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Studies on the Synthesis and Properties of Open-Chain and Macrocyclic Crown Compounds

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Abstract. Eight new open-chain crown compounds (**1–8**) and two new macrocyclic crown ethers (**9** and **10**) were synthesized from 1,5-bis(2-aminophenoxy)-3-oxapentane (**15**) and 1,8-bis(2-aminophenoxy)-3,6-dioxaoctane (**16**) which were prepared by the catalytic hydrogenation of the corresponding bis(2-nitrophenoxy) derivatives **13** and **14**. The fluorescent property investigation of two open-chain crown ethers (**7** and **8**) indicated that they exhibit complexing effects on Ag^+ and Mn^{2+} cations and can be used as fluorescent reagents for the microanalysis of the metal cations. The pyrolysis kinetics measurement for compounds **5** and **6** in nitrogen and air was carried out, and their reaction orders and activation energy were obtained. They are one-step and two-step pyrolysis reactions in nitrogen and air, respectively.

Key words: Open-chain crown compounds, macrocyclic crown ethers, fluorescent properties, pyrolysis kinetics.

1. Introduction

In the last two decades extensive studies have been carried out on various crown compounds as superior complexing agents for metal cations, anions and neutral molecules [1]. Vögtle and coworkers [2–8] reported the synthesis and complexation of a series of open-chain (noncyclic) crown ethers, and first suggested the 'end group concept' or 'terminal group concept' [4]. The open-chain crown compounds with end groups readily form stable crystalline complexes with various metal cations [3–6]. The complexation and decomplexation of open-chain crown compounds are generally faster than the corresponding macrocyclic polyethers. A series of open-chain crown ethers with carboxylic end groups were synthesized, and were investigated as cardiac stimulants because of their efficient calcium binding abilities [9]. A number of other open-chain ligands with reactive end groups were synthesized [10–12] and were used for the preparation of various macrocyclic crown compounds [13–21]. Macrocyclic crown compounds have high selectivities and stabilities for the complexation of certain ions, and have been widely used

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in industry and various research areas. A variety of macrocyclic, macropolycyclic polyethers and cryptands has been synthesized in a search for desired synthetic host molecules [22].

In the present paper, we describe the synthesis of eight new open-chain crown ethers (**1–8**), and two new amide and urea type macrocyclic crown compounds (**9** and **10**) (Figure 1). The fluorescent properties and pyrolysis kinetics results for some of these ligands are also reported.

2. Experimental

Proton NMR spectra were obtained on a Bruker Ac-80 spectrometer using $CDCl_3$ or DMSO-d_6 as the solvents with TMS as the internal standard. IR spectra were recorded on a Shimadzu IR-435 spectrometer. EI mass spectra were obtained on a Finnigan-MAT spectrometer. Elemental analyses were determined with a Carlo-Erba 1106 instrument. 1,5-Dichloro-3-oxapentane (**11**) and 1,8-dichloro-3,6-dioxaoctane (**12**) were prepared as reported [23] from di- and triethylene glycols, respectively. 1,5-Bis(2-nitrophenoxy)-3-oxapentane (**13**) and 1,8-bis(2-nitrophenoxy)-3,6-dioxaoctane (**14**) were prepared by the reaction of 2-nitrophenol with glycol dichlorides **11** and **12** via a published procedure [14, 15]. 10% Pd/C [24] and 20% $Pd(OH)_2/C$ [25] were prepared according to literature methods.

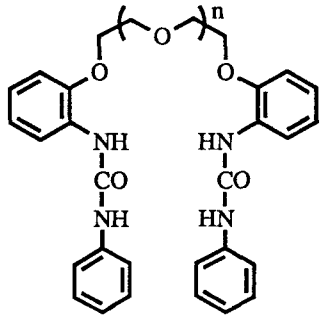
2.1. Preparation of 1,5-bis(2-phenylureidophenoxy)-3-oxapentane (1)

A mixture containing 1,5-bis(2-nitrophenoxy)-3-oxapentane (**13**) (2.0 g, 5.7 mmol), 0.2 g of 10% Pd/C and 40 mL of dioxane was shaken under 4 kg/cm^2 of hydrogen atmosphere at 70 °C for 4 h. The cooled reaction mixture was filtered and the filtrate was added dropwise to a stirred mixture of phenyl isocyanate (1.17 g, 9.8 mmol) in 10 mL of dioxane under nitrogen atmosphere at r.t. The resulting mixture was stirred at r.t. for 1 h and at refluxing temperature for 0.5 h. The cooled reaction mixture was filtered and the filtrate was evaporated under reduced pressure. Recrystallization of the white solid from acetonitrile gave 2.4 g (79%) white crystalline product **1**, m.p. 219–220 °C; ^{1}H NMR: (δ) 3.87–4.30 (m, 8 H), 6.78–8.19 (m, 18 H), 8.00 (b, 2H), 9.22 (b, 2H); IR (cm^{-1}): 3290, 3010, 1653, 1588, 1559, 1494, 1251, 1110, 744.

Anal. Calcd. for $C_{30}H_{30}O_5N_4$: C, 68.43; H, 5.74; N, 10.64; mol. wt. 526.59. *Found*: C, 68.43; H, 5.75; N, 10.82.

2.2. Preparation of 1,8-bis(2-phenylureidophenoxy)-3,6-dioxaoctane (2)

Open-chain crown compound **2** was prepared as above for **1** from 2.0 g (5.1 mmol) of 1,8-bis(2-nitrophenoxy)-3,6-dioxaoctane (**14**), 0.2 g of 10% Pd/C and 1.03 g (8.6 mmol) of phenyl isocyanate in dioxane. Recrystallization from acetonitrile gave 2.21 g (76%) of **2** as white crystals, m.p. 219–220 °C; ^{1}H NMR: (δ) 3.65–



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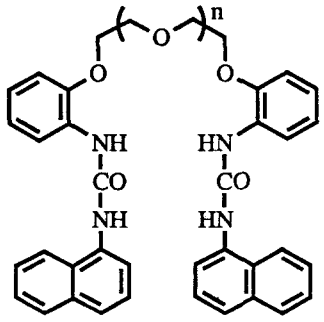

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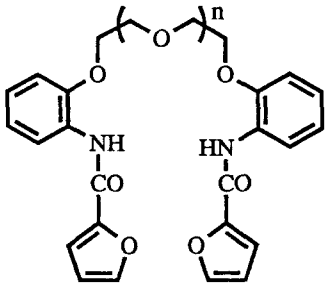

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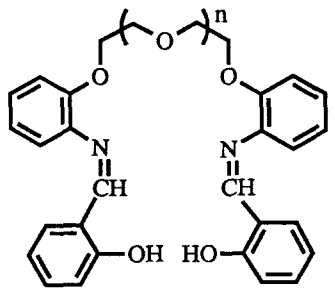

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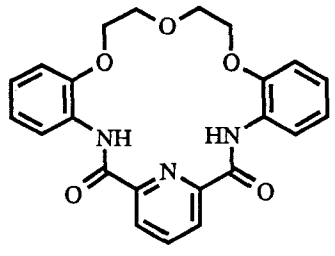

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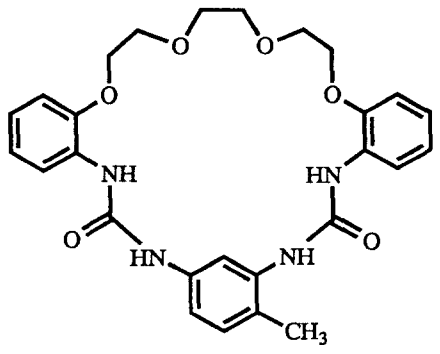

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Fig. 1. Open-chain and macrocyclic crown compounds **1–10**.

4.23 (m, 12 H), 6.79–8.22 (m, 18 H), 7.99 (b, 2H), 9.25 (b, 2H); IR (cm^{-1}): 3285, 3010, 1653, 1587, 1561, 1492, 1264, 1133, 746.

Anal. Calcd. for $C_{32}H_{34}O_6N_4$: C, 67.36; H, 6.00; N, 9.82; mol. wt. 570.64. *Found*: C, 67.30; H, 6.00; N, 9.82.

2.3. PREPARATION OF 1,5-BIS[2-(1-NAPHTHYLUREIDO)PHENOXY]-3-OXAPETANE (3)

Open-chain crown compound **3** was prepared as above for **1** from 2.0 g (5.7 mmol) of **13**, 0.2 g of 10% Pd/C and 1.47 g (8.7 mmol) of 1-naphthyl isocyanate in dioxane. Recrystallization from DMF/diethyl ether (1/4) gave 2.28 g (64%) of **3** as a white solid, m.p. 227.5–228.5 °C; ^{1}H NMR: (δ) 3.90–4.33 (m, 8 H), 6.79–8.24 (m, 22 H), 8.47 (b, 2H), 9.25 (b, 2H); IR (cm^{-1}): 3300, 3010, 1644, 1613, 1546, 1459, 1260, 1139, 806, 741.

Anal. Calcd. for $C_{38}H_{34}O_5N_4$: C, 72.83; H, 5.47; N, 8.94; mol. wt. 626.71. *Found*: C, 72.82; H, 5.49; N, 9.00.

2.4. PREPARATION OF 1,8-BIS-[1-NAPHTHYLUREIDO)PHENOXY]-3,6-DIOXAOCTANE (4)

Open-chain crown compound **4** was prepared as above for **1** from 2.0 g (5.1 mmol) of **14**, 0.2 g of 10% Pd/C and 1.66 g (9.8 mmol) of 1-naphthyl isocyanate in dioxane. Recrystallization from DMF/diethyl ether (1/4) gave 2.12 g (62%) of **4** as a white solid, m.p. 213.5–214.5 °C; ^{1}H NMR: (δ) 3.63–4.19 (m, 12 H), 6.81–8.18 (m, 22 H), 8.54 (b, 2H), 9.39 (b, 2H); IR (cm^{-1}): 3274, 3010, 1643, 1599, 1561, 1459, 1260, 1119, 822, 742.

Anal. Calcd. for $C_{40}H_{38}O_6N_4$: C, 71.63; H, 5.71; N, 8.35; mol. wt. 670.76. *Found*: C, 71.45; H, 5.71; N, 8.57.

2.5. PREPARATION OF 1,5-BIS[2-(2-FUROYLAMINO)PHENOXY]-3-OXAPENTANE (5)

0.2 g of 20% $Pd(OH)_2$/C in 10 mL of anhydrous ethanol was shaken under 4 Kg/cm^2 of hydrogen atmosphere at r.t. for 30 min. A solution of 1,5-bis(2-nitrophenoxy)-3-oxapentane (**13**) (2.0 g, 5.7 mmol) in anhydrous ethanol (40 mL) was added. The resulting mixture was shaken under 4 kg/cm^2 of hydrogen atmosphere at 65 °C for 3 h. The cooled reaction mixture was filtered and the solvent was evaporated under reduced pressure. A solution of the above residue (**15**) in 100 mL of dry benzene and a solution of 2-furoyl chloride (1.27 g, 9.7 mmol) in 100 mL of dry benzene were simultaneously dropped into 200 mL of stirred dry benzene under nitrogen atmosphere for 4 h. The resulting mixture was stirred at r.t. for 72 h. The mixture was filtered and the solvent was evaporated under reduced pressure. Purification of the solid residue by chromatography on neutral alumina using benzene/acetone : 4/1 as the eluant gave 1.19 g (44%) of **5** as a white solid. Recrystallization of the white solid from benzene/acetone/chloroform (3/1/1) gave white crystals, m.p. 147.5–148.5 °C; ^{1}H NMR: (δ) 3.93–4.32 (m, 8 H), 6.44–7.36 (m, 12 H), 8.41–8.52 (m, 2 H), 8.84 (b, 2H): IR (cm^{-1}): 3382, 3124, 1681, 1600, 1530, 1480, 1251, 1123, 759; MS: m/z 476 (M^+).

Anal. Calcd. for $C_{26}H_{24}O_7N_2$: C, 65.54; H, 5.08; N, 5.88; mol. wt. 476.48. *Found*: C, 65.48; H, 5.09; N, 5.78.

2.6. PREPARATION OF 1,8-BIS[2-(2-FUROYLAMINO)PHENOXY]-3,6-DIOXAOCTANE (**6**)

Open-chain crown compound **6** was prepared as above for **5** from 0.2 g of 20% $Pd(OH)_2/C$, 2.0 g (5.1 mmol) of **14** and 1.13 g (8.6 mmol) of 2-furoyl chloride in dry benzene. Recrystallization of the white solid from dichloromethane gave 1.06 g (40%) of **6** as white crystals, m.p. 124–125 °C; ^1H NMR: (δ) 3.69–4.23 (m, 12 H), 6.43–7.45 (m, 12 H), 8.39–8.51 (m, 2 H), 8.83 (b, 2H); IR (cm^{-1}): 3461, 3144, 1655, 1601, 1580, 1533, 1461, 1251, 1121, 755; MS: m/z 520 (M^+).

Anal. Calcd. for $C_{28}H_{28}O_8N_2$: C, 64.61; H, 5.42: n, 5.38; mol. wt. 520.53. *Found*: C, 64.78; H, 5.48; N, 5.27.

2.7. PREPARATION OF 1,5-BIS[2-(2-HYDROXYPHENYLIDENE IMINO)PHENOXY]-3-OXAPENTANE (**7**)

The reduction of **13** (2.0 g, 5.7 mmol) was carried out as above for **5** from 0.2 g of 20% $Pd(OH)_2/C$. After filtration of the catalyst, the filtrate, a solution of **15** in ethanol, was added dropwise to a stirred solution of 2-hydroxy benzaldehyde (1.20 g, 9.8 mmol) in 40 mL of anhydrous ethanol at r.t. under nitrogen atmosphere. The resulting mixture was stirred for 12 h r.t. and was set aside. The yellow crystals were collected and washed with ethanol. 1.2 g (42%) of **7** as yellow needles was obtained, m.p. 64–65 °C; ^1H NMR: (δ) 3.99–4.22 (m, 8 H), 6.87–7.34 (m, 16 H), 8.69 (s, 2H), 13.60 (b, 2H); IR (cm^{-1}): 3168, 3010, 1617, 1586, 1558, 1486, 1251, 1124, 736; MS: m/z 496 (M^+).

Anal. Calcd. for $C_{30}H_{28}O_5N_2$: C, 72.56; H, 5.68; N, 5.64; mol. wt. 496.56. *Found*: C, 72.27; H, 5.67; N, 5.45.

2.8. PREPARATION OF 1,8-BIS[2-(2-HYDROXYPHENYLIDENE IMINO)PHENOXY]-3,6-DIOXAOCTANE (**8**)

Open-chain crown compound **8** was prepared as above for **7** from 0.2 g of 20% $Pd(OH)_2/C$, 2.0 g (5.1 mmol) of **14** and 1.06 g (8.6 mmol) of 2-hydroxy benzaldehyde in anhydrous ethanol. After evaporation of the solvent, the residue was purified by chromatography on neutral alumina using chloroform/diethyl ether: 1/1 as the eluant. Recrystallization from anhydrous ethanol/chloroform (4/1) gave 1.4 g (51%) of **8** as yellow crystals, m.p. 94.5–95.5 °C; ^1H NMR: (δ) 3.90–4.20 (m, 12H), 6.87–7.42 (m, 16H), 8.71 (s, 2H), 13.60 (b, 2H); IR (cm^{-1}): 3134, 3010, 1615, 1588, 1564, 1486, 1262, 1148, 755; MS: m/e 540 (M^+).

Anal. Calcd. for $C_{32}H_{32}O_6N_2$: C, 71.09; H, 5.96; N, 5.18; mol. wt. 540.61. *Found*: C, 70.79; H, 5.93; N, 5.19.

2.9. Preparation of macrocyclic crown compound (**9**)

The reduction of **13** (2.0 g, 5.7 mmol) was carried out as above for **5**. A solution of the diamine obtained **15** in 100 mL of dry benzene and a solution of 2,6-pyridine dicarbonyl dichloride (1.0 g, 4.9 mmol) in 100 mL of dry benzene were simultaneously dropped into 200 mL of stirred dry benzene under nitrogen atmosphere for 3 h. The resulting mixture was stirred at r.t. for 70 h. The solid was filtered and the solvent was evaporated under reduced pressure. The residue was purified by chromatography on neutral alumina using chloroform/acetone: 85/15 as the eluant. Recrystallization from chloroform/benzene (1/1) gave 0.5 g (24.4%) of **9** as white needles, m.p. 247.5–248.5 °C; ^{1}H NMR: (δ) 3.84–4.31 (m, 8 H), 6.84–8.58 (m, 11 H), 9.69 (b, 2H); IR (cm^{-1}): 3396, 3050, 1681, 1598, 1528, 1454, 1256, 1150, 750.

Anal. Calcd. for $C_{32}H_{21}O_5N_3$: C, 65.86; H, 5.05; N, 10.02; mol. wt. 419.43. *Found*: C, 65.64; H, 5.12; N, 9.96.

2.10. Preparation of macrocyclic crown compound (**10**)

2.0 g (5.1 mmol) of dinitro derivative **14** was reduced as above for **5**. The obtained diamine **16** in 10 mL of dioxane was added dropwise to a stirred solution of tolylene 2,4-diisocyanate (0.76 g, 4.4 mmol) in 10 mL of dioxane under nitrogen atmosphere. The resulting mixture was stirred at r.t. for 15 min and at reflux temperature for 1 h. After the reaction mixture was cooled, the white solid was filtered and recrystallized from acetone to give 2.0 g (90%) of **10** as white crystals, m.p. 196–197 °C; ^{1}H NMR: (δ) 2.14 (s, 3H), 3.54–4.14 (m, 12H), 6.80– 8.70 (m, 11 H), 8.40 (b, 2H), 9.40 (b, 2H); IR (cm^{-1}): 3267, 3010, 1645, 1599, 1544, 1458, 1254, 1115, 830, 740.

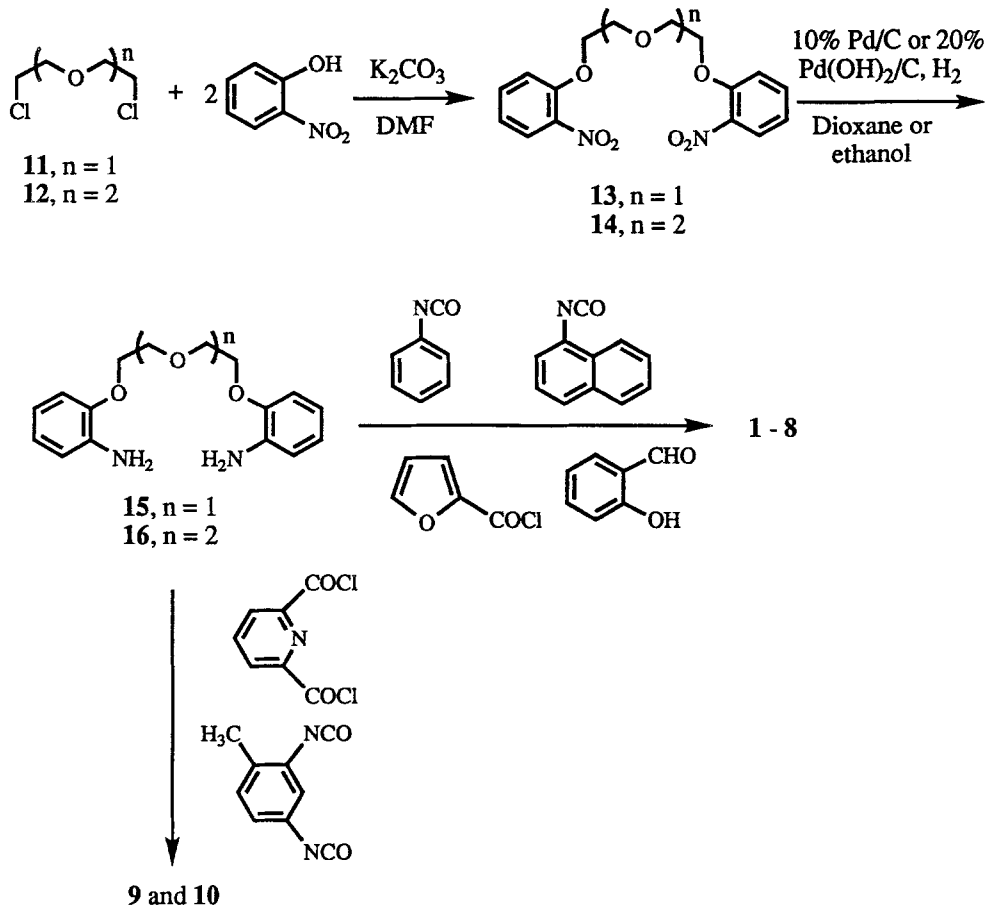
Anal. Calcd. for $C_{27}H_{30}O_6N_4$: C, 64.02; H, 5.97; N, 11.06; mol. wt. 506.55. *Found*: C, 64.23; H, 6.18; N, 11.26.

2.11. Measurements [26, 27]

The pyrolysis kinetics measurements were performed by thermogravimetry (TG) and differential thermal analysis (DTA) on a Rigaku Thermoanalyzer with α-Al_2O_3 as a reference. The reaction orders and activation energy were obtained by Coats-Redefern and Freeman-Garroll methods. UV and FL spectra were measured on HITACHI U-2000 and F-3010 spectrometers, respectively. A PHS-2 type pH meter was used to measure pH values.

3. Results and Discussion

The chlorination of di- and triethylene glycols with thionyl chloride gave the corresponding dichlorides **11** and **12** [23]. **11** and **12** were treated with excess amount of



Scheme 1. Synthesis of open-chain and macrocyclic crown compounds **1–10**.

2-nitrophenol in DMF using K_2CO_3 as a base to give 1,5-bis(2-nitrophenoxy)-3-oxapentane (**13**) and 1,8-bis(2-nitrophenoxy)-3,6-dioxaoctane (**14**) [14, 15] (Scheme 1). Several methods were used for the reduction of dinitro derivatives **13** and **14**. Hydrazine palladium-carbon [13] and tin(II) chloride dihydrate in concentrated hydrochloric acid [14] were used for the reduction of **13** to diamine **15** in low yield. **13** and **14** were reduced by amalgamated aluminium in isopropanol in 45–50% yield [15]. 10% Pd/C and 20% $Pd(OH)_2/C$ catalysts were used in our lab for the hydrogenation of dinitro derivatives **13** and **14** to their corresponding diamines **15** and **16**. This method gave high yields and very clean reaction mixtures. As a matter of fact, after filtration of the catalyst, the products were used for the next step without purification.

Diamine derivatives **15** and **16** were treated with phenyl isocyanate and 1-naphthyl isocyanate to give open-chain crown compounds **1–4** containing aromatic ureido end groups (Scheme 1). The reaction of 2-furoyl chloride with diamines **15**

and **16** gave open-chain crown compounds **5** and **6** with furoyl amino end groups. The condensation of 2-hydroxy benzaldehyde with diamines **15** and **16** gave Schiff-base type open-chain crown compounds **7** and **8**. All of these new open-chain crown compounds **1–8** (Figure 1) were obtained in high yields. Even though a number of open-chain crown compounds with various end groups were reported [2–12], the open chain crown ligands with ureido end groups were first reported. Two new macrocyclic crown compounds **9** and **10** were also synthesized from diamines **15** and **16** (Scheme 1, Figure 1). The cyclization of diamine **15** with 2,6-pyridine dicarbonyl dichloride gave the corresponding macrocyclic crown compound **9**. Diamine **16** was reacted with tolylene 2,4-diisocyanate to produce macrocyclic polyether **10** in a high yield.

There has been increasing interest in some crown compounds as fluorescent reagents for the determination of metal cations [28]. The fluorescent properties of Schiff-base type compounds **7** and **8** were investigated. The excitation and emission wave lengths were determined. The influences of solvent polarity, the concentration of crown compounds and the pH values of the solution on the fluorescence intensity of these compounds were examined. It was found that the optimum excitation and emission wave lengths for compound **7** in EtOH-H_2O (8/2, v/v) with a concentration of 5×10^{-6} M and a pH value of 12 were 381 nm and 494 nm, respectively. Under the same conditions, the optimum excitation and emission wave lengths for compound **8** were 381 nm and 496 nm, respectively. It was also found that the higher the ratio of ethanol in the solution, the stronger fluorescence intensity was observed. The relative fluorescence intensity increases with the increase of pH values of the solution. The complexing abilities of compounds **7** and **8** with Li^+, Na^+, K^+, Ca^{2+}, Sr^{2+}, Ba^{2+}, Mg^{2+}, Cr^{3+}, Mn^{2+}, Fe^{3+}, Co^{3+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Ag^+, Al^{3+}, Cd^{2+}, Bi^{3+}, As^{4+}, Mo^{6+}, Pd^{2+} nitrate salts (NO_3^-) and Rb^+, Cs^+, Y^+, Zr^+, Au^+, Hg^{2+}, Ga^{3+}, Ge^{4+}, Sn^{4+}, Te^{4+}, La^{4+}, Ce^{4+} chlorides (Cl^-) and silicates (SiO_3^{2-}) were studied by the corresponding fluorescent spectra. Compounds **7** and **8** exhibit obvious complexing effects only on Ag^+ and Mn^{2+} cations. These results indicated that compounds **7** and **8** can be used as fluorescent reagents for the micro-analysis of Ag^+ and Mn^{2+} cations by quenching the fluorescence reagent. The bathochromic shifts of UV spectra for compounds **7** and **8** in the presence of Ag^+ and Mn^{2+} cations further confirmed the complexing effect of these compounds on the cations.

The pyrolysis kinetics measurements for compounds **5** and **6** were carried out in nitrogen and air, respectively. The results of the pyrolysis kinetics measurements are listed in Table I. It was found that the mechanism for the pyrolysis in the air is similar to that deduced from the MS data. The first step of the pyrolysis reaction is the breaking of the C-N bond of an amido group, and the formation of fragments with molecular weights of 98 and 95 for **5** and **6**, respectively. These results are consistent with those of their MS. The reaction order and activation energy for the pyrolysis of compound **6** are quite different from those for the pyrolysis of **5**. The differences may be caused by the different number of ethereal oxygen atoms between **5** and **6**. The second step of the reaction is the oxidation

TABLE I. Pyrolysis kinetics and analysis data for compounds **5** and **6**.

	Reaction	Parameter	**5**	**6**
air	first step	r	0.9956	0.9935
		n	0.232	0.623
		E	95.87	170.33
		α	0.7938	0.8181
	second step	r	0.9996	0.9996
		n	0.843	0.917
		E	170.33	181.12
		α	0.2062	0.1818
N_2	first-step reaction	r	0.9990	0.9992
		n	0.387	0.281
		E	130.84	127.29
		α	0.8885	0.8949

r = correlation coefficient; n = reaction order; E = reaction energy (kJ/mol); α = weight loss ratio.

of furoyl fragments and the formation of CO_2 and H_2O at high temperature in the air. The pyrolysis reaction of **5** and **6** in nitrogen exhibited similar activation energy and weight loss ratio. These results indicated that their reaction mechanisms are identical. The molecular weight of the remaining fragment is 54 (butadiene) which was produced by the decomposition of furoyl fragments with the loss of CO_2. When the temperature was higher than 800 °C, the butadiene fragment was polymerized and thus the weight kept constant. When air was introduced into the oven, the weight decreased to zero immediately.

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