Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy 132 (2014) 771–775

Contents lists available at ScienceDirect

Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy

journal homepage: www.elsevier.com/locate/saa

A simple colorimetric chemosensor bearing a carboxylic acid group with high selectivity for CN^-

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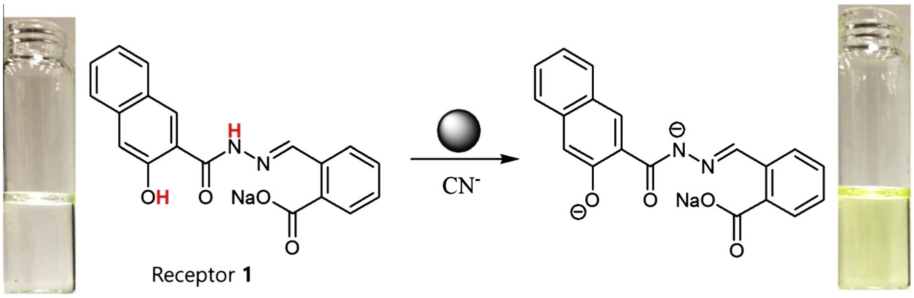
HIGHLIGHTS

- A new Schiff base was synthesized as a colorimetric CN^- sensor.
- Sensor 1 exhibited a color change from colorless to yellow via a simple deprotonation.
- Sensor 1 showed high selectivity for CN^- over other various anions.

GRAPHICAL ABSTRACT



ARTICLE INFO

Article history:
Received 15 March 2014
Received in revised form 30 April 2014
Accepted 1 June 2014
Available online 12 June 2014

Keywords:
Cyanide
Colorimetric
Naphthoic hydrazide
Carboxylic acid
Deprotonation

ABSTRACT

A new simple 'naked eye' chemosensor **1** (sodium (E)-2-((2-(3-hydroxy-2-naphthoyl)hydrazono)methyl)benzoate) has been synthesized for detection of CN^- in a mixture of DMF/H_2O (9:1). The sensor **1** comprises of a naphthoic hydrazide as efficient hydrogen bonding donor group and a benzoic acid as the moiety with the water solubility. The receptor **1** showed high selectivity toward cyanide ions in a 1:1 stoichiometric manner, which induces a fast color change from colorless to yellow for CN^- over other anions. Therefore, receptor **1** could be useful for cyanide detection in aqueous environment, displaying a high distinguishable selectivity from hydrogen bonded anions and being clearly visible to the naked eye.

Introduction

The importance of anions in biological, environmental and chemical processes has affected the development of anion sensing instruments [1]. Among various anions, cyanide has been extensively studied [2], because the cyanide anion is very often used industrially in the synthesis of organic chemicals, polymers, metallurgy as well as in gold mining [3]. In addition, it is much more famous due to its toxicity to the environment and to mammals, leading to convulsions, loss of consciousness, and eventual death [4]. Consequently, cyanide selective detection and quantification are very important, which has been the object of increasing investigation [5].

Although previous works have involved the development of a wide variety of chemical and physical sensors for the detection of CN^-, improving the detection selectivity among coexisting anions has been challenging [6]. In addition, most of these methods require expensive equipment and involve time-consuming and laborious procedures that can be carried out only by trained professionals, significantly restricting the practical application of these

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http://dx.doi.org/10.1016/j.saa.2014.06.001
1386-1425/

CN^- sensors [7]. For simplicity, convenience and low cost, easily-prepared CN^- colorimetric chemosensors are needed [8].

Over the past decades, many efforts have been devoted to design various chemosensors for the detection of cyanide, including the formation of cyanide complexes with transition metals [9], nucleophilic addition reactions to carbonyl groups [10], the displacement approach [11], hydrogen-bonding interactions [12] and deprotonation [13]. Among them, the deprotonation process of sensors with CN^- has scarcely been studied. Moreover, the development of colorimetric chemosensors using deprotonation mechanism that can discriminate CN^- from F^- and OAc^- is still a challenge as they are also well known as anions with the high basicity and the hydrogen bonding ability [14]. Thus, a technique to trace and visualize cyanide ions would be highly demanded.

In view of this requirement and as part of our research effort devoted to cyanide ion recognition, we have, first, considered the possibility of a hydrogen bond between the amide (CONH)/OH moieties of 3-hydroxy-2-naphthoic hydrazide and the targeted anions. Secondly, we tried to incorporate the carboxylate moiety to a sensor to increase water solubility. Importantly, the chemosensors with the water-soluble acid groups have hardly been reported. Our strategy led us to synthesize a new naphthoic hydrazide-carboxylate conjugated optical probe for CN^- where the naphthoic hydrazide works as a H-bonding donor and the carboxylate group increases water solubility.

Herein, we report a new chemosensor **1** for CN^-, which was synthesized in one step by condensation reaction of 3-hydroxy-2-naphthoic hydrazide and carboxylate (Scheme 1). Receptor **1** can detect cyanide by color change from colorless to yellow via the ‘naked-eye’ with high selectivity in aqueous environment ($DMF/H_2O = 9/1$).

Experimental

Reagent and apparatus

All the solvents and reagents (analytical grade and spectroscopic grade) were obtained commercially and used as received. ^1H NMR and ^{13}C NMR measurements were performed on a Varian 400 MHz spectrometer and chemical shifts are recorded in ppm. Absorption spectra were recorded using a Perkin Elmer model Lambda 2S UV–vis spectrometer at room temperature. Electrospray ionization mass spectra (ESI-mass) were collected on a Thermo Finnigan (San Jose, CA, USA) LCQTM Advantage MAX quadrupole ion trap instrument. Elemental analysis for carbon, nitrogen, and hydrogen was carried out by using a Flash EA 1112 elemental analyzer (thermo) in Organic Chemistry Research Center of Sogang University, Korea.

Synthesis of receptor **1**

3-Hydroxy-2-naphthoic acid hydrazide (0.20 g, 1 mmol) and 2-formylbenzoic acid (0.18 g, 1.2 mmol) were dissolved in 30 mL of ethanol. Then, sodium hydroxide (0.04 g, 1.2 mmol) in 5 mL of H_2O was added into the reaction mixture, which was stirred for 3 h at room temperature. The white powder was produced. The white solid was collected by filtration, washed with diethyl ether and air-dried. Yield: 0.29 g (82.3%). ^1H NMR (DMSO-d_6, 400 MHz) δ 12.80 (s, 2H), 9.26 (s, 1H), 8.56 (s, 1H), 8.05 (d, 1H), 7.85 (t, 2H), 7.71 (d, 1H), 7.53 (t, 2H), 7.47 (t, 2H), 7.40 (s, 1H), 7.31 (t, 1H) ppm. ^{13}C NMR (DMSO-d_6, 100 MHz) δ 170.25, 164.51, 155.97, 148.88, 136.74, 133.93, 131.29, 130.7, 130.11, 129.44, 128.68, 127.15, 126.73, 126.33, 123.93, 121.01, 111.52 ppm. HRMS (ESI): m/z calcd. for $C_{19}H_{13}N_2NaO_4$ ($[M-Na^+]$), 333.09; found, 333.13. Anal. calcd. for $C_{19}H_{13}N_2NaO_4$ (356.31): C, 64.05; H, 3.68; N, 7.86. Found: C, 64.01; H, 3.90; N, 7.32%.

UV–vis measurements

Receptor **1** (3.56 mg, 0.01 mmol) was dissolved in DMF (1 mL) and 9 μL of the **1** (10 mM) were diluted with 2.991 mL DMF/H_2O (9:1, v/v) to make the final concentration of 30 μM. Tetraethylammonium cyanide (TEACN, 15.6 mg, 0.1 mmol) was dissolved in a mixture of DMF/H_2O (v/v = 9:1, 1 mL). 9–90 μL of the CN^- solution (100 mM) were transferred to each receptor solution (30 μM) prepared above. After shaking the vials for a few minutes, UV–vis spectra were taken at room temperature.

Competition with other anions

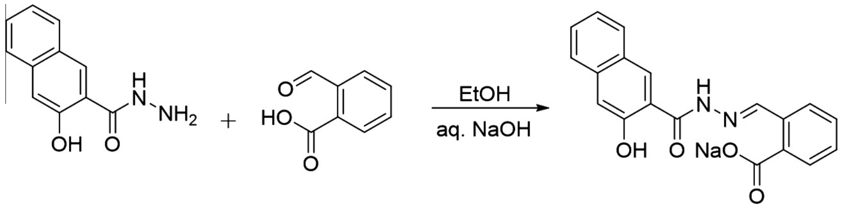
Receptor **1** (3.56 mg, 0.01 mmol) was dissolved in DMF and 9 μL of the **1** (10 mM) were diluted with 2.991 mL DMF/H_2O (9:1, v/v) to make the final concentration of 30 μM. Tetraethylammonium salt (F^-, Br^-, Cl^-, I^-, and CN^-; 0.1 mmol) or tetrabutylammonium salt (AcO^- and $H_2PO_4^-$; 0.1 mmol) were dissolved in a mixture of DMF/H_2O (v/v = 9:1, 1 mL), respectively. 45 μL of each anion solution (100 mM) were taken and added into 3 mL of each **1** solution (30 μM) prepared above to make 50 equiv. Then, 50 μL of CN^- solution (100 mM) were added into the mixed solution of each metal ion and **1** to make 50 equiv. After shaking the vials for a few minutes, UV–vis spectra were taken at room temperature.

Job plot measurement

Receptor **1** (3.56 mg, 0.01 mmol) was dissolved in a mixture of DMF/H_2O (v/v = 9:1, 1 mL). 12, 10.8, 9.6, 8.4, 7.2, 6.0, 4.8, 3.6, 2.4, and 1.2 μL of the **1** solution were taken and transferred to vials. Each vial was diluted with DMF/H_2O (9:1, v/v) to make a total volume of 2.988 mL. TEACN (1.56 mg, 0.01 mmol) was dissolved in DMF/H_2O (v/v = 9:1, 1 mL). 0, 1.2, 2.4, 3.6, 4.8, 6.0, 7.2, 8.4, 9.6, 10.8, and 12 μL of the CN^- solution were added to each diluted **1** solution. Each vial had a total volume of 3 mL. After shaking the vials for a few minutes, UV–vis spectra were taken at room temperature.

^1H NMR titration of **1**-CN^-

Four NMR tubes of **1** (0.76 mg, 0.002 mmol) dissolved in DMF-d_7 (0.7 mL) were prepared, and four different equiv (0, 0.5, 1,



Scheme 1. Synthetic procedure of **1**.

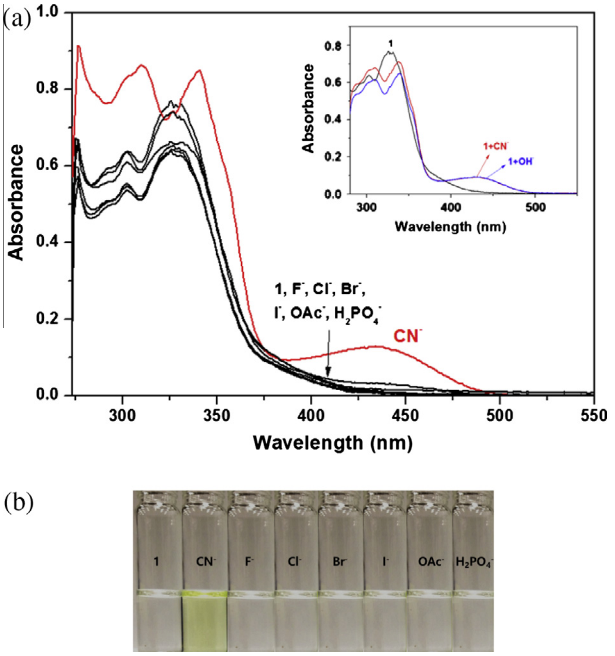


Fig. 1. (a) UV–vis spectra changes of **1** (30 μM, DMF-buffer (9:1, v/v)) upon addition of 50 equiv of various anions. (b) Colorimetric changes of **1** (30 μM) upon the addition of various anions (50 equiv). Inset: Changes in the UV–vis spectra of receptor **1** upon addition of CN^- and OH^-, respectively, in DMF-buffer (9:1, v/v).

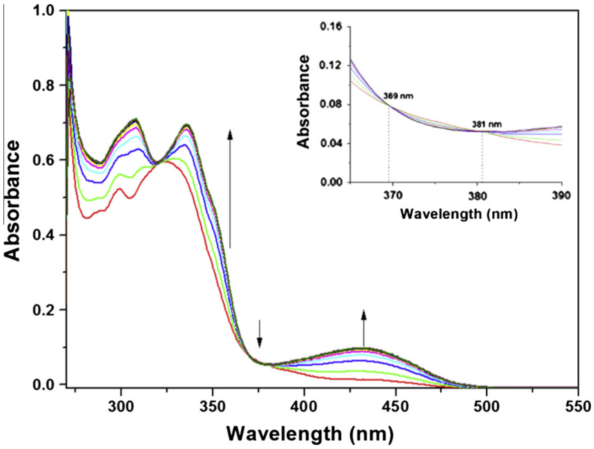


Fig. 2. UV–vis spectra changes of **1** (30 μM) in the presence of different concentrations of CN^- ion in DMF-buffer solution (9:1, v/v). Inset: The enlarged spectra at the range of 365–390 nm.

2 equiv) of tetraethylammonium cyanide dissolved in DMF-d_7 (0.3 mL) were added to the **1** solution separately. After shaking them for a minute, their ^1H NMR spectra were taken at room temperature.

Results and discussion

The colorimetric chemosensor **1** for cyanide was synthesized by condensing of 3-hydroxy-2-naphthoic acid hydrazide with 2-formylbenzoic acid (Scheme 1), and characterized by ^1H and ^{13}C NMR, ESI-mass spectrometry, and elemental analysis.

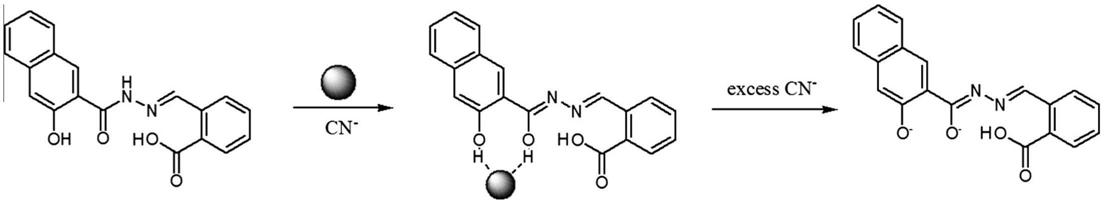
Sensing properties of 1 toward anions in aqueous solution

The colorimetric sensing abilities of **1** were primarily investigated in a mixture of DMF/H_2O (9:1, v/v) upon addition of various anions (F^-, AcO^-, Cl^-, Br^-, I^-, $H_2PO_4^-$ and CN^-). Upon the addition of 50 equiv of each anion, the **1** showed almost no change in absorption peak in the presence of F^-, AcO^-, Cl^-, Br^-, I^- and $H_2PO_4^-$ (Fig. 1a). Most importantly, only CN^- showed a distinct spectral change (Fig. 1a) and a color change from colorless to yellow (Fig. 1b), indicating that receptor **1** can serve as a potential candidate of "naked-eye" chemosensor for CN^- in aqueous solution.

The recognition properties of **1** with CN^- were further studied by UV–vis titration experiments. On treatment with CN^- of a solution of **1**, the absorption band at 374 nm slightly decreased, and three new bands at 308, 336 and 433 nm gradually reached maxima at 50 equiv of CN^- (Fig. 2). Meanwhile, two clear isosbestic points were observed at 369 and 381 nm, indicating the formation of the only one species between **1** and CN^-. The yellow color of the solution may be due to the deprotonation of phenolic and enol OH groups followed by hydrogen bonding with cyanide ions [15]. As shown in Scheme 2, the potential H-bonding pocket defined by two OH protons which have the potential to bind anions in a chelate mode, tends to bring the cyanide to their vicinity [4]. To further confirm the sensing mechanism between **1** and CN^-, the interaction between **1** and OH^- was also investigated. UV–vis spectral change of **1** upon addition of OH^- was similar with that of **1** obtained from the addition of CN^-, which indicated the deprotonation between **1** and CN^- (inset in Fig. 1).

The Job plot showed a 1:1 stoichiometry between **1** and CN^- (Fig. S1) [16], which was further confirmed by ESI-mass spectrometry analysis (Fig. 3). The negative-ion mass spectrum of **1** upon addition of 1 equiv of CN^- showed the formation of the $\mathbf{1}^{2-}$ [m/z: 593.30; calcd., 593.27]. From the UV–vis titration data, the association constant for **1** with CN^- was determined as 1.4 (±0.1) × $10^2\ M^{-1}$ using Benesi–Hildebrand equation (Fig. S2) [17]. This value is within the range of those (1.0–1.0 × 10^5) reported for CN^- sensing chemosensors [18]. The detection limit ($3\sigma/k$) of receptor **1** for the analysis of CN^- ions was calculated to be 7.0×10^{-4} M (Fig. S3) [19].

The preferential selectivity of **1** as a naked eye chemosensor for the detection of CN^- was studied in the presence of various competing anions. For competition studies, receptor **1** was treated with 50 equiv of CN^- in the presence of 50 equiv of other anions, as indicated in Fig. 4. There was no interference in the detection of CN^- in the presence of F^-, Cl^-, Br^-, I^-, OAc^-, and $H_2PO_4^-$. Thus, **1** could be used as a selective colorimetric sensor for CN^- in the presence of the competing anions.



Scheme 2. Proposed sensing mechanism of **1** for cyanide.

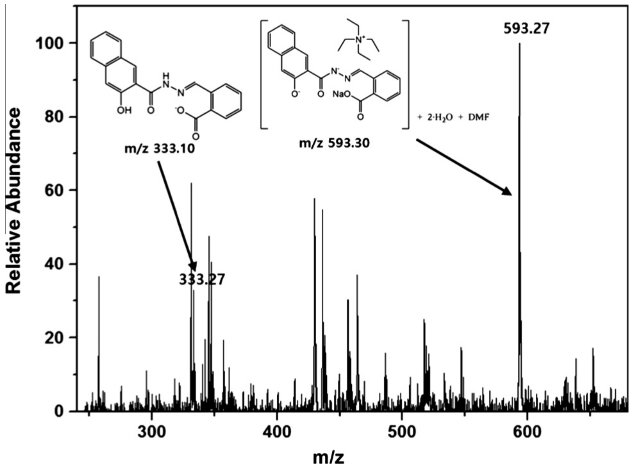


Fig. 3. Negative-ion electrospray ionization mass spectrum of **1** upon addition of TEA(CN) (1.0 equiv) in acetonitrile.

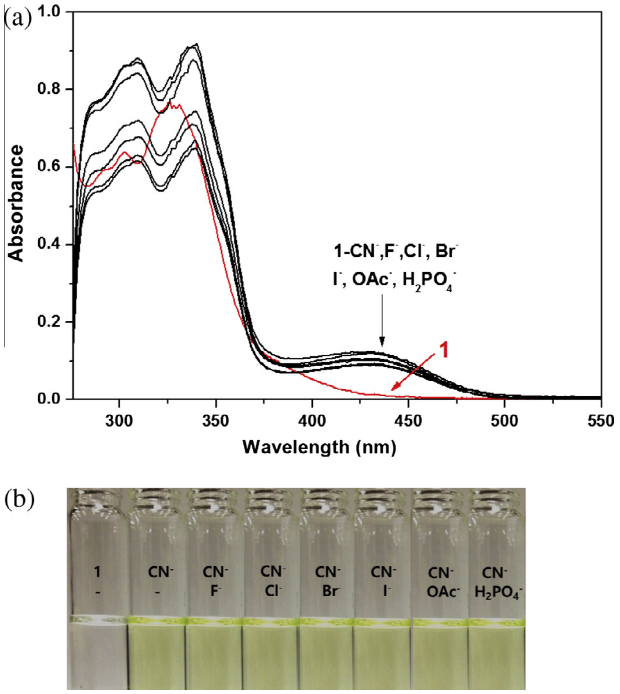


Fig. 4. (a) Competitive selectivity of **1** (30 μM) towards CN^- (50 equiv) in the presence of other anions (50 equiv). (b) Colorimetric changes of **1** (30 μM) in the presence of CN^- (50 equiv) and other anions (50 equiv).

To further understand the nature of interaction between sensor **1** and the cyanide, ^1H NMR study was initiated in DMF-d_7 (Fig. S4). Upon addition of 0.2 equiv of TEA(CN) as the cyanide source, the singlet peaks (H_1 and H_8) of naphthol and enol OH moieties were downshifted from 13.01 ppm to 13.79 ppm. The H_7 in the naphthol ring and H_9 of the imine group were also downshifted from 9.53 ppm to 9.57 ppm and from 8.79 ppm to 8.81 ppm, respectively. After the addition of 1 equiv of cyanide, the singlet protons, H_1 and H_8, disappeared. Meanwhile, all aromatic protons were shifted to upfield, which suggests that the negative charges generated from the deprotonation of **1** by CN^- were delocalized over the whole receptor molecule. In addition, no new peak was observed in the range of 5–6 ppm, which indicates exclusion for the nucleophilic addition of cyanide to the imine moiety. Therefore, this result could support the fact that the sensing mechanism of **1** for cyanide occurs by the deprotonation pathway, not by the addition reaction of cyanide to the imine moiety of **1**. Furthermore, no shift in the position of proton signals was observed on any further addition of CN^- (>1.0 equiv). Based on ESI-mass spectrometry analysis and ^1H NMR study, we finally propose that the sensing ability of receptor **1** is due to the deprotonation process by CN^-, as shown in Scheme 2.

Conclusion

We synthesized a carboxylic-functionalized water-soluble imine-based colorimetric sensor **1**, which displays high selectivity for detection of cyanide in aqueous solution. **1** sensed CN^- in a 1:1 stoichiometric manner, which induces an obvious color change from colorless to yellow. Such selectivity results from a simple deprotonation between **1** with the H-bonding donor group and CN^-. Consequently, sensor **1** appears to be a practical system for monitoring CN^- in aqueous solution.

Acknowledgements

Basic Science Research Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science and Technology (2012001725, 2011K000675, and 2012008875) are gratefully acknowledged. We thank Prof. Mi Sook Seo (Ewha Womans University) for ESI-mass running and helpful comments.

Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.saa.2014.06.001.

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