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A new ion pair receptor fulfilling a dual function as a chromogenic molecular switch for F^- and ratiometric selective recognition of HSO_4^-

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ABSTRACT

A new ditopic ion pair receptor **1** containing two biindole moieties and a bis-benzocrown ether unit shows a remarkable color switching (ON- and -OFF) function induced by anion (F^-) and cation (K^+) recognition. The ditopic receptor **1** binds in a cooperative fashion to HSO_4^- in the presence of $\mathbf{1}\cdot K^+$ and acts as a selective ditopic receptor to recognize ion pairs with a wavelength-ratiometric manner.

1. Introduction

The design and synthesis of functional molecules [1–5] serving as molecular devices for sensing [6–8], switching [9–12], and signal transduction [13], particularly from optical inputs, have been very actively pursued over the recent years. Compared with simple ion receptors, ion pair receptors are capable of forming simultaneous complexation with cations and anions, and have the potential to act as new selective extraction and membrane transport agents [14–18]. In fact, the related ditopic receptors might offer considerable advantages in terms of affinity or selectivity over monotopic hosts, because the bind guests in a cooperative manner are especially sought for the selective recognition of a specific guest [19–24]. Furthermore, recent studies on anion and ion-pair binding suggest that an anion and counter cation have a strong effect on the binding affinity [25–27]. Thus, the development of this area is stimulated by considerable benefits gained from binding an ion pair. However, the number of pyrrole [28–31] or indole-based [32–34] ion pair receptors remains limited. Therefore, we hope to design an indole-based ion pair receptor that could bind a specific cation–anion pair with high affinity in the form of an ion pair complex.

Herein, we describe the design, synthesis and binding properties of a novel heteroditopic molecular receptor **1**, which contains both biindole and bis-benzocrown moiety (Scheme 1). Biindole moiety was chosen as the anion binding site, since indole-based receptor is better hydrogen bond donor than pyrrole and more prone to deprotonation [35–39]. A bis-benzocrown ether was chosen as the second binding site of the receptor due to its well-known cation binding properties. **1** can function as a chromogenic molecular switch by the adding sequence of F^- and K^+ in DMSO solution to control its color. In addition, when co-bounding cation complex $\mathbf{1}\cdot K^+$, it shows high selectivity for detecting HSO_4^- by the naked eye, and it also demonstrates the obvious fluorescence characteristics change in a wavelength-ratiometric manner in the presence of the HSO_4^-.

2. Experimental

2.1. Apparatus and reagents

All the materials for synthesis were purchased from Alfa Aesar and other companies. DMSO was dried with CaH_2 and then distilled in reduced pressure [40]. In the titration experiments, all the anions were added in the form of tetrabutylammonium (TBA) salts, which were purchased from Alfa Aesar and Aladdin, stored in a vacuum desiccator containing self-indicating silica and fully dried before using. NMR spectra were recorded in [D_6]DMSO at 25 °C with a Varian Unity Plus 400 MHz NMR spectrometer (Varian, USA). High resolution mass spectra (HRMS) were determined on an IonSpec 7.0 T

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FT-ICR mass spectrometer (IonSpec, USA). UV–vis absorption spectra were measured with a Hitachi U-3010 UV–vis spectrophotometer (Hitachi, Japan). Fluorescence spectra were recorded at room temperature on a Varian Cary Eclipse fluorescence spectrometer (Varian, USA).

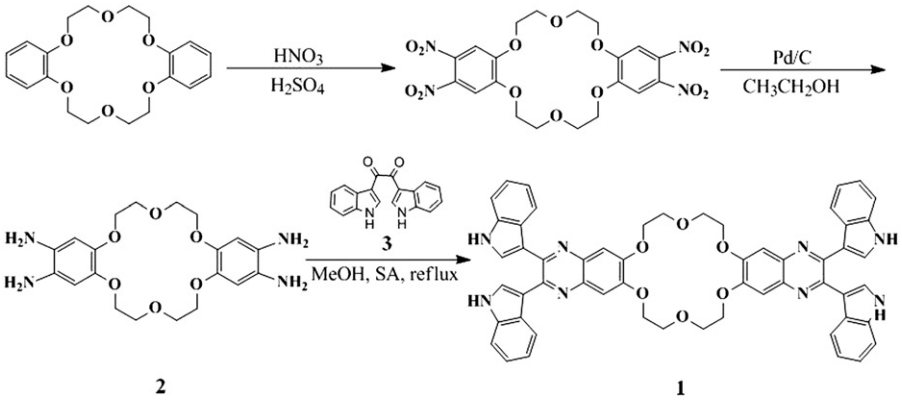
2.2. Synthesis of ion pair receptor **1**

The ditopic receptor, **1,** was prepared by condensation of 6,7,9,10,17,18,20,21-octahydrodi-zo[*b*,*k*] [1,4,7,10,13,16] hexaoxa-cyclooctadecine-2,3,13,14-tetraamine (**2**) and 2,3-diindol-3-yl diketone (**3**). **2** and **3** were prepared by previously reported procedures [41–42]. 2,3-Diindol-3-yl diketone (263 mg, 0.91 mmol) and amidosulfonic acid (SA) (670 mg, 5.49 mmol) were dissolved in methanol (30 mL) under N_2 atmosphere, then 6,7,9,10,17,18,20,21-octahydrodi-zo[*b*,*k*] [1,4,7,10,13,16] hexaoxa-cyclooctadecine-2,3,13, 14-tetraamine (167 mg, 0.40 mmol) was added, and the mixture was heated under reflux (at about 65 °C) for 10 h. The formed precipitate was collected by filtration, washed with methanol for several times and dried in vacuo to afford **1** as red-brown solid in about 60% yield. ^{1}H NMR (400 MHz, [D_6]DMSO, TMS) (ppm) 3.99 (s, 8 H), 4.32 (d, 8 H, J=29.2 Hz), 6.90 (s, 4 H), 7.01 (s, 4 H), 7.14 (s, 4 H), 7.31 (s, 4 H), 7.42 (d, 4 H, J=5.6 Hz), 7.93 (d, 4 H, J=7.4 Hz), 11.30 (s, 4 H); ESI-MS, m/z: 925.4 $[M+H]^+$, 947.4 $[M+Na]^+$; HRMS (ESI), m/z: 947.3276 $[M+Na]^+$, calcd for $C_{56}H_{44}N_8O_6$: 924.3384.

3. Results and discussion

3.1. Anion sensing

The sensing ability of **1** was first examined by interacting with some anions (F^-, $H_2PO_4^-$, AcO^-, ClO_4^-, NO_3^-, Cl^-, Br^-, I^- and



Scheme 1. The route for the synthesis of **1**.

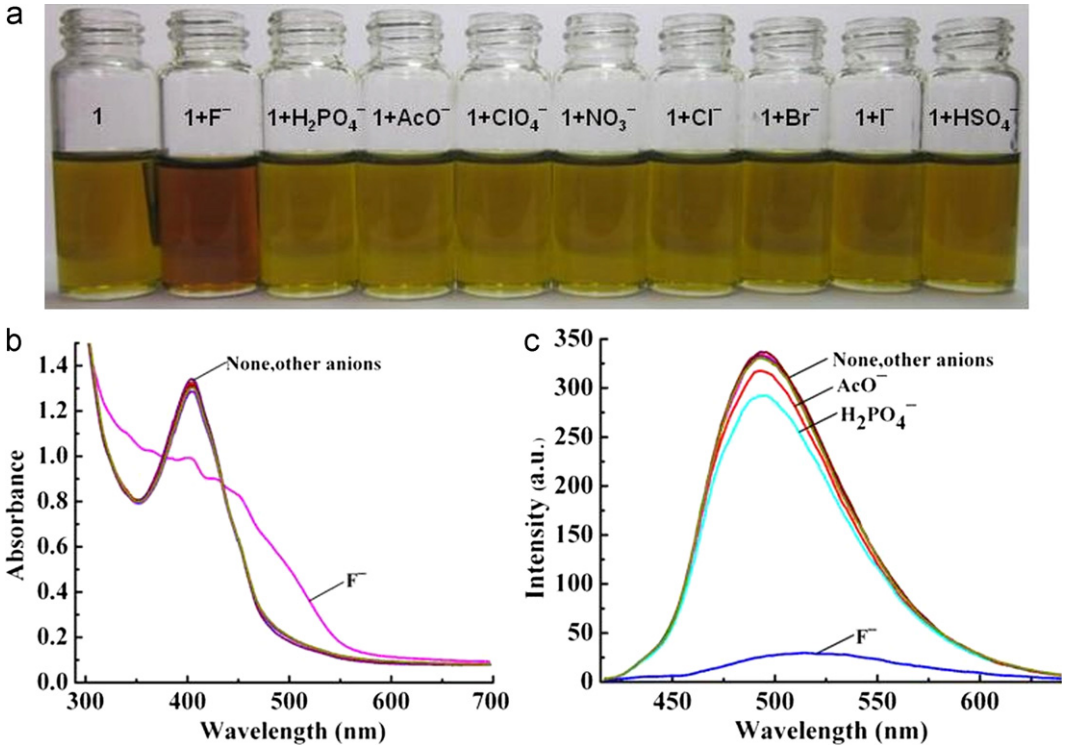


Fig. 1. (a) Color changes induced by the addition of anions (75 equiv.) to a DMSO solution of **1**. [1]=4×10^{-4} M. (b) The UV–vis spectra changes of receptor **1** in DMSO measured in DMSO upon addition of 75 equiv of respective anions (as n-bu_4N^+ salt). Other anions are $H_2PO_4^-$, AcO^-, ClO_4^-, NO_3^-, Cl^-, Br^-, I^- and HSO_4^-. (c) Fluorescence (λ_{ex}=390 nm) spectra changes of **1** measured in DMSO upon addition of 75 equiv of respective anions (as n-bu_4N^+ salt). Other anions are ClO_4^-, NO_3^-, Cl^-, Br^-, I^- and HSO_4^-. [1]=5×10^{-5} M.

HSO_4^-) as tetrabutylammonium salts (n-bu_4N^+) with UV–vis and emission spectra (see Fig. 1(a), (b) and Fig. 2). As illustrated in Fig. 1(a), the prominent color changes of **1** with F^- were observable by the naked eye whereas other anions did not show any obvious changes. The obvious changes in electronic spectra of **1** were shown in Fig. 1(b) and (c). Therefore, considering the high selectivity for F^- over other anions and the convenience without resorting to any spectrometer, **1** provides a great advantage for detecting F^-.

Then the interaction of **1** with F^- was further investigated by UV–vis absorption and emission spectra titrations (see Fig. 2). Upon addition of 75 equiv F^-, the intensity of the band at 403 nm reduced gradually accompanied with that at 350 nm increased, and a new absorption peak appeared at 490 nm. Moreover, the presence of two sharp isosbestic points at 376 nm and 435 nm implies that only two species coexist at the equilibrium point. Significant fluorescence quenching was accompanied by the fluorescent color changes on addition of 75 equiv F^-. The 1:2 binding stoichiometry was verified by a Job's plot analysis and the binding constant between **1** and F^- was determined from the emission spectra quenching at 490 nm, providing $Ka = 1.0 \times 10^6\ M^{-1}$ (error < 10%).

3.2. Cation sensing

Being bis-benzocrown ether entities, the ditopic receptor **1** exhibits selective interactions with K^+ as a potassium salt ($KClO_4$, KSCN, KBr, KI, KAcO and KH_2PO_4) to form **1**·K^+ complex. As expected, the addition of K^+ to a solution of **1** caused no color change, although a small change in emission intensity was observed (see Fig. 3). From the data, the binding constant for a 1:1 complex stoichiometry between **1** and K^+ was calculated to be $776\ M^{-1}$ from the emission spectra enhanced at 490 nm (error < 10%) [43–44].

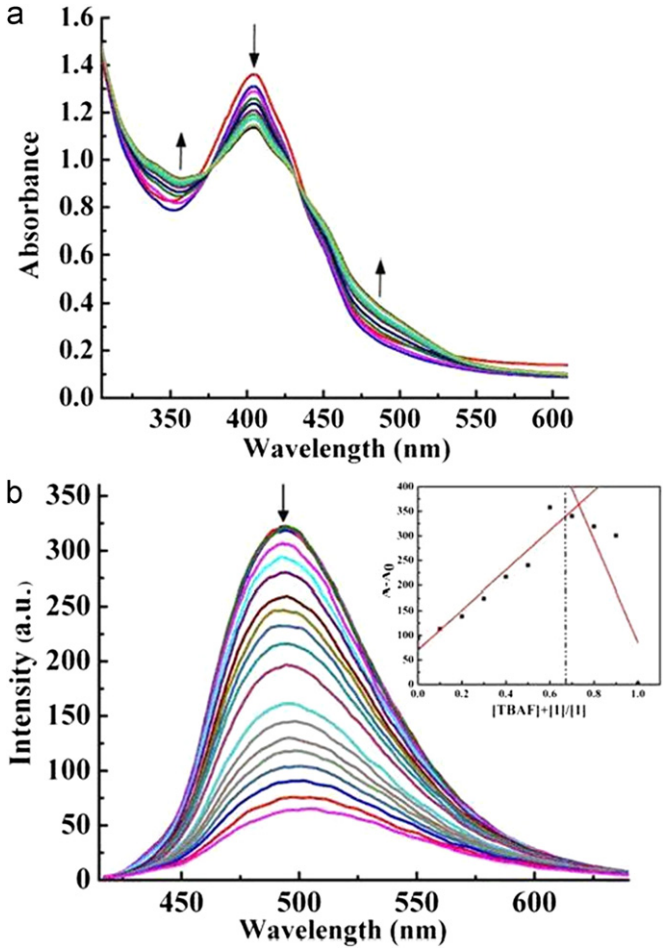


Fig. 2. (a) The UV–vis spectra changes of receptor **1** in DMSO after addition of F^- (as n-bu_4N^+ salt). (b) Fluorescence (λ_{ex}=390 nm) titration of **1** with F^- (as n-bu_4N^+ salt) in DMSO (F^- from 0 to 75 equiv.). [1]$= 5 \times 10^{-5}$ M. Inset: Job's plots for receptor **1** with F^-. [TBAF]+[Receptor]$= 1.5 \times 10^{-4}$ M.

3.3. UV–vis and fluorescent anion and cation titration studies

3.3.1. UV–vis and fluorescent F^- and K^+ titration studies

Fluoride was first chosen to examine ion pair interaction of the ditopic receptor between F^- and K^+, since receptor **1** was capable of forming complexation of **1**·K^+ as aforementioned. Gratifyingly, we observed an interesting chromogenic ON–OFF switching process. Upon addition of 75 equiv F^-, the solution of **1** turned from yellowish to brownish yellow (Fig. 1(a)). However, when 75 equiv of K^+ was titrated into a solution of **1**·F^- complex, chromogenic process was reversed; that is the brownish yellow induced by the presence of F^- was now changed to the original color (see Fig. 4).

However, when about 400 equiv of K^+ was titrated into a solution of **1**·F^- complex (see Fig. 5(a) and (b)), the absorption maximum at 490 nm disappeared in UV–vis spectra. In addition, the intensity of the emission peak increased remarkably and reached the original intensity of **1**·K^+ complex. Hence, the presence of K^+ significantly alters the F^- binding ability of **1** and the binding constant was determined from the emission spectra (see Fig. 5(c)) enhanced at 490 nm, providing $Ka = 1.1 \times 10^4\ M^{-1}$ (error < 10%) Fig. 6.

Another different titration process was performed. 75 equiv of F^- was titrated into a solution of **1**·K^+ complex, and the solution

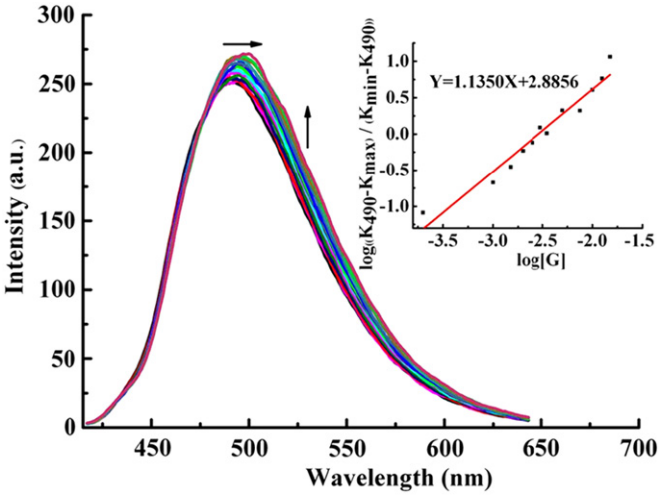


Fig. 3. Fluorescence (λ_{ex}=390 nm) titration of **1** with K^+ in DMSO. [1]$= 5 \times 10^{-5}$ M. Inset: fluorescence intensity at 490 nm of **1** versus increasing concentration of log [K^+]. The fluorescence response fits to a Hill coefficient of 1 (1.1350); it is consistent with the formation of a 1:1 stoichiometry for complex **1**·K^+ in solution.

Fig. 4. Color changes induced by the addition of K^+ (75 equiv.) to a **1**·anion (75 equiv.) complex. [1]$= 4 \times 10^{-4}$ M.

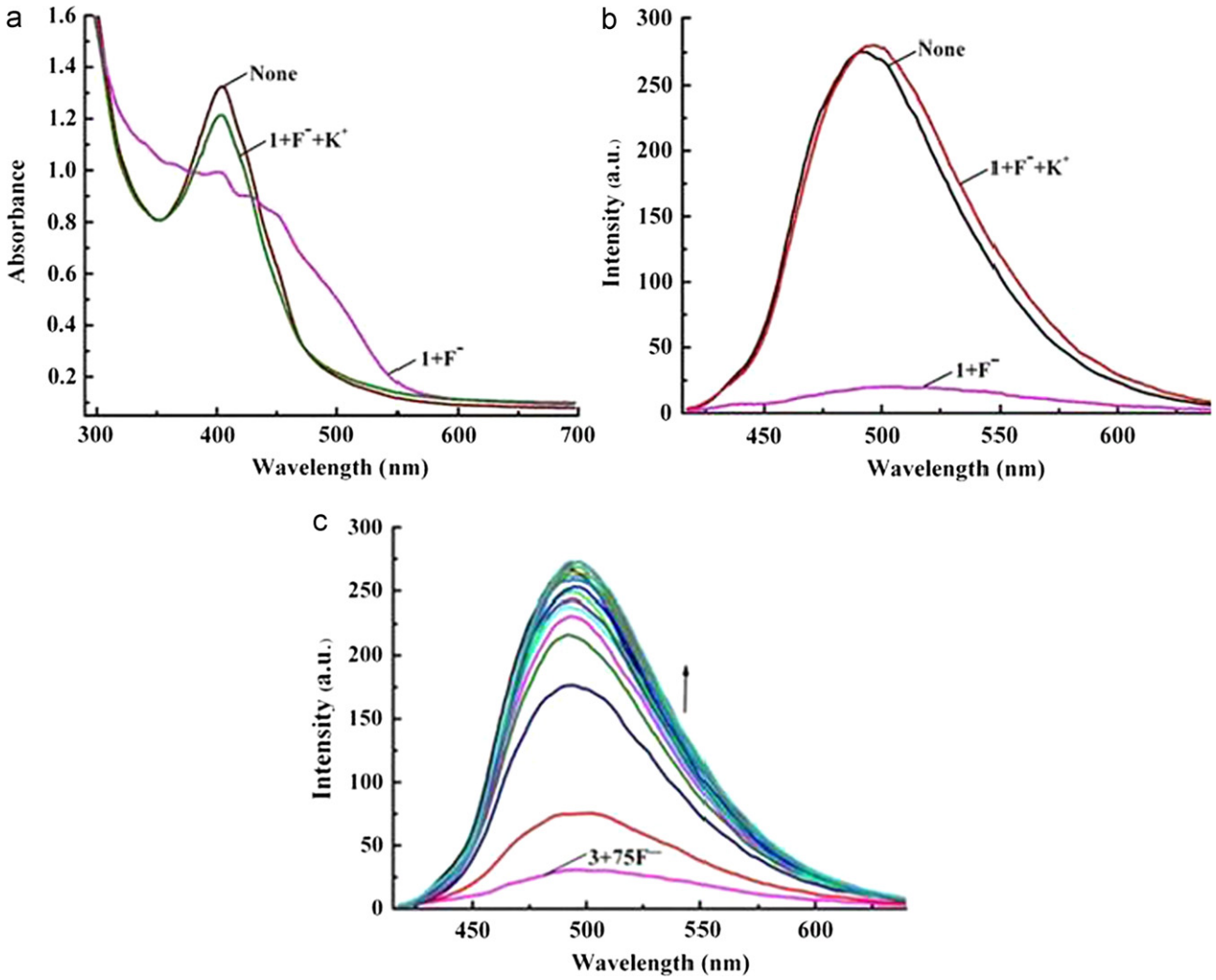


Fig. 5. (a) The UV–vis spectra changes of **1** in DMSO upon addition F^- (75 equiv.) and K^+ (400 equiv.). (b) the emission spectra of **1** upon addition F^- (75 equiv.) and K^+ (400 equiv.). (c) Fluorescence (λ_{ex}=390 nm) titration of [**1**$\cdot F^-$] with K^+ in DMSO (K^+ from 0 to 400 equiv.). [1]$=5\times10^{-5}$ M.

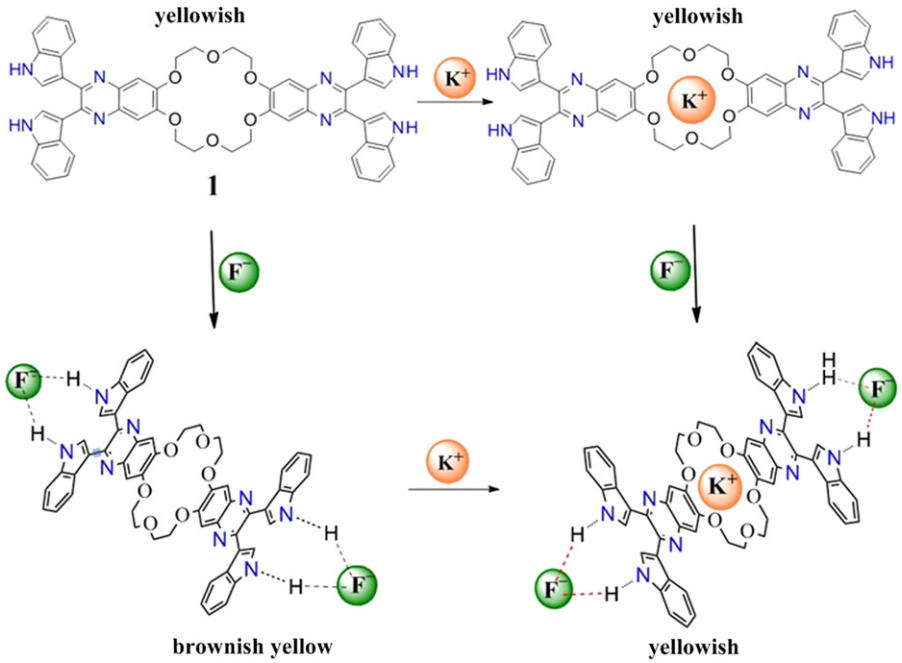


Fig. 6. Proposed binding modes for the complex of **1**, K^+ and F^-. Red dashed bond was shown weaker than black dashed bond. (For interpretation of the references to color in this figure legend, the reader is reffered to the web version of this article.)

remains yellowish and only small changes on the UV–vis absorption and emission spectra were observed. Therefore, K^+ appears to inhibit the chromogenic response of the system to F^-. But the fluorescence quenching continued to decrease even after more than 400 equiv of F^- was added (see Fig. 7). This suggests that K^+ is complexed faster by the bis-benzocrown ether than F^- by the biindole-NH of **1** (see Fig. 6).

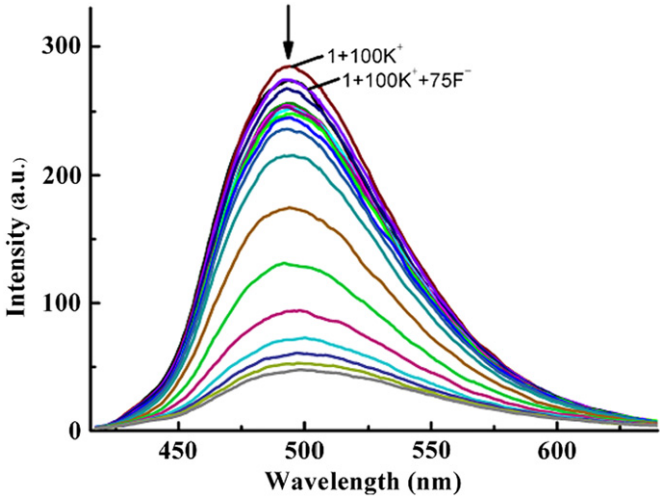


Fig. 7. The emission spectra of **1** upon the addition of K^+ (100 equiv.) and F^- (400 equiv.). [1] = 5×10^{-5} M.

3.3.2. *UV–vis and fluorescent HSO_4^- and K^+ titration studies*

The presence of K^+ significantly alters the anion binding ability of receptor **1**. HSO_4^-, a weak hydrogen bond acceptor showed no affinity with **1**. However, titration experiments of **1** with HSO_4^- in the presence of K^+ showed enhancement of affinities providing $Ka = 1.2 \times 10^3\ M^{-1}$. While no changes of the UV–vis spectrum of **1** were observed upon addition of HSO_4^- as a tetrabutylammonium salts (n-bu_4N^+) in the presence of K^+

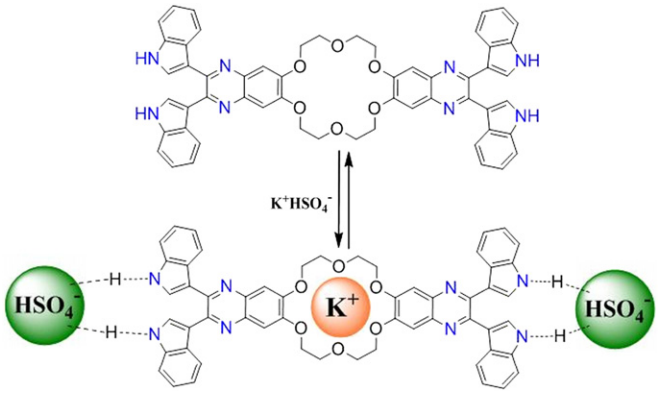


Fig. 9. Proposed binding mode for the complex of **1**, K^+ and HSO_4^-.

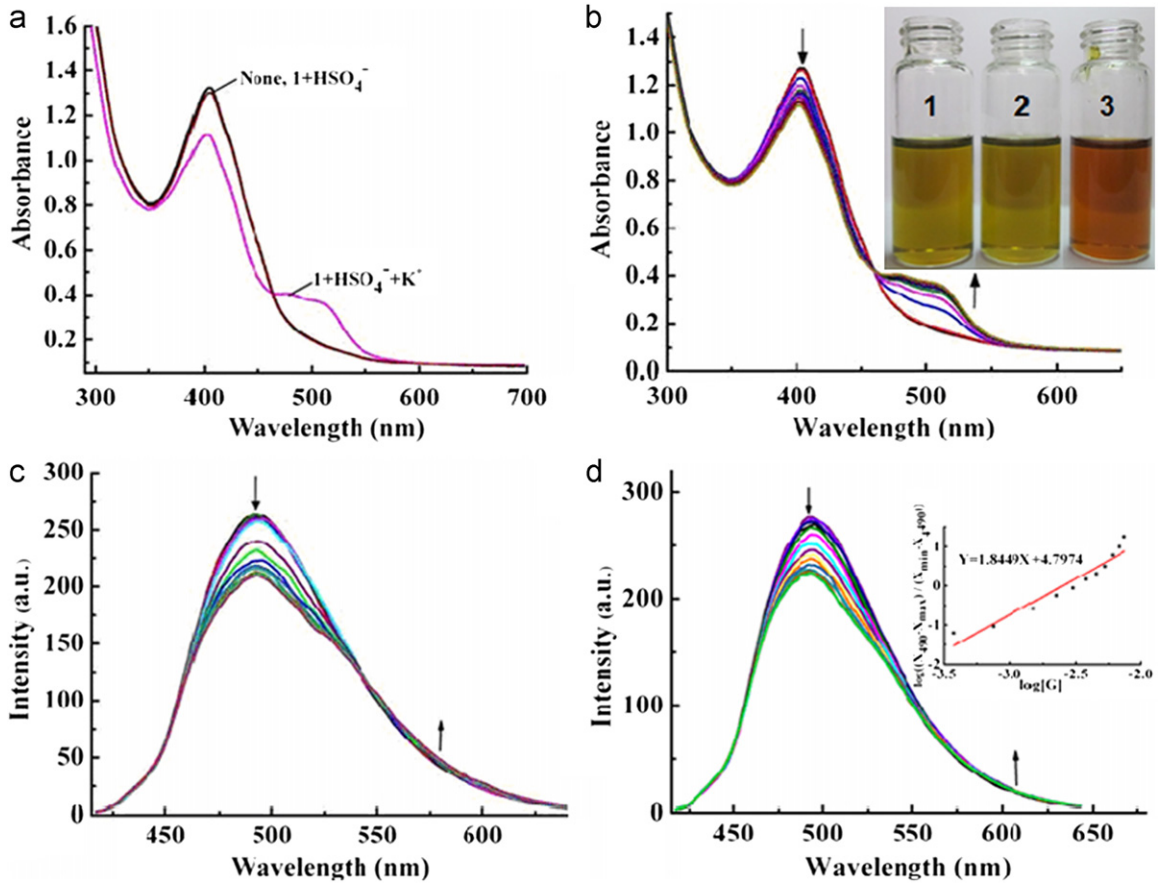


Fig. 8. (a) Distribution diagram of the species for receptor **1** (5×10^{-5} M) titrated with 75 equiv of HSO_4^- and 800 equiv of K^+. (b) The UV–vis spectra changes of receptor **1**·HSO_4^- in DMSO (5×10^{-5} M) after addition of 0–800 equiv of K^+. Inset: 1 (the color changes of receptor **1**), 2 (**1** and 75 equiv. of HSO_4^-) and 3 (100 equiv. of K^+ titration of **1**·HSO_4^-). (c) Fluorescence (λ_{ex} = 390 nm) titration of **1**·HSO_4^- complex with K^+ (800 equiv.) in DMSO. (d) Fluorescence intensity at 490 nm of **1**·K^+ versus increasing concentration of log [HSO_4^-]. The fluorescence response fits to a Hill coefficient of 2 (1.8449); it is consistent with the formation of a 1:2 stoichiometry for the **1**·K^+–HSO_4^- complex in solution.

(see Fig. 8(a)). Fig. 8 displays the visual aspects of HSO_4^- recognition and sensing in the presence of K^+. Upon addition of 75 equiv HSO_4^- to the solution of receptor **1**, there was no change in color. And subsequent addition of 100 equiv of K^+ to this solution causes the initial yellowish substantially fades to an orange (see Fig. 8(b) (inset)). Interestingly, upon addition of HSO_4^- to the solution of the **1**$\cdot K^+$ complex, the same results was observed. This phenomenon may be caused by sequestration of the HSO_4^- from the ditopic receptor **1** to form a more stable ion-pair in solution (see Fig. 9). The interaction of cation with the hydrogen sulfate lone pair promotes electron density transfer from the **1**$\cdot$ anion complex to the cation, which alters the intensity of the UV–vis spectra. In addition, a corresponding correlation between emission ratiometric response of **1** at 565 and 490 nm (I_{565}/I_{490}) demonstrated that **1** can serve as a ratiometric fluorescent receptor for HSO_4^- upon the addition of K^+ (see Fig. 8(c)). These results indicate that **1** has an excellent selectivity in the presence of K^+ for HSO_4^- over other anions.

3.4. ^{1}HNMR titration studies

3.4.1. ^{1}HNMR titration studies of F^- and K^+

NMR studies in DMSO at 25 °C were carried out in attempts to understand the binding mode of the complexes. When 30 equiv of K^+ was added to the solution of **1**, chemical shifts of protons on the bis-benzocrown ether, in particular those for the OCH_2CH_2O protons, shifted downfield slightly from 4.35 to 4.49 and from 3.98 to 4.08, for cation was involved in complexation by the bis-benzocrown ether unit. Interestingly, upon addition of 15 equiv of F^- to the solution of the **1**$\cdot K^+$ complex, the indole-NH protons signal shifted downfield slightly from 11.30 to 11.39, which indicated a weak hydrogen bonding interaction with F^- (see Fig. 10(a)).

On the other hand, when F^- was first added, the resonances corresponding to the indole-NH protons of **1** disappeared due to the deprotonation and formation of the FHF^- ion at $\delta = 16.11$ ppm (see Fig. 10(b)), similar to the previously reported phenomena [45–48]. However, when 30 equiv of K^+ were added to the above solution, the indole-NH proton signals reverted to the positions observed originally. And the bis-benzocrown proton signals reverted to the positions observed in the **1**$\cdot K^+$ complex. It is clear that from these studies the bound K^+ should interact with the bis-benzocrown ether of the ditopic receptor, which influences the electron density of **1**, so the hydrogen bonding interaction between **1** and F^- is weakened, similar to the previously reported phenomena [49–50].

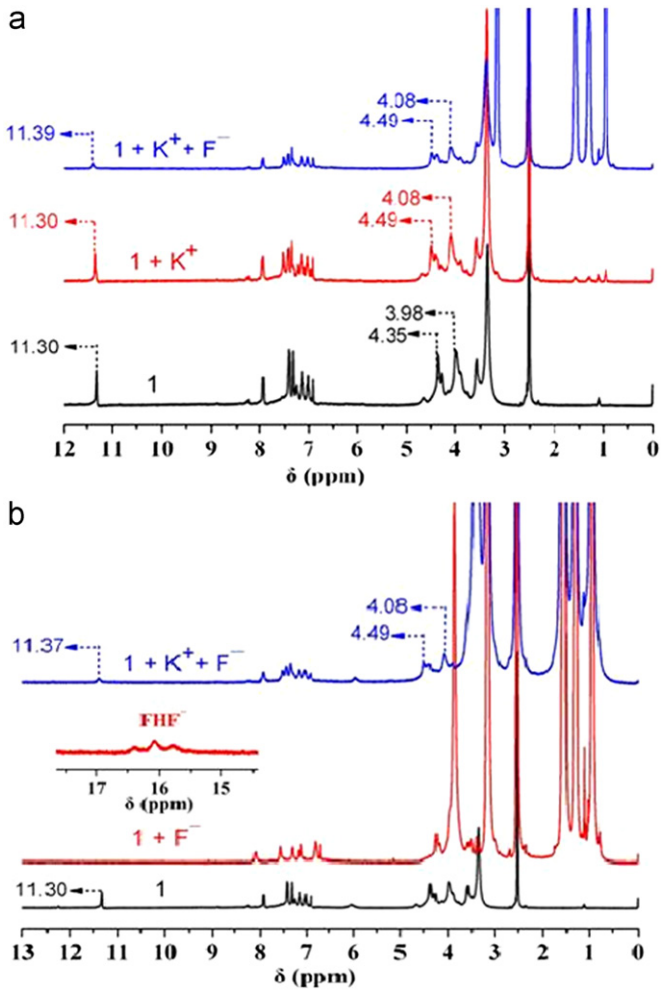


Fig. 10. (a) ^{1}H NMR (400 MHz) spectra of receptor **1** (1×10^{-2} M) in [D_6]DMSO with the addition of K^+ (30 equiv.) first and adding TBAF (15 equiv.). (b) ^{1}H NMR (400 MHz) spectra of receptor **1** (1×10^{-2} M) in [D_6]DMSO with the addition of TBAF (15 equiv.) first and then adding of K^+ (30 equiv.).

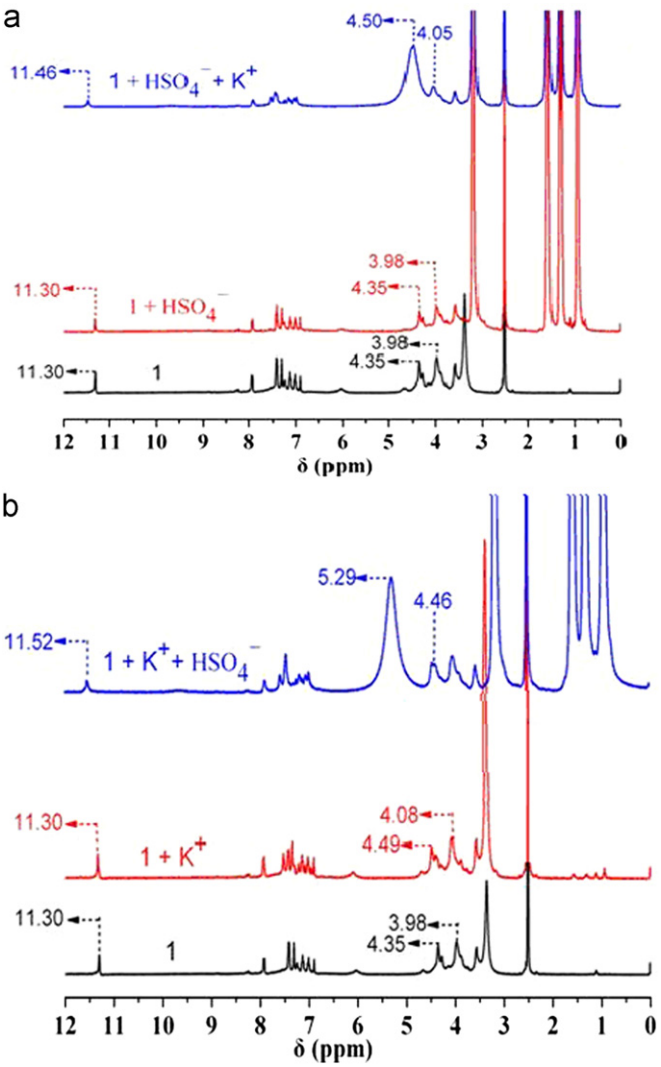


Fig. 11. (a) ^{1}H NMR (400 MHz) spectra of receptor **1** (1×10^{-2} M) in [D_6]DMSO with addition of $TBAHSO_4$ (15 equiv.) first and adding K^+ (30 equiv.). (b) ^{1}H NMR (400 MHz) spectra of receptor **1** (1×10^{-2} M) in [D_6]DMSO with addition of K^+ (30 equiv.) first and adding $TBAHSO_4$ (15 equiv.).

3.4.2. ^{1}HNMR titration studies of HSO_4^- and K^+

In order to gain more insight into the cooperative enhancement of cation recognition by the presence of an anion, ^{1}H NMR titration studies of **1** with progressive addition of HSO_4^- and K^+ were carried out in [D_6]DMSO (see Fig. 11(a)). With the addition of 15 equiv HSO_4^- to **1**, no proton signal has changed, which indicated that HSO_4^- had little effect on the recognition process. Upon addition of 30 equiv of K^+ to the solution of the **1**$\cdot HSO_4^-$ complex, the indole-NH proton signals shifted downfield to 11.46, indicating a weak hydrogen bonding interaction with HSO_4^- due to the presence of K^+. And OCH_2CH_2O protons downfield by 0.15 and 0.07 ppm, respectively, suggesting that the bis-benzocrown ether is involved in complexation with K^+. Similarly, when HSO_4^- was added to the **1**$\cdot K^+$ complex, the bis-benzocrown proton signals shifted downfield from 4.49 to 5.29 and from 4.08 to 4.46 (see Fig. 11(b)). These changes definitely imply a conformational change in the bis-benzocrown ether unit on cation-induced anion binding, and may indicate an enhancement of cation binding strength as the anion is added [51]. These observations reinforce the suggestion of cooperative binding behavior obtained from the UV–vis spectroscopic experiments.

4. Conclusion

We have successfully prepared a new ion pair receptor **1**, which can function as a chromogenic molecular switch only occurs in the presence of F^- but in the absence of K^+. In addition, in the presence of K^+, the affinities of **1** for HSO_4^- increase, and is accompanied by specific color changes of solutions and ratiometric manner of receptor **1** for detecting HSO_4^-. Hence, **1** provides an advantage for selective detecting HSO_4^- in the presence of K^+.

Acknowledgments

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