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Sulfonamide and urea-based anions chemosensors

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ABSTRACT

The detection of anions has attracted considerable interest because of their importance in various physiological processes. In this study, two sulfonamide and urea-based compounds (**1a** and **1b**) were successfully developed and their spectroscopic and anion recognition properties were fully investigated. These results showed that: (1) compounds showed high selectivity towards cyanide and fluoride ions in CH_3CN; (2) compounds only exhibited a large change in fluorescence in the presence of cyanide ions in $CH_3CN–H_2O$ (95:5, v/v); and (3) compound **1b** could act as a gel in dimethyl sulfoxide that transforms into a homogeneous solution upon exposure to cyanide ions. This research suggests that sulfonamide and urea can act as hydrogen-bond donors and provides an alternative approach to the design of novel anion chemosensors.

1. Introduction

Biologically and environmentally important anions have played a fundamental role in a wide range of organic and inorganic systems, and their application in sensing and anion transport has gained considerable interest [1–21]. In addition to the development of anion chemosensors, the binding between the chemosensor and the anion involves three main types of interaction, which include: 1) electrostatic interactions, 2) reaction-based sensors, 3) hydrogen bonding and 4) metal–ligand interactions [22–24]. Recently, hydrogen bonding has become one of the most frequently used interactions due to their relatively high energy, the significant availability of H-bond donors and their strong and selective binding with anions [25–27]. Among the various anion chemosensors, amides, hydrazides, pyrroles, ureas and thioureas are often used as hydrogen bond donors with both high affinity and selectivity [28–30]. Sulfonamides, which are similar to amides, have always served as a cation and thiol amino acid chemosensor [31–34]. Currently, a research towards anion chemosensors combining sulfonamides has been reported. Therefore, a sulfonamide can also act as a hydrogen-bond donor and form intramolecular or intermolecular hydrogen bonds. It is therefore important to design anion chemosensors with novel topological structures.

In this work, two anion chemosensors (**1a** and **1b**), based on both sulfonamide and urea were successfully developed and their spectroscopic and anion recognition properties fully investigated. Both of them show highly selective chemical signaling behavior towards cyanide and fluoride ions in CH_3CN. In particular, both compounds only exhibited a large change in fluorescence in the presence of cyanide ions in $CH_3CN–H_2O$ (95:5, v/v). The signaling process was confirmed using fluorescence spectroscopy and the limit of detection for compounds **1a** and **1b** was 12 nM and 4 nM, respectively. Significantly, compound **1b** could act as a gel in dimethyl sulfoxide that transforms into a homogeneous solution upon exposure to cyanide ions. Density functional theory (DFT) calculations and NMR titration studies suggest that both compounds act as cyanide ion fluorescence chemosensors via hydrogen bonding. Herein, we provide an alternative approach to the design of novel anion chemosensors with high sensitivity and selectivity.

2. Materials and methods

2.1. Experimental

All manipulations were carried out under an argon atmosphere using standard Schlenk techniques, unless otherwise stated. Tetrahydrofuran was dried with Na then distilled under vacuum. All

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0143-7208/

reagents and starting materials were obtained from commercial suppliers and were used without further purification. All anions for binding experiments used tetrabutylammonium salts as sources. Anions in CH_3CN were obtained by dissolution of the anions in CH_3CN. Time course for the signaling of cyanide and fluoride ions by compounds **1a** and **1b** was followed by monitoring the changes in fluorescence intensity of the solutions at 531 nm. The concentrations of the probe **1a** or **1b** and cyanide or fluoride ion were 10 μM and 10 mM, respectively, in DMSO or a mixture of DMSO and water solution (95:5, v/v). Column chromatography was used on silica gel (200–300 mesh). NMR spectra were analysed using an American Varian Mercury Plus 400 spectrometer (400 MHz) and their chemical shifts are relative to TMS. Elemental analyses (C, H, N) were performed by the Microanalytical Services, College of Chemistry, CCNU. Electrospray (EI) mass spectra were carried on Firmigan Trace. UV–Vis spectra were analysed using a U-3310 UV Spectrophotometer. Fluorescence spectra were analysed using a Fluoromax-P luminescence spectrometer (HORIBA JOBIN YVON INC.).

2.2. Synthesis

2.2.1. Synthesis of all new compounds

*2.2.1.1. Synthesis of **3**, **4a**, **4b**, **5a** and **5b**.* Compounds **3**, **4a**, **4b**, **5a** and **5b** were prepared by literature methods [35].

*2.2.1.2. Synthesis of **6a**, **6b**, **7a** and **7b**.* Compounds **6a**, **6b**, **7a** and **7b** were prepared by literature methods [36].

*2.2.1.3. Synthesis of **8a**.* A solution of benzene-1, 2-diamine (1.4 mmol) in acetone (50 mL) was cooled in an ice bath under an argon atmosphere and **7a** (0.53 mmol) in acetone (20 mL) was added dropwisely to the above solution. This solution was stirred for 2 h at room temperature. The formed precipitate was collected and the crude product was washed with ice acetone, the dried solid was recrystallized from methylene chloride to give a white solid **8a**. Yield: 42 mg, 25%. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm = 3.60 (s, 3H, CH_3), 3.74 (s, 6H, CH_3), 4.77 (s, 2H, NH_2), 6.57 (d, J = 8 Hz, 1H, Ar–H), 6.74 (d, J = 8 Hz, 1H, Ar–H), 6.80 (s, 2H, Ar–H), 6.83 (d, J = 4 Hz, 1H, Ar–H), 7.32 (d, J = 4 Hz, 1H, Ar–H), 7.69 (s, 1H, urea N–H), 8.73 (s, 1H, urea N–H). ^{13}C NMR (100 MHz, DMSO): δ ppm = 55.69, 60.21, 95.75, 115.96, 116.89, 124.01, 124.58, 124.69, 132.23, 136.34, 141.03, 152.91, 153.28. Anal. calcd for $C_{16}H_{19}N_3O_4$: C, 60.56; H, 6.03; N, 13.24. Found: C, 60.41; H, 5.99; N, 13.20. EI MS m/z = 317.26 [M]; calculated exact mass = 317.14.

*2.2.1.4. Synthesis of **8b**.* A solution of benzene-1, 2-diamine (1.4 mmol) in acetone (50 mL) was cooled in an ice bath under an argon atmosphere and **7b** (0.53 mmol) in acetone (20 mL) was added dropwisely to the above solution. This solution was stirred for 2 h at room temperature. The formed precipitate was collected and the crude product was washed with ice acetone, the dried solid was recrystallized from methylene chloride to give a white solid **8b**. Yield: 65 mg, 20%. ^{1}H NMR (400 MHz, DMSO-d_6): δ ppm = 0.84 (d, J = 8 Hz, 9H, CH_3), 1.25 (s, 24H, CH_2), 1.41 (d, J = 4 Hz, 6H, CH_2), 1.60 (t, J = 4 Hz, 2H, CH_2), 1.69 (t, J = 4 Hz, 4H, CH_2), 3.76 (t, J = 8 Hz, 2H, CH_2), 3.87 (t, J = 8 Hz, 4H, CH_2), 4.73 (s, 2H, NH_2), 6.56 (t, J = 8 Hz, 1H, Ar–H), 6.72 (d, J = 4 Hz, 3H, Ar–H), 6.83 (d, J = 8 Hz, 1H, Ar–H), 7.30 (d, J = 8 Hz, 1H, Ar–H), 7.62 (s, 1H, urea N–H), 8.60 (s 1H, urea N–H). ^{13}C NMR (100 MHz, DMSO): δ ppm = 13.87, 22.16, 25.68, 28.83, 29.85, 31.30, 31.38, 55.69, 68.10, 72.40, 96.80, 115.89, 116.79, 123.78, 124.36, 124.73, 131.98, 135.87, 140.85, 152.43, 153.14. Anal. calcd for $C_{37}H_{61}N_3O_4$: C, 72.63; H, 10.05; N, 6.87. Found: C, 72.42; H, 9.99; N, 6.85. EI MS m/z = 611.71 [M]; calculated exact mass = 611.47.

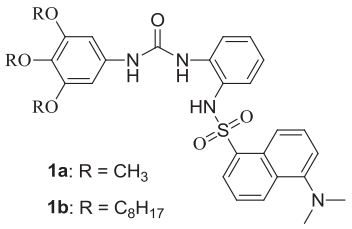
*2.2.1.5. Synthesis of **1a**.* A solution of **8a** (1 mmol) and Et_3N (1.5 mmol) in THF (50 mL) was cooled in an ice bath under an argon atmosphere and 5-(dimethylamino) naphthalene-1-sulfonyl chloride was added dropwisely to the above solution. After the mixture was stirred at room temperature, the mixture was diluted with water and extracted with EtOAc. The organic layer was washed with 2 N HCl, water, saturated $NaHCO_3$ solution and brine. Then dried over sodium sulfate, upon removed of solvent under reduced pressure and purified on a silica gel column using dichloromethane/methanol (60:1) as the eluent to obtain the target compound as a yellow solid in a yield of 60%, 307 mg. ^{1}H NMR (400 MHz, $CDCl_3$): δ ppm = 2.85 (s, 6H, CH_3), 3.76 (s, 6H, CH_3), 3.79 (s, 3H, CH_3), 6.57–6.63 (m, 4H, Ar–H), 6.75 (t, J = 4 Hz, 1H, Ar–H), 7.03–7.14 (m, 3H, Ar–H), 7.37–7.41 (m, 2H, Ar–H), 7.48–7.56 (m, 2H, Ar–H), 8.04 (d, J = 8 Hz, 1H, urea N–H), 8.39 (d, J = 8 Hz, 1H, urea N–H), 8.51 (d, J = 8 Hz, 1H, amide N–H). ^{13}C NMR (100 MHz, $CDCl_3$): δ ppm = 45.17, 55.78, 60.82, 97.24, 115.03, 118.26, 122.72, 122.88, 124.09, 126.76, 127.24, 127.72, 128.59, 129.43, 129.96, 130.86, 133.31, 133.90, 134.43, 134.63, 151.77, 152.92, 153.56. Anal. calcd for $C_{28}H_{30}N_4O_6S$: C, 61.08; H, 5.49; N, 10.18. Found: C, 61.00; H, 5.19; N, 10.28. EI MS m/z = 550.43 [M]; calculated exact mass = 550.19.

*2.2.1.6. Synthesis of **1b**.* A solution of **8b** (1 mmol) and Et_3N (1.5 mmol) in THF (50 mL) was cooled in an ice bath under an argon atmosphere and 5-(dimethylamino) naphthalene-1-sulfonyl chloride was added dropwisely to the above solution. After the mixture was stirred at room temperature, the mixture was diluted with water and extracted with EtOAc. The organic layer was washed with 2 N HCl, water, saturated $NaHCO_3$ solution and brine. Then dried over sodium sulfate, upon removed of solvent under reduced pressure and purified on a silica gel column using dichloromethane/methanol (90:1) as the eluent to obtain the target compound as a pale yellow solid in a yield of 65%, 511 mg. ^{1}H NMR (400 MHz, $CDCl_3$): δ ppm = 0.88 (t, J = 4 Hz, 12H, CH_2CH_3), 1.44 (s, 21H, CH_2CH_3), 1.71–1.77 (m, 12H, CH_2CH_3), 2.84 (s, 6H, CH_3), 3.89 (t, J = 4 Hz, 4H, CH_2), 3.96 (t, J = 4 Hz, 2H, CH_2), 6.55 (t, J = 8 Hz, 2H, Ar–H), 6.68 (d, J = 8 Hz, 1H, Ar–H), 6.68 (t, J = 8 Hz, 1H, Ar–H), 7.03–7.09 (m, 2H, Ar–H), 7.11–7.13 (m, 1H, Ar–H), 7.21 (s, 1H, Ar–H), 7.36–7.42 (m, 2H, Ar–H), 7.48–7.55 (m, 2H, Ar–H), 8.02 (d, J = 4 Hz, 1H, urea N–H), 8.38 (d, J = 8 Hz, 1H, urea N–H), 8.48 (d, J = 8 Hz, 1H, amide N–H). ^{13}C NMR (100 MHz, $CDCl_3$): δ ppm = 14.07, 22.64, 26.09, 29.36, 30.25, 31.81, 45.27, 68.91, 73.50, 98.80, 114.90, 118.47, 122.64, 122.81, 122.99,124.04, 126.82, 127.24, 127.72, 128.65, 129.55, 130.14, 130.79, 133.89, 134.06, 134.51, 151.81, 153.12, 153.50. Anal. calcd for $C_{49}H_{72}N_4O_6S$: C, 69.63; H, 8.59; N, 6.63. Found: C, 69.35; H, 8.39; N, 6.60. EI MS m/z = 844.97 [M]; calculated exact mass = 844.52.

3. Results and discussion

3.1. Synthesis

Sulfonamides, which are similar to amides, serve as a hydrogen-bond donor and can easily form intramolecular or intermolecular hydrogen bonds. A dansyl unit was chosen as the fluorophore because of its desirable spectroscopic properties as well as being the smallest available fluorophore. Therefore, the signaling process of the sulfonamide fluorescent dye could be confirmed by fluorescence spectroscopy. Hence, compounds **1a** and **1b** bearing sulfonamide and urea functionality were synthesized as shown in Scheme 1. The syntheses of compounds **1a** and **1b** are outlined in Scheme 2. 5-Isocyanato-1, 2, 3-trimethoxybenzene **7a** was prepared using a Curtius rearrangement reaction [35]. The intermediate 3, 4, 5-trimethoxybenzoyl azide was unstable and easily decomposed into its corresponding nitrene followed by rapid

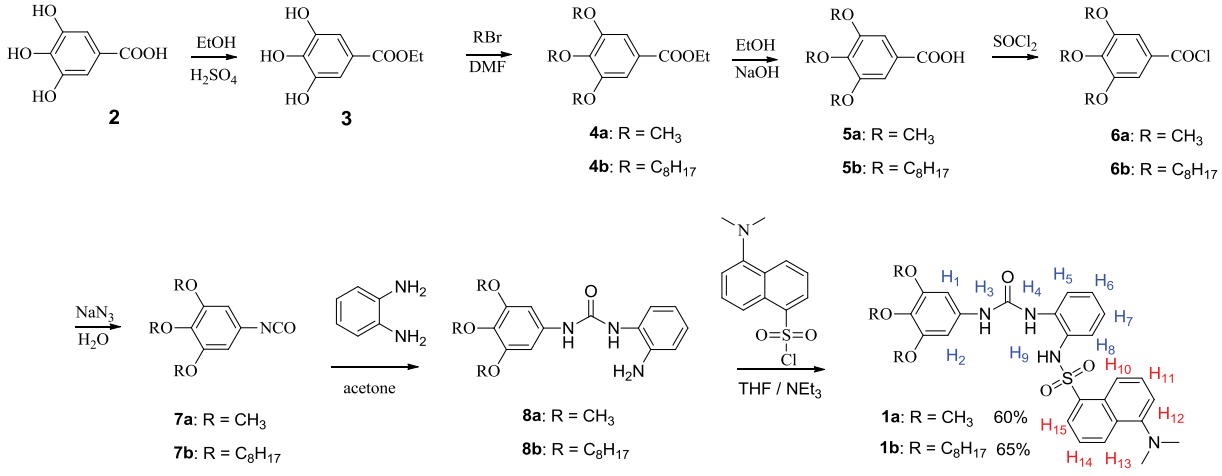


Scheme 1. The structures of probe **1a** and **1b**.

migration of the aryl group in the nitrene to produce the desired isocyanate product **7a**. Compound **8a** was synthesized using a procedure based on a previously described method [27]. Isocyanatobenzene **7a** in acetone was added dropwise to an acetone solution containing excess o-phenylenediamine and the resulting solution was stirred at room temperature for 2 h. The precipitate formed was collected, dried and recrystallized from methanol to give compound **8a** as a white solid in 60% yield. Probe **1a** bearing sulfonamide and urea functionality was synthesized using a previously described method [37]. Dansyl chloride in THF was added dropwise to a solution of **8a** in the THF and NEt_3 and the resulting solution stirred at room temperature for 24 h. The precipitate formed was collected and purified using column chromatography on a silica gel column to obtain the target compound as a yellow solid in 70% yield. The synthesis procedure of **1b** was similar to that used to prepare **1a** in an overall yield of 68%. All new compounds were characterized by ^1H NMR and ^{13}C NMR spectroscopy, EI mass spectroscopy and elemental analysis.

3.2. Optical responses according to different solvent systems

With probes **1a** and **1b** in hand, we first investigated their spectroscopic behavior in CH_3CN upon addition different anions including $CH_3CO_2^-$, F^-, Cl^-, CN^-, NO_3^-, Br^-, I^-, $H_2PO_4^-$, ClO_4^- and HSO_4^-. No obvious colorimetric change was observed by the naked eye when the anions were added, as shown in Fig. S1. Subsequently, we attempted to study their fluorescence properties. A selective change in fluorescence was observed for compounds **1a** and **1b** in the presence of F^- and CN^- ions upon irradiation at an excitation



Scheme 2. The synthesis of probe **1a** and **1b**.

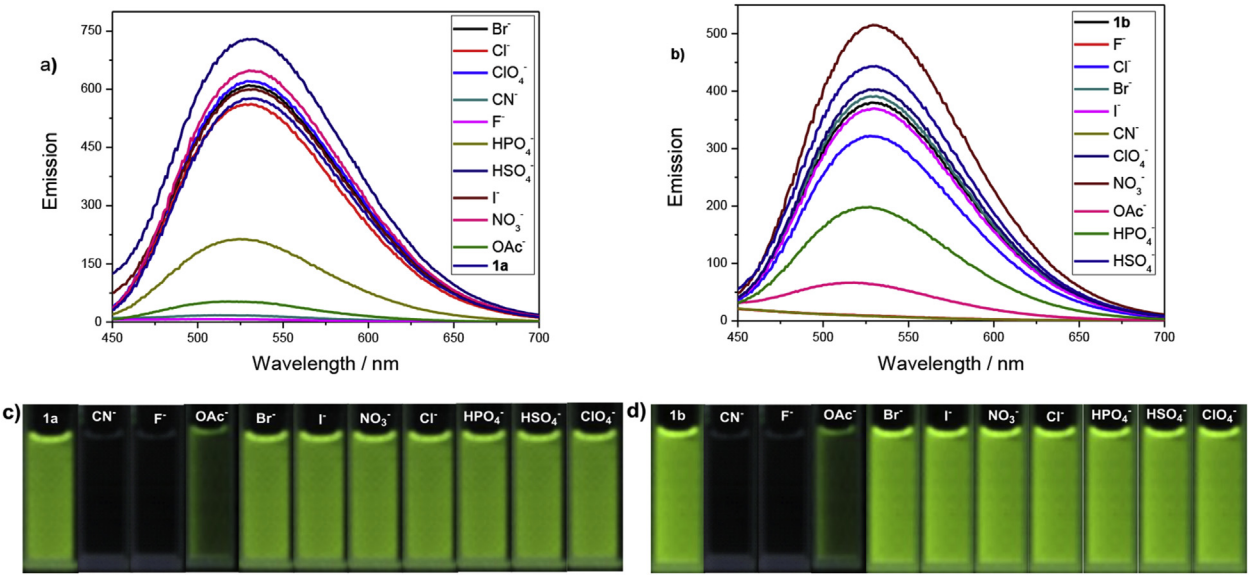


Fig. 1. (a), (b) Fluorescence changes and (c), (d) Photograph of the Fluorescence color changes of **1a**, **1b** (10 μm) upon the addition of different anions (10 eq) in CH_3CN (λ = 380 nm, Slit: 5 nm/5 nm).

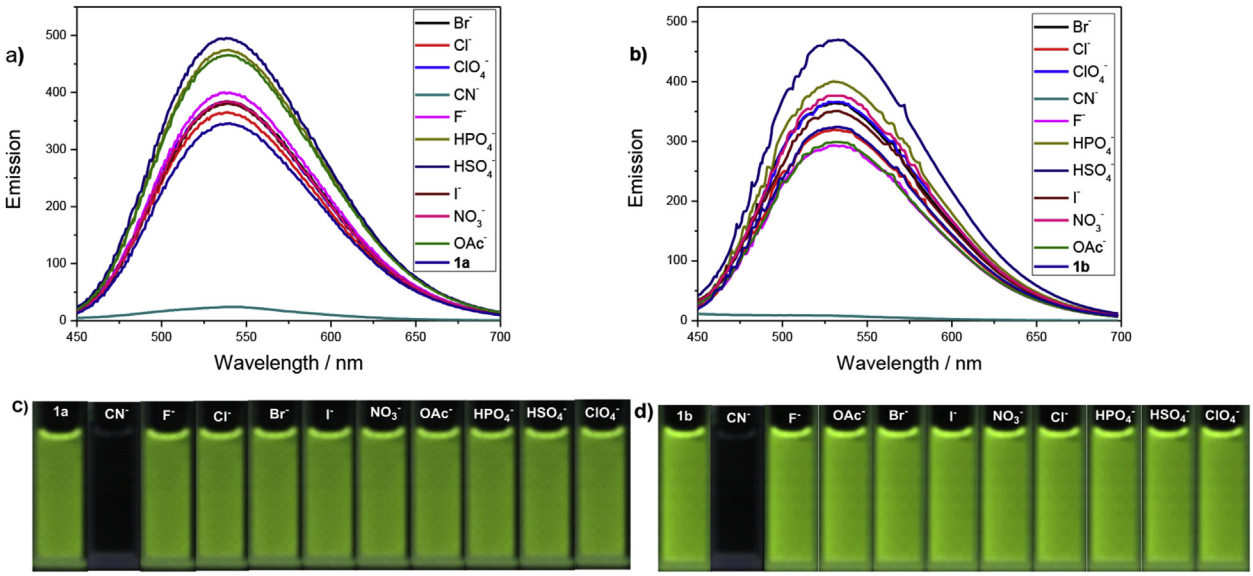


Fig. 2. (a), (b) Fluorescence changes and (c), (d) Photograph of the Fluorescence color changes of **1a**, **1b** (10 μm) upon the addition of different anions (10 eq) in CH_3CN–H_2O (95:5, v/v) (λ = 380 nm, Slit: 5 nm/5 nm).

wavelength of 380 nm. As shown in Fig. 1 and Fig. S2, when an excess of F^- and CN^- ions (10 eq) was added to **1a** and **1b**, obvious fluorescence quenching was observed. Photographs showing the changes in fluorescence color for **1a** and **1b** were in good agreement with the spectroscopic changes. These results suggest that compounds **1a** and **1b** show highly selective chemical signaling behavior towards cyanide and fluoride ions in CH_3CN.

When the solvent system was changed slightly upon the addition of 5% water, the changes in fluorescence were dramatically different for the selectivity of **1a** and **1b** towards F^- and CN^- ions. As presented in Fig. 2 and Fig. S3, the significant enhancement in fluorescence for F^- ions was observed, which could be attributed to the favourable solvation effect of F^- ions with water. In other words, obvious fluorescence quenching was observed with only CN^- ions.

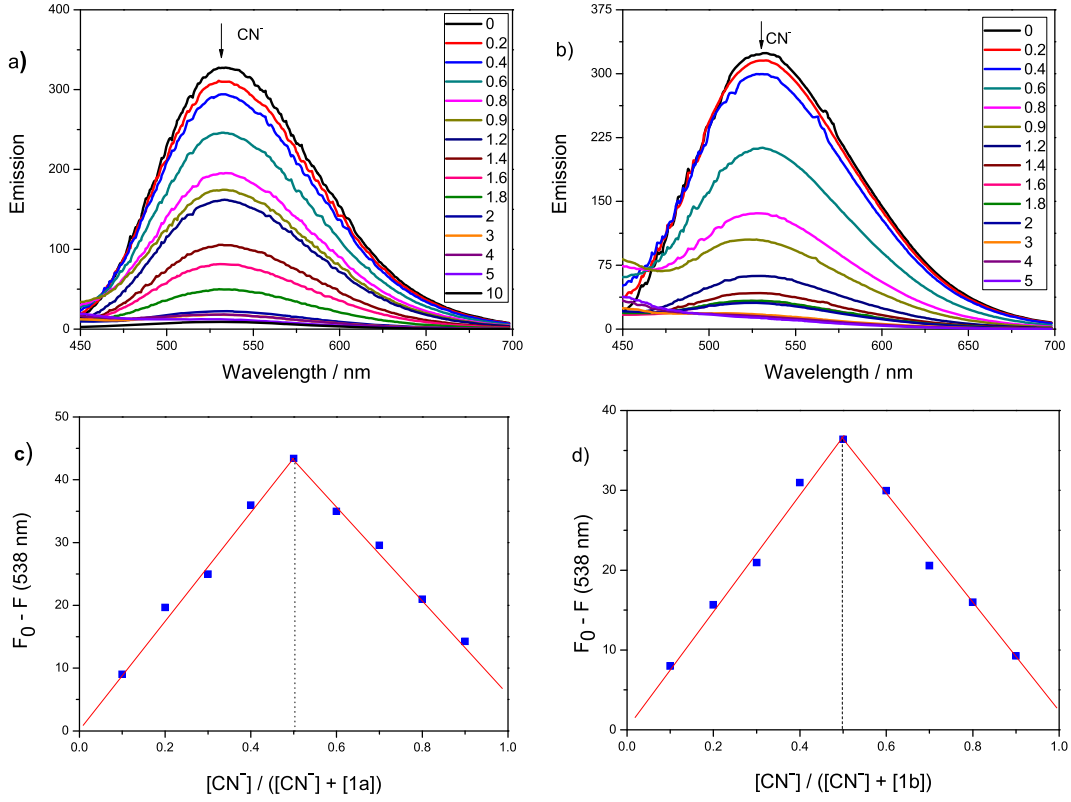


Fig. 3. (a), (b) Fluorescence titrations of **1a** and **1b** with different amounts of CN^- in CH_3CN–H_2O (95:5, v/v); (c), (d) Job's plot of **1a** and **1b** with CN^- ([probe] + [CN^-] = 10 μm) in CH_3CN–H_2O (95:5, v/v) (λ = 380 nm, Slit: 5 nm/5 nm).

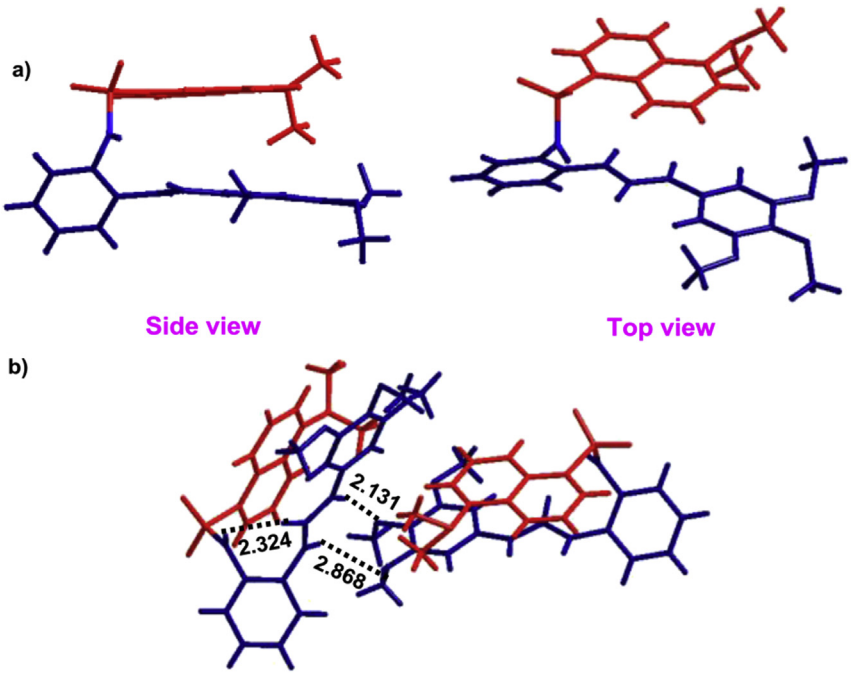


Fig. 4. (a) Single crystal structure of **1a**; (b) The H-bond of intermolecular and intramolecular in complex **1a**.

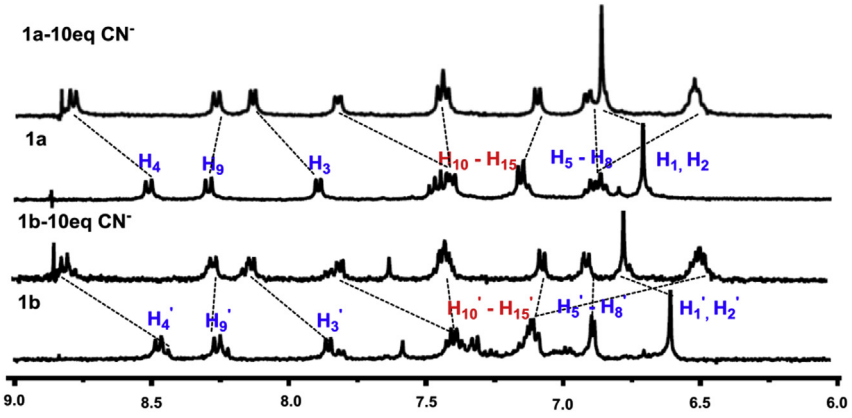


Fig. 5. ^{1}H NMR spectra of **1a** and **1b** in CD_3CN–D_2O (95:5, v/v) upon addition of CN^-.

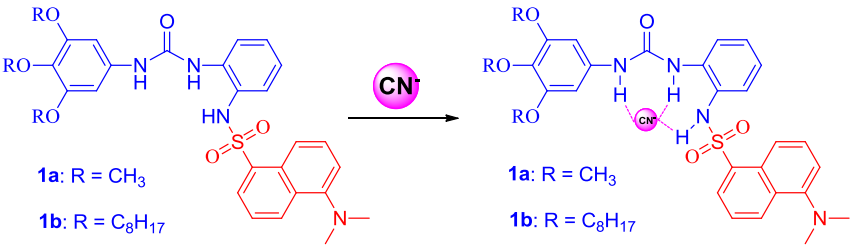


Fig. 6. The possible binding mechanism of **1a** and **1b** with CN^-.

Compounds **1a** and **1b** only exhibit a large change in fluorescence with cyanide ions in CH_3CN–H_2O (95:5, v/v), which suggests that the selectivity of **1a** and **1b** towards cyanide ions could be controlled by changing the solvent.

Subsequently, the response of compounds **1a** and **1b** to CN^- ions in CH_3CN–H_2O (95:5, v/v) was investigated in detail. As presented in Fig. 3, upon the addition of CN^- ions, the fluorescence intensity decreased gradually, the association constant for CN^- was calculated to be 1.25×10^5 L mol^{-1} and the limit of detection of **1a** with CN^- ions calculated as 1.255×10^{-8} M. Significantly, the fluorescence quenching at 538 nm was linear with respect to the concentration of CN^- ions at low concentrations ($R^2 = 0.986$) (in

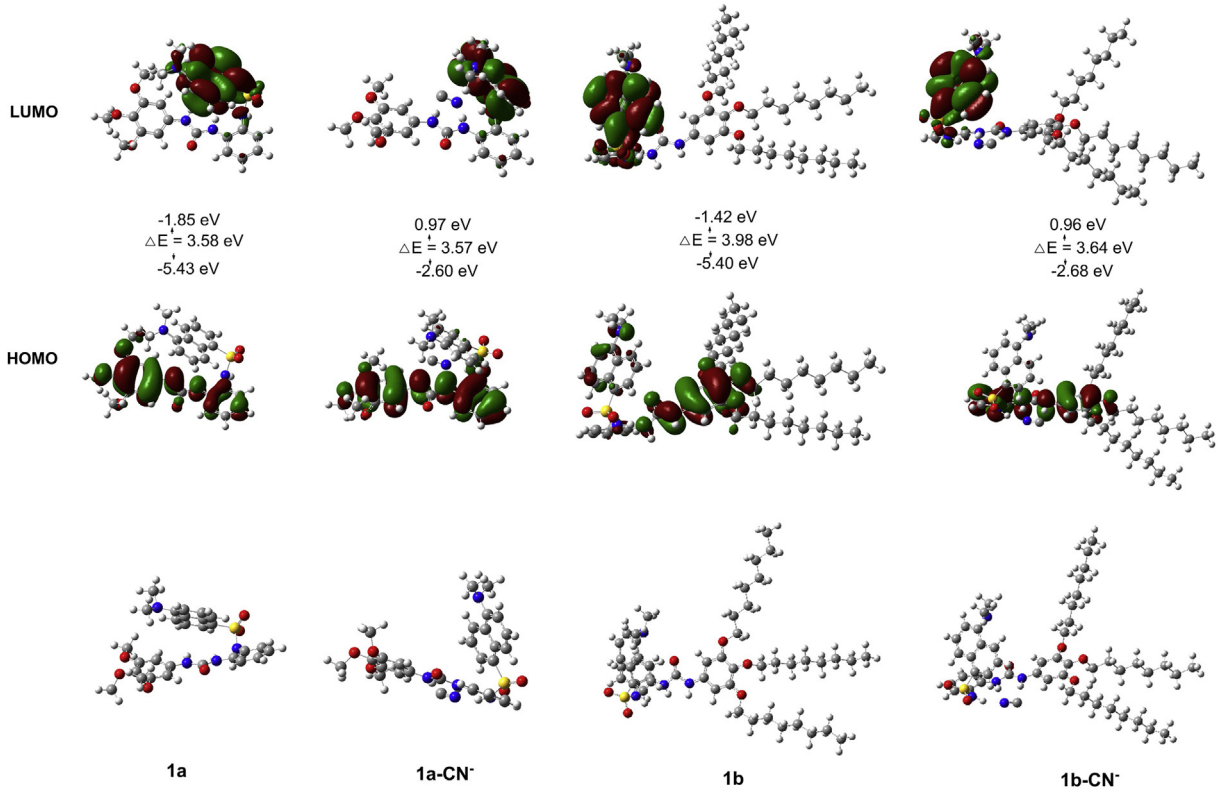


Fig. 7. The optimized structures, LUMO, HOMO and their corresponding energy gaps of **1a** and **1b** with CN^- at B3LYP/6–31G* level, by using Gaussian 03 program.

Fig. S4). In order to understand the binding modes with CN^-, Job plot analyses were obtained. The results indicate that **1a** formed a 1:1 stoichiometric complex with CN^-. Similar fluorescence changes were also obtained with a solution of **1b** (in Fig. 3). Upon the addition of CN^- ions, the fluorescence intensity decreased gradually, the association constant for CN^- was calculated to be 2.88×10^6 L mol^{-1} and the limit of detection of **1b** with CN^- ions was calculated as 0.4×10^{-8} M. Job plot analyses were obtained and the results indicate that **1b** formed a 1:1 stoichiometric complex with CN^-. In comparison to **1a**, the association constant of **1b** for CN^- ions was greater with a lower limit of detection.

3.3. The sensing mechanism

Considering that stoichiometry of the host–guest complex between **1a** or **1b** and CN^- was 1:1, we tried to understand their binding mechanism with CN^-. Luckily, a single crystal of compound **1a**, for X-ray diffraction analysis, was grown by slow diffusion of hexane into a solution of **1a** in dichloromethane. The solid-state structure shown in Fig. 4 clearly suggests the presence of both intermolecular and intramolecular H-bonds in **1a**. The intramolecular hydrogen bond between the hydrogen atom of the sulfonamide group and the oxygen atom of the urea group possessed an N … H distance of 2.324 Å. The intermolecular hydrogen bond between the hydrogen atoms of the urea group and the oxygen atoms of the methoxy groups has an N … H distance of 2.131 Å and 2.868 Å, respectively. The naphthalene ring and the trimethoxybenzene ring were nearly in a parallel position with a dihedral angle between them of 6.91°. In addition, the dihedral angle between the naphthalene ring and benzene ring is 53.50° and the dihedral angle between the trimethoxybenzene ring and benzene ring is 53.36°. Subsequently, the partial ^{1}H NMR spectra of **1a** and

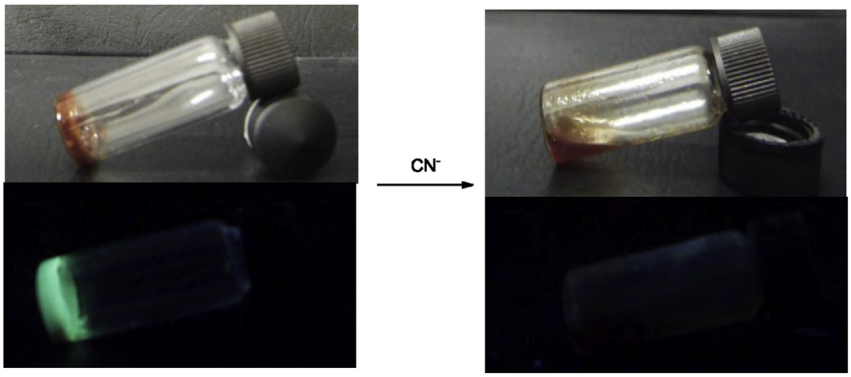


Fig. 8. The response of **1b** (6 mg/mL) in DMSO under the cyanide anion.

1b upon addition of CN^- ions in $CH_3CN–H_2O$ (95:5, v/v) were acquired, as shown in Fig. 5. Upon addition of CN^- ions, the resonance of the protons in the urea group of **1a** displayed a downfield shift when compared to those for **1a** in the absence of CN^- ions as a result of the formation of hydrogen bonding between NH groups and CN^-. Similarly, the resonance of the proton in the sulfonamide group of **1a** displayed a slight upfield shift. The resonance of the protons in the naphthalene ring displayed both downfield and upfield shifts, which were similar to those observed for protons on the benzene rings. Similar proton resonance changes were also obtained in the ^1H NMR spectra of **1b** in $CH_3CN–H_2O$ (95:5, v/v) (in Fig. 5). These results suggest that **1a** and **1b** act as cyanide fluorescence sensors via hydrogen bonding. The possible binding mechanism of both compounds with CN^- ions is presented in Fig. 6.

In order to further investigate the binding mode of **1a** or **1b** with CN^- ions, DFT calculations at the B3LYP/6–31G* level using the Gaussian 03 program were used. As presented in Fig. 7, when compared with the optimized structure for **1a**, upon the addition of CN^- the cavity becomes smaller after binding with CN^-. The hydrogen atoms in the urea and sulfonamide group interact with the CN^- ion, which was consistent with our experimental observations. The LUMO of **1a**-CN^- was distributed over the sulfonamide group and the HOMO of **1a**-CN^- was distributed over the urea group. The energy level gap was 3.57 eV. Similarly, when compared with the optimized structure for **1b**, upon the addition of CN^-, the cavity becomes smaller after binding with CN^-. The hydrogen atoms in the urea and sulfonamide groups interact with the CN^- ion due to the formation of hydrogen bonding between the cyanide ion and NH groups on the urea and sulfonamide units. The LUMO orbital of **1b**-CN^- was distributed over the sulfonamide group and the HOMO orbital of **1b**-CN^- was distributed over the urea group. The energy level gap was 3.64 eV. So, we propose that the binding mode of the proton on the urea and sulfonamide groups of **1a** and **1b** with CN^- was accomplished, which was consistent with the observations from our ^1H NMR spectroscopy studies.

3.4. The gel properties of *1b*

Compound **1b** can act as a gel in DMSO. By placing a small quantity (6.0 mg) of **1b** in a vial, adding DMSO (1.0 mL), heating the suspension until complete dissolution and then allowing the solution to cool to room temperature, an obvious fluorescence quenching of **1b** was observed in DMSO upon the addition 10 eq CN^- (Fig. S5). More importantly, the response of gel **1b** towards the cyanide anions in DMSO led to easy-to-discern changes in the fluorescence color. As shown in Fig. 8, the fluorescence color of gel **1b** was green and when exposed to cyanide ions the gel of **1b** transformed into a homogeneous solution with obvious fluorescence quenching. These results suggest that **1b** could serve as a cyanide sensor not only in solution but also as a gel.

4. Conclusion

In summary, two sulfonamide bearing compounds were successfully developed and their spectroscopic and anion recognition properties fully investigated. The results showed that both compounds show high selectivity towards cyanide and fluoride ions in CH_3CN. In particular, they only exhibited a large change in fluorescence with cyanide in $CH_3CN–H_2O$ (95:5, v/v). More importantly, compound **1b** can act as a gel in DMSO that can transform into a homogeneous solution upon exposure to cyanide ions. This research suggests that sulfonamide and urea can act as a hydrogen-bond donor and provides an alternative approach to the design of novel anion chemosensors. Further work will focus on anion chemosensors with novel topological structures.

Acknowledgments

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Appendix A. Supplementary material

Supplementary data related to this article can be found at doi:10.1016/j.dyepig.2015.03.036.

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