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Spectral study on the inclusion complex of cryptophane-E and $CHCl_3$

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ABSTRACT

Cryptophane-E was synthesized from vanillin by a three-step method, and its absorption and fluorescence spectroscopic properties were determined. Two absorption bands at about 245–260 and 280–290 nm were observed for cryptophane-E and the fluorescence emission maxima were at 320–330 nm depending on the solvent used. The interaction of cryptophane-E with $CHCl_3$ was studied in detail by absorption and fluorescence spectroscopies. The results showed that cryptophane-E and $CHCl_3$ can easily form a stable 1:1 host–guest inclusion complex. Their binding constant (K) was determined by Benesi–Hildebrand equation and the nonlinear least squares fit method. The binding constant is largest in ethyl acetate, followed by dioxane and with acetonitrile as the smallest. In addition, the effect of guest volume on the host–guest inclusion complex was investigated. Guest molecules including CH_2Cl_2 and CCl_4 were unable to form inclusion complex with cryptophane-E because of sizes mismatching with the host cavity.

1. Introduction

In recent years, the design of hosts capable of binding neutral substrates has attracted much attention in supramolecular chemistry, and, in this context, the capture of the smallest aliphatic hydrocarbons still remains a challenging problem [1–9]. Cryptophanes are a group of interesting cage-like hosts consisted of two rigid cone-shaped units (cyclotriveratrylene) linked by three O–Z–O bridges [10–12]. They are well suited for inclusion of molecules of various sizes and shapes because the dimensions of the host cavity can be easily modified by simple structural changes. For instance, NMR studies and CPK models indicate that cryptophane-A, with three $-OCH_2CH_2O-$ bridges, preferentially binds CH_4 and CH_2Cl_2 [13,14] whereas cryptophane-E with a larger cavity of three $-OCH_2CH_2CH_2O-$ bridges, as shown in Scheme 1, prefers to bind $CHCl_3$ [15–17].

The complex of cryptophane-E and $CHCl_3$ has been studied by NMR spectroscopy and computational chemistry methods [15–18]. It is found that $CHCl_3$ behaves as an integral part of the cryptophane-E host after inclusion. The inclusion complex is relatively stable and the binding constant was estimated to be 470 M^{-1} in $(CDCl_2)_2$ at 300 K by NMR. Tosner et al. [19] also reported the formation of cryptophane-E and $CHCl_3$ inclusion complex using advanced solid state NMR spectroscopy. Their results show that the mobilities of the encaged guest in the solid state and in the isotropic liquid solution were very similar. Although their techniques can provide valuable information on the binding constant of the inclusion complex, NMR spectrometers are still relatively expensive and not commonly available in most laboratories. As such, cheaper equipment such as UV–vis absorption and fluorescence spectrophotometers for studying the inclusion complex behavior of host–guest interaction should be explored. To the best of our knowledge, there are very few studies on cryptophane-E and halogenated compounds using fluorescence and absorption spectroscopies.

In this article, the fundamental spectroscopic characteristics of cryptophane-E in different organic solvents, the inclusion complex of cryptophane-E and $CHCl_3$, binding constants, solvent effect on the complex and interaction of cryptophane-E with CH_2Cl_2 and CCl_4 have been investigated in detail. Our results are to some extent consistent with the reported NMR spectroscopic data. These fundamental data and the spectral method should provide valuable information for studying the structure, dynamics, and ordering behavior of the host–guest inclusion complexes.

2. Experimental

2.1. Materials

Acetonitrile, dibromoethane, diethyl ether, dioxane, ethanol, ethyl acetate, methanol, sodium borohydride ($NaBH_4$), and vanillin

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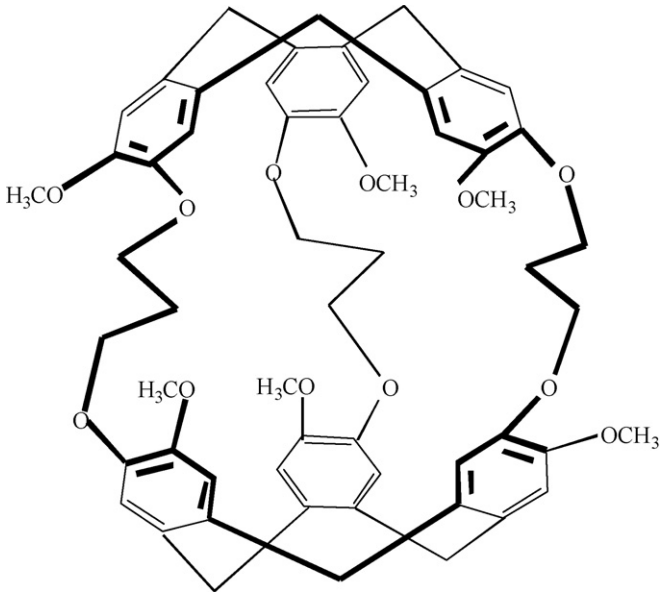


Scheme 1. The structure of cryptophane-E.

were purchased from Beijing Chemical Plant (Beijing, China). Organic solvents were purified before use and were checked to ensure they contained no fluorescence impurities at the working excitation wavelengths (292–298 nm). Other chemicals and reagents for the synthesis of cryptophane-E were used as received unless otherwise stated.

2.2. Synthesis of cryptophane-E

Cryptophane-E was synthesized from vanillin using a three-step method [20]. The compound 1,3-bis(4-formyl-2-methoxyphenoxy)propane was prepared by reacting vanillin with 1/2 equivalents of dibromoethane. The bis-aldehyde derivative was then reduced to its corresponding bis-vanillyl alcohol by $NaBH_4$ in methanol. The ring closure reaction of benzylic alcohol was performed in a 1.4–1.5 mmol L^{-1} formic acid solution at 60 °C for 3 h. The reaction mixture was purified by column chromatography on silica gel to obtain 13.0% yield of cryptophane-E. The ^{1}HNMR ($CDCl_3$) data of cryptophane-E were acquired: δ = 6.70 (s, 6H, Ar), 6.62 (s, 6H, Ar), 4.65 (d, 6H, CH_a), 4.04 (m, 12H, OCH_2), 3.87 (s, 18H, OCH_3), 3.50 (d, 6H, CH_e), and 2.31 (m, 6H, CH_2). Satisfactory elemental analysis (C, H) was obtained, Anal. Calcd for $C_{57}H_{60}O_{12}$: C, 73.06; H, 6.45; O, 20.49. Found: C, 72.18; H, 6.03; O, 20.62. These data were consistent with the literature values [20].

2.3. Instrumentation

Fluorescence spectra were taken on a F4500 Hitachi spectrofluorometer (Tokyo, Japan). Both excitation and emission slits were set at 5 nm. All the experiments were carried out at 20 ± 1 °C. ^{1}H NMR spectra were done on a Bruker Avance DRX 300 MHz nuclear magnetic resonance spectrometer (Fällanden, Switzerland).

3. Results and discussion

3.1. Absorption spectra of cryptophane-E in different organic solvents

Fig. 1 displays the UV–vis absorption spectra of cryptophane-E (3.21×10^{-5} mol L^{-1}) in different organic solvents including acetonitrile, $CHCl_3$, diethyl ether, dioxane, ethanol, ethyl acetate, and methanol. Cryptophane-E shows two absorption peaks at

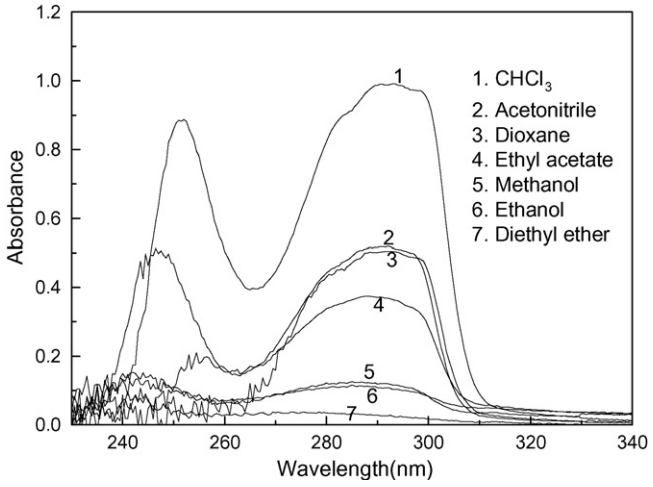


Fig. 1. UV–vis absorption spectra of cryptophane-E (3.21×10^{-5} mol L^{-1}) in different organic solvents.

~245–260 and ~280–300 nm depending on the type of solvent used. The absorption intensity of cryptophane-E in $CHCl_3$ is highest, followed by acetonitrile, dioxane and ethyl acetate. The lowest absorption intensities are in diethyl ether, ethanol and methanol. The absorption bands of diethyl ether, dioxane, ethanol, and methanol and are not as distinctive (~245–260 nm) as that of acetonitrile, $CHCl_3$ and ethyl acetate. The more obvious and distinctive absorption bands of cryptophane-E in $CHCl_3$ are possibly attributed to the formation of inclusion complex of cryptophane-E and $CHCl_3$.

3.2. Inclusion complex of cryptophane-E and $CHCl_3$ determined by absorption

It has been reported that cryptophanes can form inclusion complexes with various halogenomethanes [14–16]. The absorption spectra of cryptophane-E with various $CHCl_3$ concentrations in ethyl acetate, dioxane and acetonitrile are depicted in Fig. 2(a)–(c), respectively. The absorption intensity of cryptophane-E decreases with the increase in $CHCl_3$ concentration. The spectral data at their absorption peak maxima were then analyzed with the Benesi–Hildebrand equation [21] as depicted in the insets of Fig. 2. The binding constants were found to be 542.2, 316.7 and 189.6 M^{-1} in ethyl acetate, dioxane and acetonitrile, respectively. The linear correlation coefficients (r) were higher than 0.993. The results suggest that the decrease in absorption intensity is mainly attributed to the formation of host–guest inclusion complexes between cryptophane-E and $CHCl_3$. The binding constants decrease in the trend: ethyl acetate > dioxane > acetonitrile, demonstrating that different solvents can affect the binding of cryptophane-E and $CHCl_3$. The binding constant is smallest in acetontitrile, possibly attributed to its competition with $CHCl_3$ for binding with cryptophane-E. In addition, as dioxane has similar structure to cryptophane-E which has a rigid framework of three "–$OCH_2CH_2CH_2O$–" bridges, it is possible that dioxane competes with cryptophane-E for interacting with $CHCl_3$; as a result, the binding constant in dioxane is lower.

3.3. Fluorescence spectra of cryptophane-E in various organic solvents

Fig. 3 displays the fluorescence emission spectra of cryptophane-E (2.91×10^{-5} mol L^{-1}) in different organic solvents including acetonitrile, diethyl ether, dioxane, ethanol, ethyl acetate, and methanol. Table 1 summarizes their fluorescence excitation (λ_{ex}) and emission (λ_{em}) maxima. The λ_{ex} and λ_{em} of

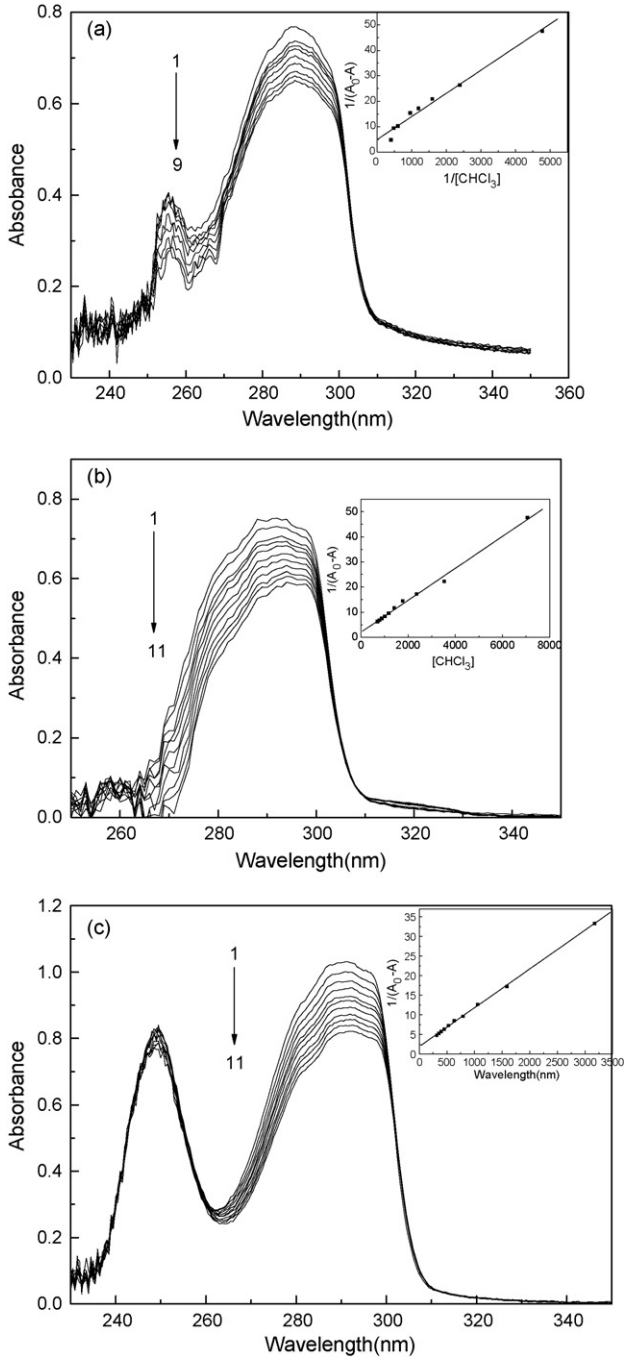


Fig. 2. A (a) UV–vis absorption spectra of cryptophane-E (3.21×10^{-5} mol L^{-1}) with $CHCl_3$ in ethyl acetate. The concentrations of $CHCl_3$ are (1) 0.00 mol L^{-1}, (2) 2.01×10^{-4} mol L^{-1}, (3) 4.19×10^{-4} mol L^{-1}, (4) 6.29×10^{-4} mol L^{-1}, (5) 8.38×10^{-4} mol L^{-1}, (6) 1.05×10^{-3} mol L^{-1}, (7) 1.68×10^{-3} mol L^{-1}, (8) 2.09×10^{-3} mol L^{-1}, and (9) 2.51×10^{-3} mol L^{-1}. The inset displays the Benesi–Hildebrand plot and the absorption values were taken at 288 nm. (b) UV–vis absorption spectra of cryptophane-E (6.42×10^{-5} mol L^{-1}) with $CHCl_3$ in dioxane. The concentrations of $CHCl_3$ are (1) 0.00 mol L^{-1}, (2) 1.42×10^{-4} mol L^{-1}, (3) 2.83×10^{-3} mol L^{-1}, (4) 4.52×10^{-3} mol L^{-1}, (5) 5.66×10^{-3} mol L^{-1}, (6) 7.08×10^{-3} mol L^{-1}, (7) 8.49×10^{-3} mol L^{-1}, (8) 9.91×10^{-3} mol L^{-1}, (9) 1.13×10^{-2} mol L^{-1}, (10) 1.27×10^{-2} mol L^{-1}, and (11) 1.41×10^{-2} mol L^{-1}. The inset displays the Benesi–Hildebrand plot and the absorption values were taken at 294 nm. (c) UV–vis absorption spectra of cryptophane-E (6.42×10^{-5} mol L^{-1}) with $CHCl_3$ in acetonitrile. The concentrations of $CHCl_3$ are (1) 0.00 mol L^{-1}, (2) 3.16×10^{-4} mol L^{-1}, (3) 6.31×10^{-4} mol L^{-1}, (4) 9.47×10^{-4} mol L^{-1}, (5) 1.26×10^{-3} mol L^{-1}, (6) 1.88×10^{-3} mol L^{-1}, (7) 1.89×10^{-3} mol L^{-1}, (8) 2.21×10^{-3} mol L^{-1}, (9) 2.52×10^{-3} mol L^{-1}, (10) 2.84×10^{-3} mol L^{-1}, and (11) 3.16×10^{-3} mol L^{-1}. The inset displays the Benesi–Hildebrand plot and the absorption values were taken at 291 nm.

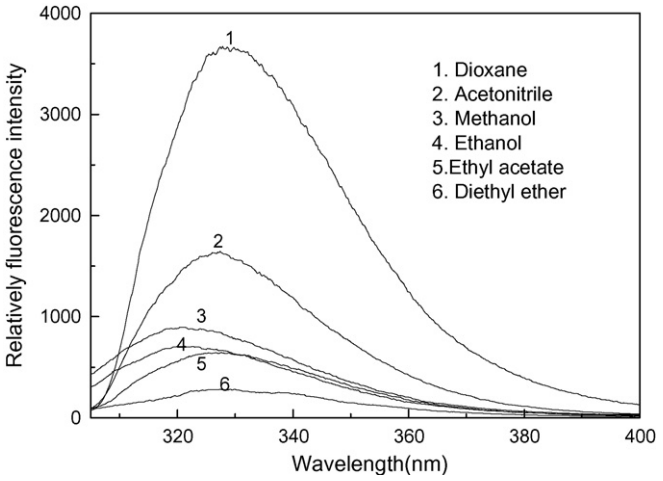


Fig. 3. Fluorescence emission spectra of cryptophane-E (2.91×10^{-5} mol L^{-1}) in different organic solvents. Excitation wavelengths are at their corresponding excitation maxima.

cryptophane-E in these solvents were 292–298 and 322–330 nm, respectively. It seems that cryptophane-E does not vary too much with the change in solvent polarity, possibly attributing to the rigid nature of the molecule. This shows that solvent polarity has not much effect on λ_{ex} and λ_{em} under different solvent systems in our work, which is different from the literatures that the λ_{ex} and λ_{em} are slightly red-shifted in more lipophilic solvent systems [22,23]. The fluorescence intensity of cryptophane-E in these solvents follows the trend: dioxane > acetonitrile > methanol > ethanol > ethyl acetate > diethyl ether; however, this trend is still not clearly understood.

3.4. Inclusion complex of cryptophane-E and $CHCl_3$ studied by fluorescence

Cryptophane-E displays good fluorescence in ethyl acetate as depicted in Fig. 4. It has a λ_{em} of 325 nm when excited at 296 nm. When $CHCl_3$ was added to the cryptophane-E solution, the fluorescence intensity decreased and the emission spectrum also slightly shifted hypsochromically. This can be explained by the fact that an inclusion complex of cryptophane-E and $CHCl_3$ was formed which could affect the fluorescence yield and spectral characteristics of cryptophane-E.

The binding constant can be estimated by the least squares fit to the experimental data obtained from the fluorescence titrations [24] as follows:

$$\Delta F = \frac{1}{2}\left\{\alpha\left([H]_0 + [G]_0 + \frac{1}{K}\right) - \sqrt{\alpha^2\left([H]_0 + [G]_0 + \frac{1}{K}\right)^2 - 4[H]_0[G]_0\alpha^2}\right\}$$

where $[H]_0$ and $[G]_0$ are the initial concentrations of host cryptophane-E and guest $CHCl_3$, respectively. ΔF denotes the change of the fluorescence intensity of cryptophane-E with the

Table 1
The fluorescence excitation (λ_{ex}) and emission (λ_{em}) maxima of cryptophane-E in various organic solvents.

Solvent	Acetonitrile	Diethyl ether	Dioxane	Methanol	Ethyl acetate	Ethanol
λ_{ex} (nm)	295	295	298	292	296	292
λ_{em} (nm)	323	324	330	322	325	324

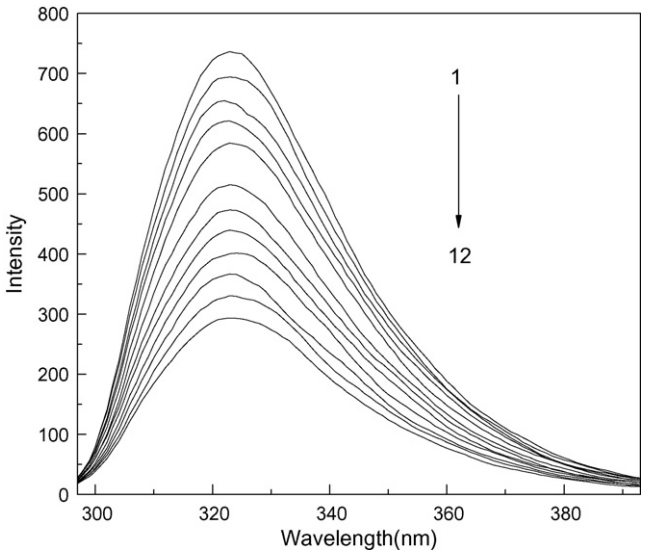


Fig. 4. Fluorescence quenching effect of $CHCl_3$ on the emission intensity of cryptophane-E (2.07×10^{-5} mol L^{-1}) in ethyl acetate. (1) 0.00 mol L^{-1}, (2) 4.20×10^{-4} mol L^{-1}, (3) 9.21×10^{-4} mol L^{-1}, (4) 1.50×10^{-3} mol L^{-1}, (5) 2.16×10^{-3} mol L^{-1}, (6) 2.91×10^{-3} mol L^{-1}, (7) 3.74×10^{-3} mol L^{-1}, (8) 4.66×10^{-3} mol L^{-1}, (9) 5.66×10^{-3} mol L^{-1}, (10) 6.74×10^{-3} mol L^{-1}, (11) 7.90×10^{-3} mol L^{-1}, and (12) 9.15×10^{-3} mol L^{-1} $CHCl_3$. Excitation wavelength is 296 nm. The inset displays the Stern–Volmer plot for cryptophane-E with different $CHCl_3$ concentrations.

addition of $CHCl_3$. α is a sensitive factor of the structure change of the complex cryptophane-E–$CHCl_3$ at the interactive course. The binding constant was estimated to be $106 \pm 9\ M^{-1}$ and r is 0.9972 as shown in Fig. 5. The result of nonlinear fitting is good, indicating that the inclusion complex was formed with a stoichiometric ratio of 1:1. The binding constants were found to be 46 ± 2 and $18 \pm 1\ M^{-1}$ in dioxane and acetonitrile, respectively.

In addition, the inclusion complex of cryptophane-E–$CHCl_3$ was studied by ^{1}HNMR in $(CDCl_2)_2$ at 300 K [17]. The peaks at δ_f 7.28 and δ_c 2.84 ppm corresponding to the free and complexed $CHCl_3$ molecules, respectively slowly exchanged at this temperature. The binding constant of the complex was more than 100 M^{-1} by ^{1}HNMR and was in good agreement with our result by fluorescence. X-ray crystallography confirmed that cryptophane-E–$CHCl_3$ was a 1:1 host–guest [17]. Our result shows some similarity with the reported results [17].

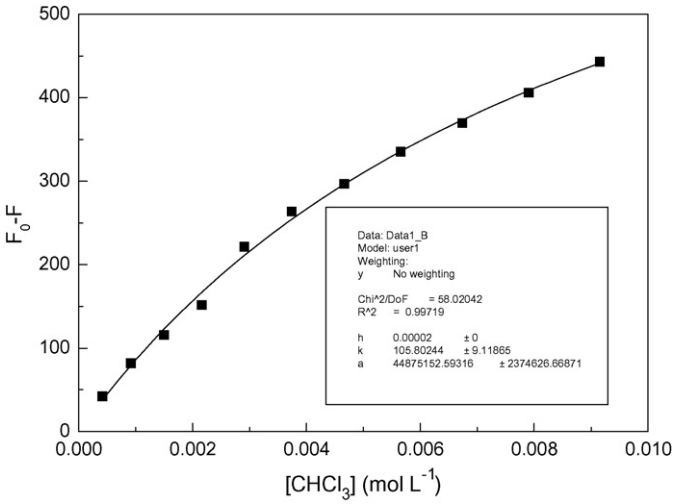
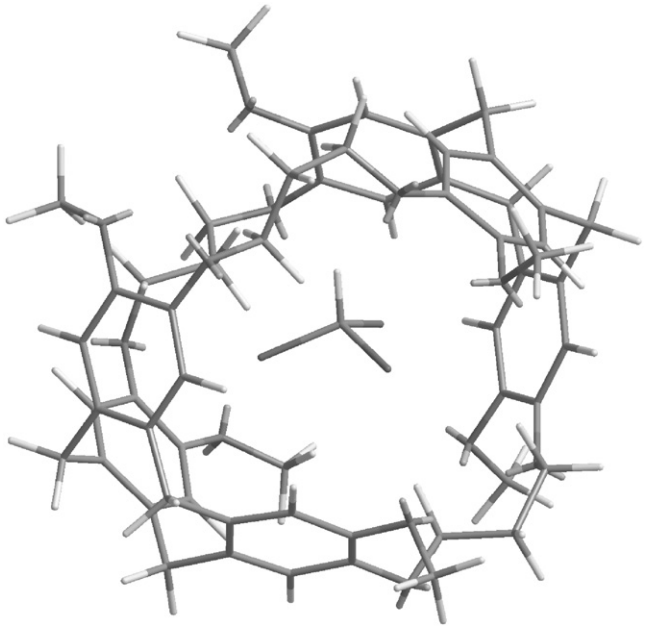


Fig. 5. Nonlinear curve fits for the cryptophane-E–$CHCl_3$ complex in ethyl acetate.



Scheme 2. The optimal structure of the cryptophane-E–$CHCl_3$ complex simulated by the computer modeling.

3.5. Molecular modeling studies

The cryptophane-E–$CHCl_3$ complex was simulated by the molecular dynamic calculation using the Computer Molecular Modeling System CS Chem 3D Pro 7.0 (CambridgeSoft, Cambridge, MA, USA). The optimal configuration of the cryptophane-E–$CHCl_3$ complex that has the lowest total energy is displayed in Scheme 2. This result shows that $CHCl_3$ can penetrate into the cavity of cryptophane-E to form a 1:1 host–guest system.

3.6. Solvent effect on the inclusion complex of cryptophane-E and $CHCl_3$

The binding constants from the absorption and fluorescence data were found to follow the same trend: ethyl acetate > dioxane > acetonitrile; however, there are some slight differences in the binding constants obtained by these two methods. This is probably due to differences in calculation methods. Comparing with ethyl acetate, the acetonitrile molecules are smaller and can compete with the guest $CHCl_3$ molecules causing the decrease in the binding constant. By contrast, ethyl acetate is too bulky to enter the cavity of cryptophane-E; thus, the binding constant is comparatively larger. In addition, cryptophane-E has a rigid framework with three "–$OCH_2CH_2CH_2O$–" bridges and its structure shows some similarities with dioxane. Therefore, dioxane can be a good candidate to compete with cryptophane-E for interacting with $CHCl_3$. This can explain why the binding constant of cryptophane-E and $CHCl_3$ in dioxane solvent is smaller than ethyl acetate. In essence, the formation of inclusion complex of cryptophane-E and $CHCl_3$ not only depends on the host–guest interaction but also encompasses solvent–guest and solvent–host interactions.

3.7. Effect of guest volume

The interaction of cryptophane-E with smaller (CH_2Cl_2) or larger (CCl_4) guests was examined by fluorescence spectroscopy and their occupancy factors [13] as depicted in Table 2. When CH_2Cl_2 was added to cryptophane-E in ethyl acetate solvent, the fluorescence intensity decreased and the binding constant was determined to

Table 2
The guest volume (V_{vdw}) and the occupancy factor of guest in cryptophane-E.

Guest	CH_2Cl_2	$CHCl_3$	CCl_4
K (M^{-1})	2.0 ± 0.2	106 ± 9	–
V_{vdw} (Å^3)	55.6	72.2	86.8
Occupancy factor	0.48	0.62	0.74

The van der Waals volume of the cavity for cryptophane-E is 117 Å^3.

be $2.0 \pm 0.2\ M^{-1}$ which is much smaller than the binding constant of cryptophane-E and $CHCl_3$. CCl_4 does not cause any fluorescence quenching on cryptophane-E. These results indicated that the larger CCl_4 guest cannot penetrate into the cavity of cryptophane-E while the smaller CH_2Cl_2 guest can only weakly interact with cryptophane-E as their sizes do not match well with the cavity of cryptophane-E. The cavity of cryptophane-E is estimated to be 117 Å^3 according to the reference by Mecozzi and Rebek [25]. The occupancy factor is 0.62 [13,25] for one molecule of $CHCl_3$ in one cryptophane-E, which is quite reasonable for such a complex, signifying that it is neither too tight nor too loose. The cryptophane-E and $CHCl_3$ complex thus represents a van der Waals molecule. CH_2Cl_2 has an occupancy factor of 0.48 which is 20% smaller than $CHCl_3$. Thus, it has more space to move in and out of the cavity of cryptophane-E, resulting in an unstable inclusion complex. It is more difficult for CCl_4 to form inclusion complex with cryptophane-E as it has an occupancy factor of 0.74 [13,25] which is too large for the cavity of cryptophane-E. The results of cryptophane-E to guests fit within the range of the widely quoted rule: the ratio of the guest volume to the host volume, is in the range of 0.55 ± 0.09 [25]. These findings are also supported by the CPK model that the six $-OCH_3$ groups of the host can obstruct CCl_4 molecules from entering the cavity of cryptophane-E.

4. Conclusion

In summary, cryptophane-E was synthesized and possesses fluorescence property. Its host property can be studied using molecular spectral methods. Our experimental results demonstrate that the formation of cryptophane-E–$CHCl_3$ inclusion complex is governed by the good matching size of the guest with the cryptophane-E cavity. Ethyl acetate is a favorable solvent for studying the cryptophane-E–$CHCl_3$ inclusion complex but acetonitrile and dioxane are not ideal as they can compete with $CHCl_3$. In addition, the larger CCl_4 or smaller CH_2Cl_2 guest molecules do not suit the cavity of cryptophane-E and so they cannot form stable inclusion complexes with cryptophane-E. Our proposed absorption and fluorescence methods can provide simple and cheaper ways of investigating the inclusion complex.

Acknowledgements

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