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Intramolecular excimer formation and complexing behavior of 1,*n*-bis(naphthalenecarboxy)oxaalkanes as fluorescent chemosensors for calcium and barium ions

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Abstract

,*n*-Bis(1-naphthalenecarboxy)oxaalkanes (1NP*n*N, $n = 1$–6) and 1,*n*-bis(2-naphthalenecarboxy)oxaalkanes (2NP*n*N, $n = 1$–6) as fluorescent sensors by excimer emission (EX) for metal ions were synthesized. We investigated the relationship between the length of the polyether chain and the metal ion recognition of NP*n*N (NP*n*N = 1NP*n*N or 2NP*n*N, $n = 1$–6) in acetonitrile solution, and the influence of the naphthalene substitution position (α-position or β-position) for metal ion recognition. Changes in the fluorescence spectra of NP*n*N ($n = 1$–3) were not observed by the addition of alkali and alkaline earth metal salts. However, the spectra of NP*n*N ($n = 4$–6) were changed by the addition of calcium and barium salts. Especially in NP*n*N ($n = 5$, 6), the changes in the shape and intensity of the fluorescence spectra were great. The values of $\log K$ for Ca^{2+} and Ba^{2+} were 2NP6N > 1NP6N > 2NP5N > 1NP5N. In the cases of same length of the polyether chain, 2NP*n*N is an excellent fluorescent chemosensor for Ca^{2+} and Ba^{2+}, better than 1NP*n*N. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Naphthalene derivatives; Excimer emission; Metal ion recognition; Fluorescent chemosensors

1. Introduction

Metal ions and organic molecular recognition is a subject of considerable interest because of its implications in many fields: biology, medicine, environment, etc. In particular, the detection of metal cations involved in biological processes (sodium, potassium, calcium) has received considerable attention. Our aim is to design fluorescent sensors [1–3] that undergo photophysical changes as marked as possible upon cation binding.

Many kinds of crown ether-type macrocyclic compounds have been used for analytical applications, such as chemical sensors and spectrophotometric analyses. In fluorometry applications, fluorescent reagents, which have two aromatic hydrocarbons at both terminals of a linear polyether as an analogue of a crown ether, have also been used [4–6]. However, there are few studies of the relation between the length of the polyether chain and metal ion recognition. The fluorescent aromatic hydrocarbons in these compounds are usually pyrene or anthracene [7]. The disadvantage of a pyrene or anthracene derivative is its ability to form an intermolecular excimer even at very low concentration of solutions ($10^{-5}\,\mathrm{mol\,dm^{-3}}$) used for spectroscopic studies. It is difficult to distinguish intermolecular or intramolecular excimer emissions (EXs) in low concentrations. However, in naphthalene compounds intermolecular interaction occurs at more than $10^{-1}\,\mathrm{mol\,dm^{-3}}$. Therefore, we have synthesized some naphthalene derivatives as fluorescent sensors for metal ions by intramolecular excimer or exciplex emission [8–11]. In this report, first, we investigated the photophysical and photochemical properties of 1,*n*-bis(1-naphthalenecarboxy)oxaalkanes (1NP*n*N, $n = 1$–6) and 1,*n*-bis(2-naphthalenecarboxy)oxaalkanes (2NP*n*N, $n = 1$–6) before the addition of metal ions. Second, we investigated the relationship between the length of the polyether chain and metal ion recognition of NP*n*N (NP*n*N = 1NP*n*N or 2NP*n*N), and the influence of the naphthalene substitution position (α-position or β-position) for metal ion recognition.

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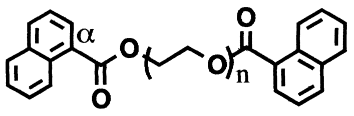
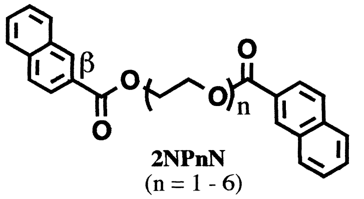
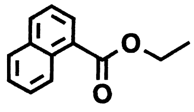


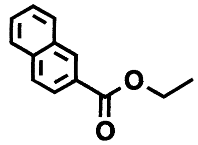
1NPnN
(n = 1 - 6)



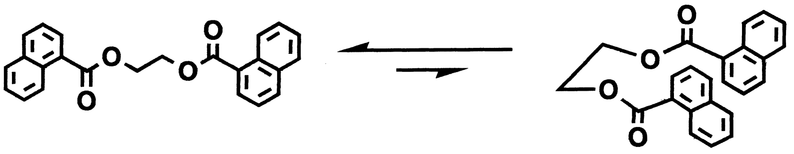
2NPnN
(n = 1 - 6)



1EN

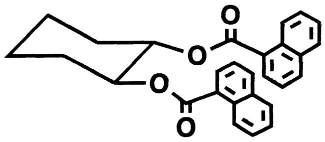


2EN



1NP1N
"extended"

1NP1N
"folded"



cyclo-1NP1N

2. Experimental details

2.1. General

^{1}H and ^{13}C NMR spectra were recorded on a JEOL GX 270 spectrometer with TMS as an internal standard. UV–visible absorption spectra were taken on a Hitachi U-2001 or 228 spectrometer. Fluorescence spectra were taken on a Hitachi F-4500 fluorophotometer.

Measurements of the fluorescence spectra were carried out in an acetonitrile solution of the NP*n*N (1.0×10^{-5} mol dm^{-3} where no intermolecular interaction was found) at room temperature, and alkali or alkaline earth metal salts ($LiClO_4$, $NaClO_4$, $KClO_4$, $Mg(ClO_4)_2$, $Ca(ClO_4)_2$ and $Ba(ClO_4)_2$) were added to the solution. To prevent any nonlinearity of the fluorescence intensity, the isosbestic points (ca. 265 or 295 nm) of the absorption spectra of NP*n*N were chosen as the excitation wavelengths. Fluorescence quantum yields were determined at room temperature relative to those of naphthalene (0.205 in a polar solvent) [12] for solutions of matched absorbance (0.1) at the excitation wavelength.

2.2. Synthesis

1,*n*-Bis(1-naphthalenecarboxy)oxaalkanes (1NP*n*N, $n=$ 1–6): the MS spectra of all the 1NP*n*N (n = 1–6) showed corresponding molecular ion peaks and similar fragments, 199 (naphthalene–$COOC_2H_4^+$), 172 (naphthalene–CO_2H^+), 155 (naphthalene–CO^+), 127 (naphthalene$^+$). The preparation of 1NP3N is typical for all 1NP*n*N from the corresponding polyethyleneglycol and 1-naphthoic acid. To a stirred solution of 0.20 cm^3 (1.5 mmol) of triethyleneglycol, 0.51 mg (3.0 mmol) of 1-naphthoic acid and 0.012 g (0.10 mmol) of 4-dimethylaminopyridine (DMAP) in dichloromethane (10 cm^3), 0.61 g (3.0 mmol) of *N*,*N*-dicyclohexyl carbodiimide (DCC) at 0°C was added. The stirring was continued for 1 h at 0°C and then overnight at room temperature. White precipitates were filtered off and the filtrate was washed with 1 mol dm^{-3} HCl and then with saturated sodium bicarbonate. Drying over anhydrous magnesium sulfate and concentration under vacuum yielded a crude product. Chromatography on silica gel with benzene/ethylacetate (90/10, in volume) as eluent yielded pure 1,8-bis(1-naphthalenecarboxy)oxaalkanes (1NP3N) (0.39 g, 57%). The physical data for 1NP*n*N (n = 1–6) were reported in the literature [8].

Ethyl 1-naphthoate (1EN) was prepared from ethyl alcohol and 1-naphthoic acid, and 1,2-bis(1-naphthalenecarboxy) cyclohexane (cyclo-1NP1N) was prepared from *trans*-1,2-cyclohexandiol and 1-naphthoic acid by the same method of preparation as 1NP*n*N. The MS spectra of 1EN and cyclo-1NP1N showed corresponding molecular ion peaks and similar fragments, 199 (naphthalene–$COOC_2H_4^+$), 172 (naphthalene–CO_2H^+), 155 (naphthalene–CO^+), 127 (naphthalene$^+$).

EN: ^{1}H NMR ($CDCl_3$) δ 1.46 (3H, t, $J = 7.0$ Hz, CH_3), 4.48 (2H, q, $J = 7.0$ Hz, CH_2O), 7.45–8.94 (7H, m, naphthalene); ^{13}C NMR (70 MHz, $CDCl_3$) δ 14.39 (CH_3), 61.03 (CH_2O), 124.48, 125.84, 126.15, 127.58, 127.65, 128.32, 130.01, 131.36, 133.15, 133.87 (naphthalene), 167.64 (C=O).

Cyclo-1NP1N: ^{1}H NMR ($CDCl_3$) δ 1.43–1.80 (4H, m, 4, 5-H of cyclohexane ring), 1.80–2.00 (2H, m, 3, 6-Hax of cyclohexane ring), 2.30–2.50 (2H, m, 3, 6-Heq of cyclohexane ring), 5.30–5.50 (2H, m, 1, 2-H of cyclohexane ring), 7.36–8.89 (14H, m, naphthalene); ^{13}C NMR (70 MHz, $CDCl_3$) δ 23.70, 30.50, 74.46 (cyclohexane ring), 124.55, 125.70, 126.15, 127.20, 127.65, 130.10, 131.30, 133.20, 133.75 (naphthalene), 167.00 (C=O).

,*n*-Bis(2-naphthalenecarboxy)oxaalkanes (2NP*n*N, n = 1–6) and ethyl 2-naphthoate (2EN) were prepared from the corresponding polyethyleneglycol or ethyl alcohol and 2-naphthoic acid by the same method of preparation as for 1NP*n*N. The physical data for 2NP*n*N (n = 1–6) were identical with literature values [6]. The MS spectra of the 2EN showed corresponding molecular ion peaks and similar fragments, 199 (naphthalene–$COOC_2H_4^+$), 172 (naphthalene–CO_2H^+), 155 (naphthalene–CO^+), 127 (naphthalene$^+$).

2EN: ^{1}H NMR ($CDCl_3$) δ 1.45 (3H, t, $J = 7.0$ Hz, CH_3), 4.45 (2H, q, $J = 7.0$ Hz, CH_2O), 7.50–8.61 (7H, m, naphthalene); ^{13}C NMR (70 MHz, $CDCl_3$) δ 14.40 (CH_3), 61.05 (CH_2O), 125.25, 126.55, 127.73, 127.83, 128.04, 128.11, 129.32, 130.90, 132.55, 135.50 (naphthalene), 166.74 (C=O).

3. Results and discussion

3.1. Absorption spectra and emission spectra

The UV absorption spectra of NP*n*N (n = 1–6) are essentially identical with the reference compounds 1EN or 2EN. Fig. 1 shows the spectra of 1NP4N and the reference compound 1EN as a typical example. Ground state intramolecular interactions, such as charge transfer (CT), were excluded by the absence of a new band at a longer wavelength for NP*n*N.

In contrast, emission spectra of NP*n*N showed striking differences from that of the reference compound 1EN or 2EN. Although 1EN and 2EN showed only emission from the naphthalene chromophore in acetonitrile, structureless broad emissions were observed in the longer wavelength region for NP*n*N (n = 1–6). For example, on excitation at 295 nm, the fluorescence spectra of 1NP*n*N (n = 1–6) in acetonitrile at room temperature were shown in Fig. 2 after normalization at 368 nm. The excitation spectra of NP*n*N observed at 360 and 450 nm are identical with the absorption spectra, indicating that these new longer wavelength emissions are due to the excited state complex formation, i.e. intramolecular excimer formation.

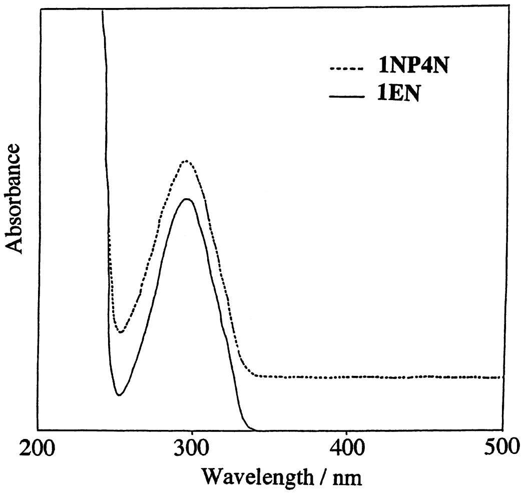


Fig. 1. UV absorption spectra of 1NP4N and 1EN in acetonitrile at 25°C.

Fluorescence quantum yields (Φ) relative to that of naphthalene of NP*n*N in acetonitrile are given in Table 1, in which Φ_{total} is the quantum yield for total emission, Φ_{LE} is that of a locally excited emission and Φ_{EX} is that of the EX. The emission spectra of the excimer were obtained by subtraction of the spectra of 1EN or 2EN from those of NP*n*N after normalization at the peak wavelength of naphthalene, i.e. to say, $\Phi_{\text{EX}} = \Phi_{\text{total}} - \Phi_{\text{LE}}$. The order of the ratio of $\Phi_{\text{EX}}/\Phi_{\text{LE}}$ of NP*n*N (n = 1–6) is $2 > 3 > 4 > 5 > 6 > 1$ in acetonitrile. An EF requires actual overlapping of the two chromophores. However, the intensity of the EX of NP1N was lower than that of NP*n*N (n = 2–6). It is well established that the s-*trans* conformation of the ester group C–O–C(=O) is more stable than the s-*cis* conformation, at least in the ground state, even for a simple ester like methyl acetate by ca. 35 kJ mol^{-1}

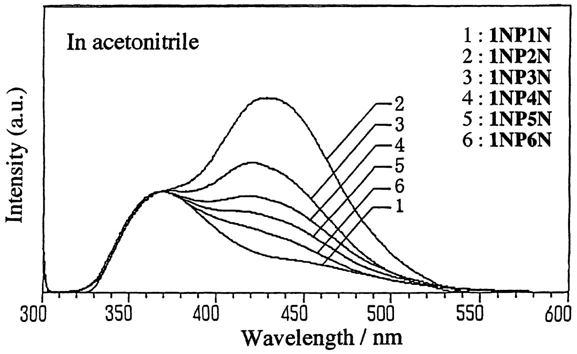


Fig. 2. Fluorescence spectra of 1NP*n*N (n = 1–6) in acetonitrile at 25°C after normalization at 368 nm.

Table 1
Fluorescence quantum yields of 1NP*n*N and 2NP*n*N (n = 1–6) in acetonitrile at 25°C

Solvents (ε)	1NP1N	1NP2N	1NP3N	1NP4N	1NP5N	1NP6N
Acetonitrile (36.2)						
Φ_{total}	0.15	0.11	0.12	0.15	0.16	0.17
Φ_{LE}	0.11	0.03	0.05	0.08	0.09	0.11
Φ_{EX}	0.04	0.08	0.07	0.07	0.07	0.06
$\Phi_{\text{EX}}/\Phi_{\text{LE}}$	0.36	2.70	1.40	0.88	0.78	0.54
Solvents (ε)	2NP1N	2NP2N	2NP3N	2NP4N	2NP5N	2NP6N
Acetonitrile (36.2)						
Φ_{total}	0.17	0.08	0.15	0.15	0.13	0.13
Φ_{LE}	0.13	0.04	0.10	0.11	0.10	0.10
Φ_{EX}	0.04	0.04	0.05	0.04	0.03	0.03
$\Phi_{\text{EX}}/\Phi_{\text{LE}}$	0.31	1.00	0.50	0.36	0.30	0.30

[13]. For NP1N with bulky aromatic groups on both sides of the ester group, the s-*trans* conformation must be considerably more favorable than the s-*cis*; therefore, the conformation required for the two chromophores to be in proximity would be energetically costly. Accordingly, weak EX would be observed in NP*n*N. NP1N would be the extended conformation. Recently, ab initio molecular orbital calculations (Gaussian 98) using a Hartree–Fock SCF method with a 6-31G basis set were carried out for 1NP1N as a flexible chain-linked molecule which formed an excimer to provide the most favorable orientation of the two chromophores [14]. The results of the molecular orbital calculations suggest that the extended conformation is more favorable than the folded one. If 1NP1N was in a folded conformation, a strong EX would be observed. Therefore, we synthesized cyclo-1NP1N, which has a folded conformation for the two chromophores with respect to the C_1–C_2 bond of the cyclohexane ring. It can be used as a rigid model compound for the folded conformer of 1NP1N. Cyclo-1NP1N showed a strong EX as shown in Fig. 3. When the chain length is increased, the two chromophores can easily approach close enough to interact by rotation around other C–C or C–O bonds even with the fixed s-*trans* ester group. The best chain length to excimer formation would be $n = 2$. However, the degree of the intensity of EX is decreased as the polyethylene chain length increases, because the two chromophores cannot easily approach close enough to interact.

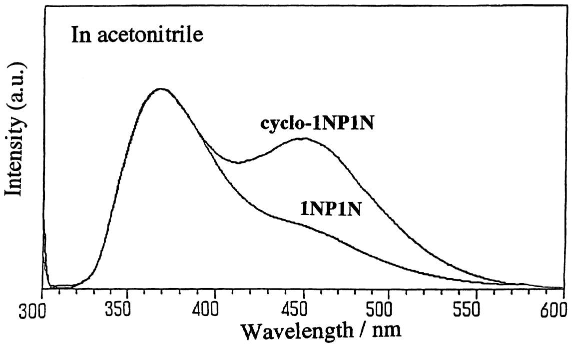


Fig. 3. Fluorescence spectra of 1NP1N and cyclo-1NP1N in acetonitrile at 25°C.

3.2. Metal ion recognition

Measurement of fluorescence spectra was carried out in acetonitrile solution of NP*n*N (n = 1–6; 1.0×10^{-5} mol dm^{-3}) at room temperature, and alkali or alkaline earth metal salts ($LiClO_4$, $NaClO_4$, $KClO_4$, $Mg(ClO_4)_2$, $Ca(ClO_4)_2$ and $Ba(ClO_4)_2$) were added to the solution. To prevent nonlinearity of the fluorescence intensity, the isosbestic points of the absorption spectra of NP*n*N were chosen as the excitation wavelength of NP*n*N.

When lithium, sodium, potassium, and magnesium salts were added to an acetonitrile solution of NP*n*N (n = 1–6), the shape and intensity of fluorescence spectra were not changed, and when calcium and barium salts were added, changes in the fluorescence spectra of NP*n*N (n = 1–3) were not observed. However, the spectra of NP*n*N (n = 4–6) changed by addition of calcium and barium salts.

To study the complexation behavior of NP*n*N (n = 4–6) with Ca^{2+} and Ba^{2+}, the measurement of the fluorescence was carried out in detail. The fluorescence spectra of NP*n*N ($n = 5, 6$) in the presence of several concentrations of $Ca(ClO_4)_2$ and $Ba(ClO_4)_2$ are shown in Figs. 4 and 5 as typical examples. We separated the emission spectra of NP*n*N (n = 4–6) in the presence of several concentrations of Ca^{2+} and Ba^{2+} by the emission spectra of the reference compounds 1EN or 2EN, and we obtained locally excited emission (LE) and EX spectra of NP*n*N. In both the cases of $Ca(ClO_4)_2$ and $Ba(ClO_4)_2$, the LE emission decreased by the addition of the metal salt, and the EX emission increased.

The association constants (K) and fluorescence intensities of the complex were evaluated from these intensities. Even though the solvent takes part in the association interaction, the solvent concentration is virtually unaffected. Therefore, we express the interaction of metal salts in terms of the equilibrium [15]:

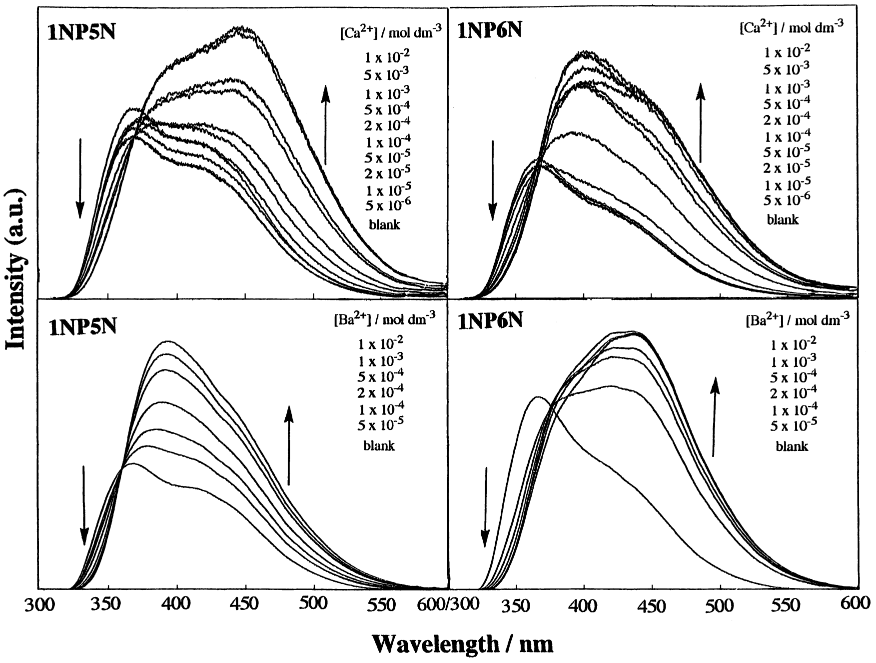


Fig. 4. Fluorescence spectra of 1NPnN (n = 5, 6) and their Ca^{2+} and Ba^{2+} complexes, as excited at 295 nm. [1NPnN] = 1×10^{-5} mol dm^{-3} in acetonitrile at 25°C.

$$\mathrm{NP}n\mathrm{N} + \mathrm{M}^{2+} \rightleftarrows \mathrm{NP}n\mathrm{N}\cdot\mathrm{M}^{2+}$$
$$(\mathrm{NP}n\mathrm{N} = 1\mathrm{NP}n\mathrm{N}\ \text{or}\ 2\mathrm{NP}n\mathrm{N},\ \mathrm{M}^{2+} = \mathrm{Ca}^{2+}\ \text{or}\ \mathrm{Ba}^{2+}) \tag{1}$$

Also, the association constants (K) should be expected as follows:

$$K = \frac{[\mathrm{NP}n\mathrm{N}\cdot\mathrm{M}^{2+}]}{[\mathrm{NP}n\mathrm{N}][\mathrm{M}^{2+}]} = \frac{[\mathrm{NP}n\mathrm{N}\cdot\mathrm{M}^{2+}]}{\{([\mathrm{NP}n\mathrm{N}]_0-[\mathrm{NP}n\mathrm{N}\cdot\mathrm{M}^{2+}])([\mathrm{M}^{2+}]_0-[\mathrm{NP}n\mathrm{N}\cdot\mathrm{M}^{2+}])\}} \tag{2}$$

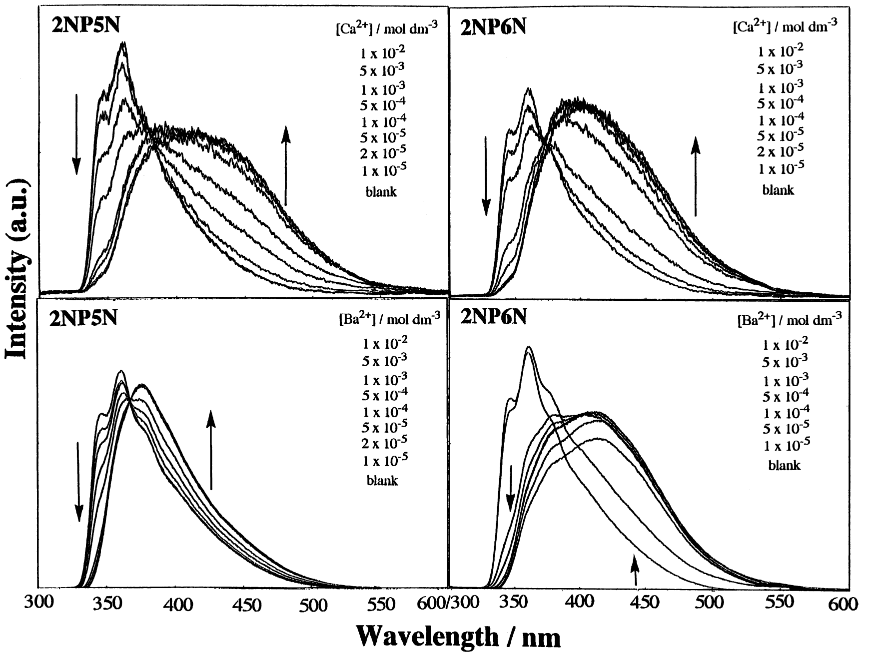


Fig. 5. Fluorescence spectra of 2NPnN (n = 5, 6) and their Ca^{2+} and Ba^{2+} complexes, as excited at 265 nm. [2NPnN] = 1×10^{-5} mol dm^{-3} in acetonitrile at 25°C.

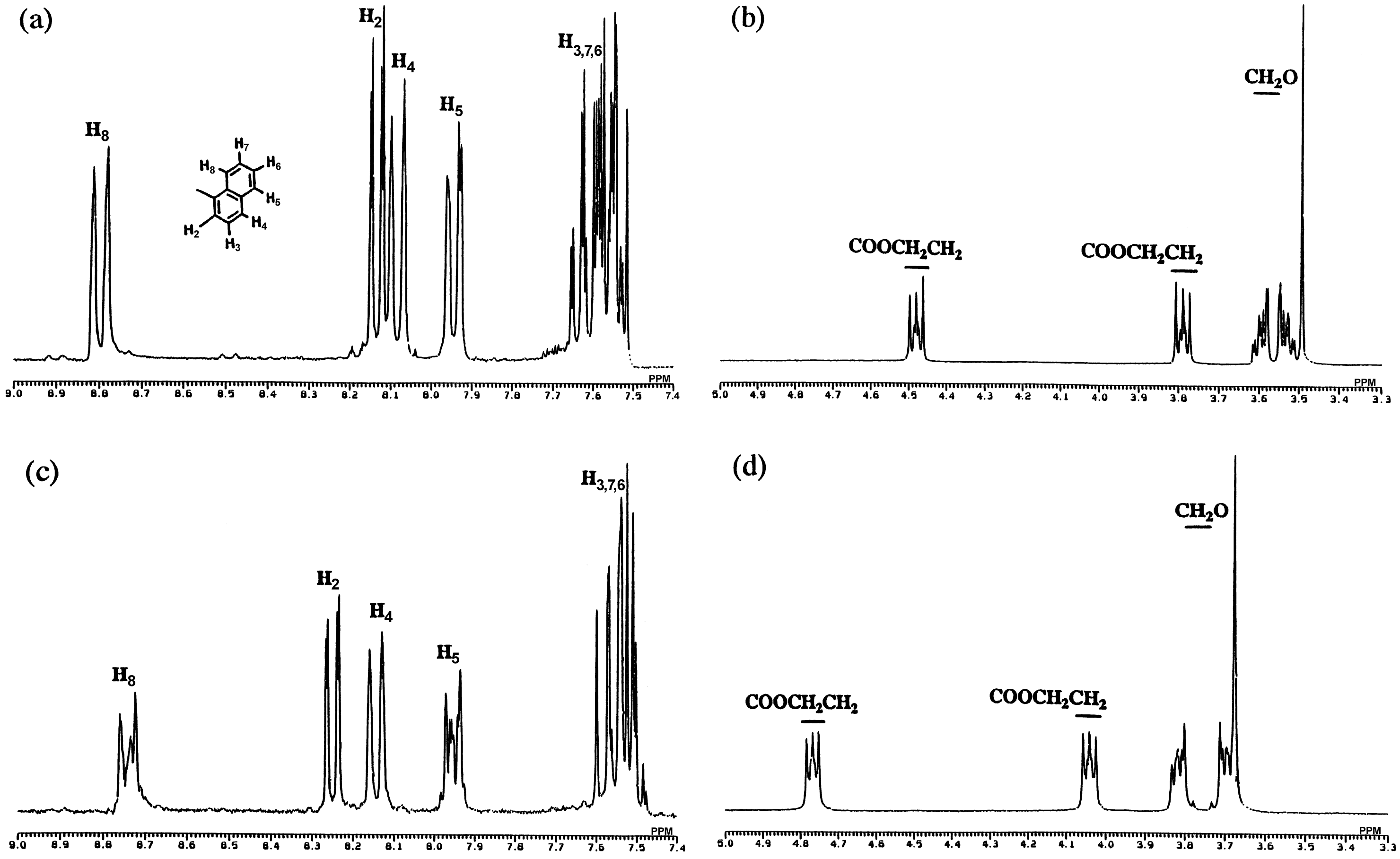


Fig. 6. ^1H NMR spectra of 1NP5N in the absence ((a) and (b)) and presence ((c) and (d)) of barium salt in acetonitrile-d_3 at room temperature.

Table 2
The association constants (K) of 1NPnN nad 2NPnN ($n = 5, 6$)

	log K			
	1NP5N	1NP6N	2NP5N	2NP6N
Ca^{2+}	3.72	4.36	4.19	4.95
Ba^{2+}	3.86	4.78	4.32	4.88

$$[\mathrm{NP}n\mathrm{N}\cdot\mathrm{M}^{2+}] = a[\mathrm{NP}n\mathrm{N}]_0 = \left\{\frac{(I_{\max}-I)}{(I_{\max}-I_0)}\right\}[\mathrm{NP}n\mathrm{N}]_0 \qquad (3)$$

From Eqs. (2) and (3), the following equation could be derived:

$$[\mathrm{M}^{2+}]_0 = \frac{a}{K(1-a)} + a[\mathrm{NP}n\mathrm{N}]_0, \qquad (4)$$

where $[\mathrm{NP}n\mathrm{N}]_0$ and $[\mathrm{M}^{2+}]_0$ are the initial concentrations of NPnN and the metal salts, I and $I_{\max}$ the observed LE intensities of the NPnN in the presence and in the absence of the metal ion and I_0 the observed LE intensity of the complex of the NPnN and the metal ion. A self-written nonlinear curve-fitting computer program (Eq. (4)) was used to fit the experimental titration curves. The association constants were determined from the emission-intensity changes at 365 nm (for 1NPnN) and 360 nm (for 2NPnN) using the equation. When calcium and barium salts were added to an acetonitrile solution of NP4N, the shape and intensity of the fluorescence spectra varied only slightly, and the values of log K for Ca^{2+} and Ba^{2+} were low. NP4N formed the most unstable complex with Ca^{2+} and Ba^{2+}. Oxygen atoms of polyether chain are necessary to bind metal ions. In the case of scant oxygen atoms, ability of complexation for metal ions would decrease. Actually, NPnN (n = 1–3) having a shorter polyether chain cannot form complexes with metal ions. While in NPnN (n = 5, 6), the shape and intensity of the fluorescence spectra varied greatly. These showed that the complexing ability was mostly determined by polyether chain length. The values of log K of NPnN (n = 5, 6) are reported in Table 2.[1] The order of the value of log K for Ca^{2+} and Ba^{2+} is 2NP6N > 1NP6N > 2NP5N > 1NP5N. 2NP6N formed the most stable complex with Ca^{2+} and Ba^{2+}. In the cases of same length of the polyether chain, 2NPnN having β-substituted naphthalene is an excellent fluorescent chemosensor for Ca^{2+} and Ba^{2+}, better than 1NPnN having α-substituted naphthalene. There is a relation between EX and the overlapping of two chromophres. In β-substituted naphthalene, correlation of carbonyl group with aromatic ring is conjugated coplanar. While in α-substituted naphthalene, it is difficult to form coplanar between carbonyl group and aromatic ring, because carbonyl group would be twisted by steric hindrance for the hydrogen atom of peri-position. Therefore, for the overlapping of two chromophores, 2NPnN having β-substituted naphthalene would be better than 1NPnN ($n = 5, 6$) having α-substituted naphthalene. These results suggest that in the cases of same length of the polyether chain, 2NPnN is an excellent fluorescent chemosensor for Ca^{2+} and Ba^{2+}, better than 1NPnN.

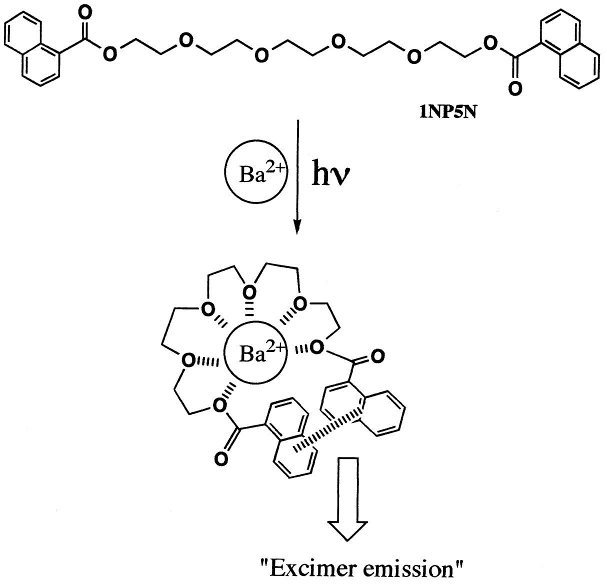


Fig. 7. Complex formation of 1NP5N with Ba^{2+}.

Binding interactions of NPnN with metal ions were examined using ^{1}H NMR spectroscopy. ^{1}H NMR spectra in acetonitrile-d_3 gave information as to the structures of the complexes. When NPnN (n = 4–6) is complexed with Ca^{2+} and Ba^{2+}, all peaks of the polyether chain protons shifted to lower magnetic field. Ethereal protons shift downfield because the electron density of the oxygen atoms is reduced by the coordinated cation. ^{1}H NMR spectra of 1NP5N in the absence and presence of barium salt are shown in Fig. 6 as typical examples. The H_2 proton of the naphthalene ring shifted to lower magnetic field too. The downfield shift of the aromatic proton could be attributed to the ring current effect of each aromatic unit. From these results, the mechanism of the complex formation is suggested in Fig. 7.

The ionic diameters (r) of Li^+ and Mg^{2+} are smaller (ca. 0.13 nm) than other metal ions (r = 0.20–0.27 nm), and Li^+, Na^+ and K^+ are monovalent ions. These results showed that NPnN (n = 5, 6) formed a more stable complex with the divalent metal ions than with the monovalent metal ions, and the desirable size of metal ionic diameters to form a complex was $r > 0.2$ nm. The magnesium ion is divalent, but NPnN (n = 5, 6) could not form complex with Mg^{2+} having small ionic diameters, i.e. ca. 0.13 nm.

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[1] The values of log K are not consistent with the values in [8]. In this paper, we used perchlorates as metal salts, and the values of K were recalculated by a self-written nonlinear curve-fitting computer program (Eq. (4)).

4. Conclusion

In the absence of metal salts, the excimer formation of NP*n*N ($n = 4$–6) is not easier than for NP*n*N ($n = 2, 3$), because the two chromophores cannot easily approach close enough to interact. However, in the presence of calcium and barium salts, the ratio of $\Phi_{\mathrm{EX}}/\Phi_{\mathrm{LE}}$ is increased. These results suggest that the two chromophores can easily approach close enough to interact by binding of the oxygen atoms of the polyether chain in NP*n*N ($n = 4$–6) with Ca^{2+} and Ba^{2+}. The changes in the shape and intensity of fluorescence spectra were great for NP*n*N ($n = 5, 6$). The values of $\log K$ for Ca^{2+} and Ba^{2+} were 2NP6N > 1NP6N > 2NP5N > 1NP5N. In the cases of same length of the polyether chain, 2NP*n*N is an excellent fluorescent chemosensor for Ca^{2+} and Ba^{2+}, better than 1NP*n*N.

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