| 1 | $4\text{-}Me_2NC_6H_4$ | $4\text{-}Me_2NC_6H_4$ | CN | CN | **3** | 81 |
| 2 | $4\text{-}Me_2NC_6H_4$ | $4\text{-}Me_2NC_6H_4$ | CN | COPh | **4** | 88 |
| 3 | $4\text{-}Me_2NC_6H_4$ | $4\text{-}Me_2NC_6H_4$ | COOEt | COPh | **5** | 80 |
| 4 | $4\text{-}Me_2NC_6H_4$ | $4\text{-}Me_2NC_6H_4$ | $NO_2$ | H | **6** | 67 |
| 5 | $SCH_2CH_2S$ | | CN | CN | **8** | 62 |
| 6 | $SCH_2CH_2S$ | | CN | COOMe | **9** | 56 |
| 7 | $SCH_2CH_2S$ | | CN | COPh | **10** | 74 |
| 8 | $SCH_2CH_2S$ | | $NO_2$ | H | **11** | 68 |
| 9 | $Me_2N$ | H | CN | CN | **13** | 48 |
| 10 | $Me_2N$ | H | CN | COPh | **14** | 57[c)] |
| 11 | Ph | Morpholino | CN | CN | **16** | 46 |
| 12 | Ph | Morpholino | CN | COPh | **17** | 0 |
| 13 | 2-Thienyl | Morpholino | CN | CN | **19** | 62 |
| 14 | 2-Thienyl | Morpholino | CN | COPh | **20** | 35[c)] |

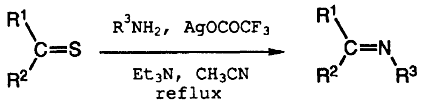
a) Molar ratio of thiocarbonyl compounds : active methylene compounds : $AgOCOCF_3$ : $Et_3N$ was 1 : 1.2 : 2.5 : 3.6. b) Isolated yields based on thiocarbonyl compounds. c) *E*,*Z*-Configuration was not determined.

amide (**15**,**29**) also reacted with aniline and ethylene glycol to afford amidine (**23**) and amide acetal (**25**) derivatives.

**Reaction Mechanism.** It is considered that these reactions proceed according to the pathway shown in Scheme 1. $Ag^+$ attacks the sulfur atom of the thiocarbonyl moiety to generate a cationic intermediate (**A**); a nucleophile then attacks the activated thiocarbonyl carbon to form a C–C linkage (**B**). A sulfur atom is removed as $Ag_2S$ due to an excess of silver(I) ions to afford the product. There are two main factors which control these reactions. The first is the character of a substituent on the thiocarbonyl moiety. It is considered that an electron-donating substituent stabilizes the cationic intermediate (**A**), so that its reactivity toward the nucleophile is weakened. The other factor is the nucleophilicity of active methylene compounds. Malononitrile has a strong nucleophilicity among the active methylene compounds used in this work, and gave the expected product in a reaction with **15**. Benzoylacetonitrile or other active methylene compounds could not react with **15**. The structure of the thiocarbonyl moiety also has some effects on the reaction. When the reaction of **15** is compared with that of **18**, any nucleophilic attacks of active methylene compounds must occur more smoothly, due to a chelate formation (Fig. 1).

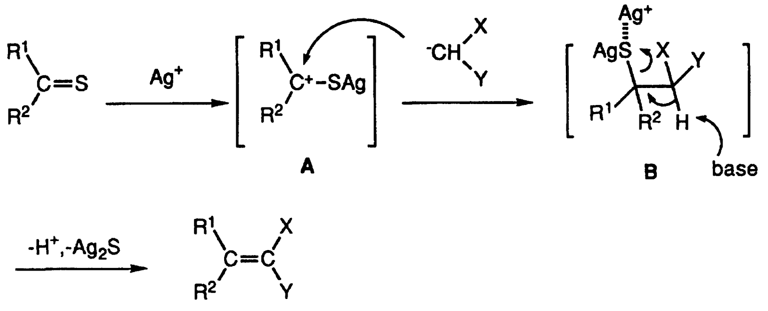
In conclusion, silver ion-mediated desulfurization-condensation of some thiocarbonyl compounds with several nucleophiles can be used as C=C, C=N or acetal bond formation reactions under a mild basic condition.

Table 3. Reaction of Thiocarbonyl Compounds with Aniline Derivatives in the Presence of Silver Trifluoroacetate[a)]



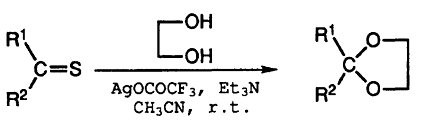
| Entry | R$^1$ | R$^2$ | R$^3$ | Product | Yield/%[b)] |
|---|---|---|---|---|---|
| 1 | 4-Me$_2$NC$_6$H$_4$ | 4-Me$_2$NC$_6$H$_4$ | Ph | **21** | 55 |
| 2 | SCH$_2$CH$_2$S | | 4-ClC$_6$H$_4$ | **22** | 45 |
| 3 | Ph | Morpholino | 4-ClC$_6$H$_4$ | **23** | 50[c)] |

a) Molar ratio of thiocarbonyl compounds : anilines : AgOCOCF$_3$ : Et$_3$N was 1 : 1.2 : 2.5 : 3.6. b) Isolated yields based on thiocarbonyl compounds. c) *E*, *Z*-Configuration was not determined.



Scheme 1.

Table 4. Reaction of Thiocarbonyl Compounds with Ethylene Glycol in the Presence of Silver Trifluoroacetate[a)]



| Entry | R$^1$ | R$^2$ | Product | Yield/%[b)] |
|---|---|---|---|---|
| 1 | 4-Me$_2$NC$_6$H$_4$ | 4-Me$_2$NC$_6$H$_4$ | **24** | 69 |
| 2 | 4-NO$_2$C$_6$H$_4$ | Morpholino | **25** | 77 |

a) Molar ratio of thiocarbonyl compounds : ethylene glycol : AgOCOCF$_3$ : Et$_3$N was 1 : 1.2 : 2.5 : 3.6. b) Isolated yields based on thiocarbonyl compounds.

Most of these reactions proceeded at room temperature. Although these bond formations are usually achieved in acidic media from carbonyl compounds, such as ketones or aldehydes, it is difficult to obtaine these condensation products from diaryl ketones, amides or dithiocarbonates with the nucleophiles. On the other hand, thiocarbonyl compounds it was necessary to activate by alkylation.[17,18)] This procedure therefore provides a useful and convenient synthetic method to build such structures.

## Experimental

All of the melting points were uncorrected. $^1$H and $^{13}$C NMR spectra were recorded on a Bruker AC-200 spectrometer using TMS as an internal standard. The mass spectra were taken on a Shimadzu (GCMS-QP2000A) spectrometer. IR spectra were measured on a JASCO (FT-IR 5300) spectrometer using KBr disks.

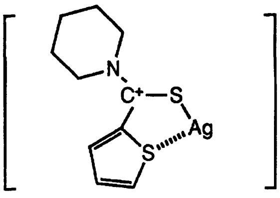


Fig. 1.

**Typical Procedure for the Reaction of the Thiocarbonyl Compounds and Nucleophiles.** A typical experimental procedure is described for the reaction of **1** with methyl cyanoacetate in the presence of AgOCOCF$_3$. To a solution of **1** (1 mmol), methyl cyanoacetate (1.2 mmol) and triethylamine (3.6 mmol) in acetonitrile (3 ml) were added successively AgOCOCF$_3$ (2.5 mmol) in acetonitrile (3 ml) over 5 min. The reaction mixture was continuously stirred for an additional 1 h at room temperature. After evaporation of acetonitrile and triethylamine under reduced pressure, NaCl aq and ethyl acetate were added to the residue. After removing Ag$_2$S and AgCl by suction, an organic layer was separated, washed with NaCl aq, dried by MgSO$_4$, and the solvent was evaporated under reduced pressure. The obtained crude product was purified by recrystallization from MeOH to afford analytically pure methyl 3,3-bis[4-(dimethylamino)phenyl]-2-cyano-2-propenoate (86% yield).

The following spectroscopic data were observed for the compounds obtained by the reaction of some thiocarbonyl compounds with nucleophiles according to the above-mentioned manner.

**Methyl 3,3-Bis[4-(dimethylamino)phenyl]-2-cya-**

**no-2-propenoate (2):** Yellow needles; mp 186—187 °C (EtOH); $^{1}$H NMR ($CDCl_3$) $\delta$=3.05 (12H, s), 3.74 (3H, s), 6.6—6.7 (4H, m), 7.0—7.1 (2H, m), 7.3—7.4 (2H, m); $^{13}$C NMR ($CDCl_3$) $\delta$=39.84, 51.98, 92.36, 110.30, 110.56, 120.32, 125.56, 133.10, 133.62, 152.18, 152.61; MS $m/z$ 350 (M+1); IR $\nu$ 2206, 1700 cm$^{-1}$. Found: C, 72.41; H, 6.64; N, 11.91%. Calcd for $C_{21}H_{23}N_3O_2$: C, 72.18; H, 6.63; N, 12.63%.

**Bis[4-(dimethylamino)phenyl]methylenemalononitrile (3):** Purple needles; mp 253—255 °C ($CH_3CN$) (lit,[19] 244—245 °C).

**2-Benzoyl-3,3-bis[4-(dimethylamino)phenyl]-2-propenenitrile (4):** Yellow needles; mp 198—198.5 °C (EtOH); $^{1}$H NMR ($CDCl_3$) $\delta$=2.9 (6H, s), 3.1 (6H, s), 6.3—6.4 (2H, m), 6.6—6.7 (2H, s), 6.9—7.0 (2H, s), 7.2—7.4 (3H, m), 7.4—7.5 (2H, m), 7.7—7.8 (2H, m); $^{13}$C NMR ($CDCl_3$) $\delta$=39.98, 40.10, 101.53, 110.98, 120.80, 125.32, 126.16, 128.04, 129.27, 132.22, 133.41, 133.56, 137.54, 152.10, 152.60, 168.94, 192.51; MS $m/z$ 396 (M+1); IR $\nu$ 2199, 1714 cm$^{-1}$. Found: C, 78.81; H, 6.17; N, 10.42%. Calcd for $C_{26}H_{25}N_3O$: C, 78.96; H, 6.37; N, 10.62%.

**Ethyl 2-Benzoyl-3,3-bis[4-(dimethylamino)phenyl]-2-propenoate (5):** Yellow needles; mp 153—155 °C (MeOH); $^{1}$H NMR ($CDCl_3$) $\delta$=0.98 (3H, t, $J$=7 Hz), 2.86 (6H, s), 3.01 (6H, s), 4.04 (2H, q, $J$=7 Hz), 6.3—6.4 (2H, m), 6.6—6.7 (2H, m), 6.9—7.0 (2H, m), 7.1—7.4 (5H, m), 7.8—7.9 (2H, m); $^{13}$C NMR ($CDCl_3$) $\delta$=13.83, 40.01, 40.22, 60.55, 110.98, 111.13, 126.16, 127.76, 128.03, 128.23, 129.05, 131.76, 132.00, 132.64, 138.60, 151.14, 151.29, 158.40, 168.56, 199.89; MS $m/z$ 443 (M+1); IR $\nu$ 1716, 1639 cm$^{-1}$. Found: C, 75.65; H, 6.74; N, 6.29%. Calcd for $C_{28}H_{30}N_2O_3$; C, 75.99; H, 6.83; N, 6.33%.

**1,1-Bis[4-(dimethylamino)phenyl]-2-nitroethylene (6):** Red needles; mp 179.5—181 °C (EtOH); $^{1}$H NMR ($CDCl_3$) $\delta$=3.02 (6H, s), 3.03 (6H, s), 6.6—6.7 (4H, m), 7.1—7.3 (4H, m), 7.34 (1H, s); $^{13}$C NMR ($CDCl_3$) $\delta$=40.09, 40.14, 110.99, 111.47, 122.79, 125.01, 129.01, 131.48, 131.91, 151.25, 152.25, 153.67; MS $m/z$ 312 (M+1); IR $\nu$ 1513, 1352 cm$^{-1}$. Found: C, 69.57; H, 6.77; N, 13.38%. Calcd for $C_{18}H_{21}N_3O_2$: C, 19.43; H, 6.80; N, 13.49%.

**2-Dicyanomethylene-1,3-dithiolane (8):** Colorless prisms; mp 199—200 °C (benzene) (lit,[20] 199—200 °C).

**Methyl (1,3-Dithiolane-2-ylidene)cyanoacetate (9):** Colorless prisms; mp 138—139 °C (toluene) (lit,[20] 145— 146 °C).

**2-(Benzoylcyanomethylene)-1,3-dithiolane (10):** Colorless needles; mp 174— 176 °C (EtOH): $^{1}$H NMR ($CDCl_3$) $\delta$=3.5—3.7 (4H, m), 7.4—7.6 (3H, m), 7.8—8.0 (2H, m); $^{13}$C NMR ($CDCl_3$) $\delta$=36.41, 40.28, 92.24, 105.05, 128.26, 128.44, 136.74, 186.07, 187.54; MS $m/z$ 247 (M$^{+}$); IR $\nu$ 2197, 1622 cm$^{-1}$. Found: C, 58.41; H, 3.48; N, 5.69; S, 26.16%. Calcd for $C_{12}H_9NOS_2$: C, 58.27; H, 3.67; N, 5.66; S, 25.93%.

**2-Nitromethylene-1,3-dithiolane (11):** Colorless; mp 105—106 °C (EtOH) (lit,[18] 112 °C).

**2-(Dimethylaminomethylene)malononitrile (13):** Colorless needles; mp 83—84 °C (benzene)(lit,[16] 82—83 °C).

**2-Benzoyl-3-dimethylamino-2-propenenitrile (14):** Colorless needles; mp 115.5 °C (AcOEt–hexane); $^{1}$H NMR ($CDCl_3$) $\delta$=3.28 (3H, s), 3.48 (3H, s), 7.4—7.5 (3H, m), 7.7—7.8 (2H, m), 7.95 (1H, s); $^{13}$C NMR ($CDCl_3$) $\delta$=38.89, 48.16, 79.56, 120.17, 127.98, 128.11, 131.42, 138.38, 159.28, 190.20; MS $m/z$ 200 (M$^{+}$) IR $\nu$ 2191, 1645 cm$^{-1}$. Found: C, 71.86; H, 5.80; N, 13.82%. Calcd for $C_{12}H_{12}N_2O$: C, 71.98; H, 6.04; N, 13.99%.

**($\alpha$-Morpholinobenzylidene)malononitrile (16):** Colorless needles; mp 177—178 °C ($CH_3CN$) (lit,[18] 178 °C).

**[Morpholino(2-thyenyl)methylene]malononitrile (19):** Colorless; mp 150—150.5 °C (EtOH) (lit,[17] 145 °C).

**2-Benzoyl-3-morpholino-3-(2-thienyl)-2-propenenitrile (20):** Pale yellow needles; mp 177—177.5 °C (AcOEt–hexane); $^{1}$H NMR ($CDCl_3$) $\delta$=3.48 (4H, br), 3.8—3.9 (4H, m), 7.1—7.2 (1H, m), 7.3—7.5 (4H, m), 7.65 (1H, d, $J$=5 Hz), 7.7—7.9 (2H, m); $^{13}$C NMR ($CDCl_3$) $\delta$=53.17, 66.96, 85.39, 128.21, 128.55, 128.66, 132.07, 132.76, 134.33, 134.94, 138.63, 164.74; MS $m/z$ 324 (M$^{+}$) IR $\nu$ 2195, 1601 cm$^{-1}$, Found: C, 66.68; H, 4.90; N, 8.67; S, 9.93%. Calcd for $C_{18}H_{16}N_2O_2S$: C, 66.64; H, 4.97; N, 8.64; S, 9.88%.

***N*-{Bis[4-(dimethylamino)phenyl]methylene}aniline (21):** Pale yellow needles; mp 173—174 °C (AcOEt–hexane); $^{1}$H NMR ($CDCl_3$) $\delta$=2.92 (6H, s), 3.01 (6H, s), 6.4—6.5 (2H, m), 6.6—6.7 (2H, m), 6.7—6.8 (2H, m), 6.8—6.9 (1H, m), 7.0—7.1 (2H, m), 7.1—7.2 (2H, m), 7.6—7.7 (2H,m); $^{13}$C NMR ($CDCl_3$) $\delta$=40.01, 40.15, 110.61, 110.98, 121.48, 121.87, 123.81, 128.30, 128.42, 130.96, 131.52, 149.90, 151.78, 152.53, 168.00. Found: C, 80.70; H, 7.32: N, 12.24%. Calcd for $C_{23}H_{25}N_3$: C, 80.43; H, 7.34; N, 12.23%

**4-Chloro-*N*-(1,3-dithiolan-2-ylidene)aniline (22):** Colorless needles; mp 89—90°C (*i*-$Pr_2O$–hexane); $^{1}$H NMR ($CDCl_3$) $\delta$=3.4—3.7 (4H, m), 6.8—7.0 (2H, m), 7.2—7.4 (2H, m); $^{13}$C NMR ($CDCl_3$) $\delta$=35.25, 38.02, 121.65, 129.16, 129.82, 150.68, 172.48. Found: C, 46.85; H, 3.51; N, 6.09; S, 28.16%. Calcd for $C_9H_8ClNS_2$: C, 47.05; H, 3.51; N, 6.09; S, 27.91%.

**4-Chloro-*N*-($\alpha$-morpholinobenzylidene)aniline (23):** Colorless prisms; mp 111.5—112.5 °C (*i*-$Pr_2O$); $^{1}$H NMR ($CDCl_3$) $\delta$=3.40 (4H, br), 3.7—3.8 (4H, m), 6.4—6.5 (2H, m), 6.9—7.0 (2H, m), 7.0—7.1 (2H, m), 7.2—7.3 (3H, m); $^{13}$C NMR ($CDCl_3$) $\delta$=46.49, 66.71, 123.90, 126.26, 128.15, 128.43, 128.93, 132.62, 149.47, 160.94. Found: C, 67.87; H, 5.63; N, 9.23%. Calcd for $C_{17}H_{17}ClN_2O$: C, 67.88; H, 5.70; N, 9.31%.

**2,2-Bis[4-(dimethylamino)phenyl]-1,3-dioxolane (24):** Colorless prisms; mp 128—128.5 °C (*i*-$Pr_2O$); $^{1}$H NMR ($CDCl_3$) $\delta$=2.92 (12H, s), 4.02 (4H, s), 6.6—6.7 (4H, m), 7.3—7.4 (4H, m); $^{13}$C NMR ($CDCl_3$) $\delta$=40.56, 64.59, 110.05, 111.84, 127.38, 130.13, 150.22. Found: C, 72.85; H, 7.56; N, 8.85%. Calcd for $C_{19}H_{24}N_2O_2$: C, 73.05; H, 7.74; N, 8.97%.

**2-Morpholino-2-(4-nitrophenyl)-1,3-dioxolane (25)** Colorless prisms; mp 141— 142 °C (AcOEt– hexane); $^{1}$H NNR ($CDCl_3$) $\delta$=2.5—2.7 (4H, m), 3.6—3.7 (4H, m), 3.8—4.0 (2H, m), 4.0—4.2 (2H, m), 7.6—7.8 (2H, m), 8.1—8.3 (2H, m); $^{13}$C NMR ($CDCl_3$) $\delta$=45.55, 65.11, 66.74, 116.34, 123.09, 127.87, 146.14, 147.83. Found:C 55.68; H, 5.49; N, 9.75%. Calcd for $C_{13}H_{16}N_2O_5$: C, 55.71; H, 5.75; N, 9.99%.

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