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Three hydroxy aurone compounds as chemosensors for cyanide anions



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HIGHLIGHTS

- Three 4-hydroxy aurone compounds were synthesized.
- The compounds can recognize cyanide anions with obvious color and fluorescence change.
- The compounds allow naked eye detection of cyanide anions.

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Introduction

It is well known that cyanide anions are extremely toxic to living organism and a small amount of cyanide anions will lead to vomiting, convulsions, loss of consciousness, and even eventual death [1]. Despite their toxicity, cyanides are still widely applied to various processes as raw materials for synthetic fiber, synthetic resin, herbicide, and the gold-extraction process [1–3], which releases cyanide into the environment as a toxic contaminant. According to the standard of World Health Organization, cyanide

GRAPHICAL ABSTRACT



ABSTRACT

Three new 4-hydroxy aurone compounds **1–3** with dimethylamino (**1**), bromine (**2**) and cyano (**3**) as terminal group have been synthesized. Their photophysical properties as well as recognition properties for cyanide anions in acetonitrile and aqueous solution have also been examined. These compounds exhibit remarkable response to cyanide anions with obvious color and fluorescence change owing to hydrogen bonding reaction between cyanide anions and the O–H moiety of the sensors, which allows naked eye detection of cyanide anions.

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concentrations in drinking water cannot be more than $1.9 \,\mu$ M [4,5]. Therefore, highly selective probes for cyanide anions are of considerable interest due to their applicability to environmental cyanide detection and to the pathological bioimaging of the anions.

In recent years, the optical chemosensors for cyanide anions, especially sensors exhibiting obvious color and/or fluorescence change easily observed by the naked eye directly have attracted considerable attention because of their simplicity, low cost, and rapid measurement [6–25]. Our group is devoted to the research on chemosensors for cyanides for several years [26–28].

Here three new 4-hydroxy aurone receptors with high sensitivity and selectivity for cyanide anions through hydrogen bonding were synthesized and the recognition properties to cyanide anions were investigated. These compounds exhibit remarkable response

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to cyanide anions with obvious color and fluorescence changes, which allows naked eye detection of cyanide anions.

Experimental

Chemical and instruments

2',6'-Dihydroxyacetophenone, p-dimethylaminobenzaldehyde, p-bromobenzaldehyde and 4-formylbenzonitrile were purchased from Aladdin Reagents. Other reagents were purchased from Shanghai Reagents and were used as received directly without further purification. NMR spectra were recorded on a Bruker Avance III 400 MHz spectrometer at ambient temperature. Mass spectra were recorded on an Agilent Q-TOF6510 spectrometer. Elemental analyses were carried out on a PE 2400 autoanalyzer.

Synthesis and characterization

The synthetic routes to compounds **1–3** is outlined in Scheme S1. The compounds were synthesized via four steps [29]. Firstly, one hydroxyl group in 2',6'-dihydroxyacetophenone was protected using methoxymethoxy group by the reaction of 2',6'-dihydroxyacetophenone with methyl chloromethyl ether in acetone to afford 2'-hydroxy-6'-methoxymethoxy acetophenone (**S**-**1**) as colorless oil with a high yield (83%). Secondly, **S**-**1** reacts with the aldehydes to afford the corresponding 2'-hydroxy-6'-methoxymethoxy aurone compounds (**S**-**2**). Thirdly, 4-methoxymethoxy aurone compounds (**1**'-**3**') were obtained by the cyclization of chalcones using mercury (II) acetate in pyridine according to Ref. [**30**]. Finally, the methoxymethoxy group in **1**'-**3**' was deprotected to afford the 4-hydroxy products **1**-**3** with high yield.

4'-N,N-Dimethylamino-4-hydroxy-aurone (**1**) Compound **1** was red solid in 92% yield. ¹H NMR (CDCl₃, 400 MHz), *δ* (ppm): 3.07(s, 6H), 6.58 (d, J = 8.2 Hz, 1H), 6.76(d, J = 8.9 Hz, 2H), 6.77(d, J = 8.1 Hz,1H), 6.85(s, 1H), 7.46 (t, J = 8.2 Hz, 1H), 7.83(d, J = 8.9 Hz, 2H), 7.97 (s, 1H). ¹³C NMR (CDCl₃, 100 MHz), *δ* (ppm): 40.18, 103.60, 109.16, 110.47, 112.10, 115.55, 119.90, 133.89, 138.18, 144.62, 151.65, 156.69, 164.36, 185.00. MS for (M + H)⁺, Calcd exact mass: 282.1130, found 282.1119. Anal. Calcd for C₁₇H₁₅NO₃: C, 72.58; H, 5.37; N, 4.98. Found C, 72.71; H, 5.39; N, 4.99.

4'-Bromide-4-hydroxy-aurone (**2**). Compound **2** was yellow solid in 97% yield. ¹H NMR (CDCl₃, 400 MHz), *δ* (ppm): 6.64(d, J = 8.4 Hz, 1H), 6.76(s, 1H), 6.81(d, J = 8.4 Hz, 1H), 7.54(t, J = 8.4 Hz, 1H), 7.59(d, J = 8.8 Hz, 2H), 7.77(d, J = 8.8 Hz, 2H), 7.78 (s, 1H). ¹³C NMR (CDCl₃, 100 MHz), *δ* (ppm): 103.78, 109.56, 110.31, 111.80, 124.72, 131.06, 132.36, 132.96, 139.60, 146.98, 156.84, 164.94, 185.63. MS for (M + H)⁺, Calcd exact mass: 316.9813, found 316.9825. Anal. Calcd for C₁₅H₉BrO₃: C, 56.81; H, 2.86. Found C, 56.92; H, 2.87.

4'-Cyano-4-hydroxy-aurone (**3**) Compound **3** was yellow solid in 96% yield. ¹H NMR (CDCl₃, 400 MHz), *δ* (ppm): 6.67(d, J = 8.4 Hz, 1H), 6.78(s, 1H), 6.82(d, J = 8.0 Hz, 1H), 7.57(t, J = 8.0 Hz, 1H), 7.73(d, J = 8.4 Hz, 2H), 7.75(s, 1H), 7.98(d, J = 8.4 Hz, 2H). ¹³C NMR (CDCl₃, 100 MHz), *δ* (ppm): 103.72, 109.11, 110.00, 110.72, 112.82, 118.50, 131.57, 132.52, 136.47, 139.94, 148.05, 156.86, 164.91, 185.36. Anal. Calcd for C₁₆H₉NO₃: C, 73.00; H, 3.45; N, 5.32. Found C, 73.16; H, 3.46; N, 5.31.

Photophysical properties and response to cyanide anions

Solutions of compounds **1–3** with 10 μ M in acetonitrile (CH₃CN) or CH₃CN-H₂O solution were prepared for photophysical measurements. UV–vis absorption and steady-state fluorescence spectra were recorded on a Shimadzu UV2550 spectrophotometer and an

Edinburgh FLS 920 spectrometer, respectively. The compounds were titrated with cyanide anions by addition of a solution TBACN in acetonitrile.

Structure determination

Single crystal of compound **1** was obtained by slow diffusion of the compound in dichloromethane. X-ray diffraction of a red single crystal of **1** (0.5 mm × 0.5 mm × 0.2 mm) was collected on a Bruker Smart APEX-II CCD X-ray single crystal diffractometer. The crystal belongs to the monoclinic system, P 2₁/*n* space group, with formula C₁₇H₁₅NO₃ and molecular weight 281.30; *T* = 296.0 (2) K, *a* = 7.5419 (2) Å, *b* = 23.7783 (8) Å, *c* = 8.4789 (2) Å, *β* = 110.193 (2)°, *V* = 1427.09(9) Å³, *Z* = 4, *F*(000) = 592, μ = 0.090 mm⁻¹, *d*_{calcd} = 1.309 Mg m⁻³, *R*₁ = 0.0439, w*R*₂ = 0.1483.

Results and discussion

Synthesis and crystal structure

The hydroxyl aurone compounds **1–3** were synthesized via four steps with 2',6'-dihydroxyacetophenone and p-substituted benzaldehyde as the starting materials with high yield. All the new compounds were fully characterized by NMR, mass spectra and elemental analyses.

The molecular structure of compound **1** along with the atom labeling is shown in Fig. 1. Benzofuran group possesses perfect planarity with the maximum deviation for the non-hydrogen atoms being 0.017 Å for O1. Atoms O1 and O2 are also almost in benzofuran plane with the deviations are 0.006 Å (O1) and 0.019 Å (O2), respectively. The molecular backbone exhibits perfect planarity with the dihedral angle between benzofuran and benzene group being 7.62°, which is much larger than that of aurone without hydroxyl group (5.80°) [30]. That means that the import of hydroxyl group slightly decreases the molecular planarity.

Linear absorption and steady fluorescence properties

Linear absorption and steady fluorescence properties of the compounds in acetonitrile are shown in Table 1 and the spectra are shown in Fig. 2. Compound 1 can be classified as D- π -A type of molecule with dimethylamino and benzofuran as electron-donating and withdrawing groups, respectively. But compounds 2 and 3 can be classified as A- π -A' type of molecules with two electron-withdrawing groups (2: bromide and benzofuran; 3: cyano and benzofuran). From Fig. 2, we can see that compound 1 exhibits strong linear absorption in blue region with the main absorption peak at 449 nm, which can be ascribed to charge transfer from dimethylamino to benzofuran. Compounds 2 and 3 possess similar absorption spectra with the main absorption peak at 389 nm, which also can be ascribed to intramolecular charge transfer and



Fig. 1. The molecular structure of compound 1.

Table 1

Photophysical pr	roperties of	compounds	1-3 in	acetonitrile.
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Compounds	λ^{abs} (nm)	$\epsilon (10^4 \text{M}^{-1} \text{cm}^{-1})$	λ^{fluo} (nm)
1	449	3.73	552
2	308, 389	2.42, 2.85	487
3	305, 389	3.23, 2.53	510



Fig. 2. Linear absorption and steady fluorescence spectra of compounds 1-3 in acetonitrile.

the absorption peak has a 60 nm blue shift relative to that of **1**. Compound **1** emits strong yellow–green fluorescence at 552 nm, but compound **2** and **3** exhibit strong green or blue fluorescence at 510 nm(**2**) and 487 nm (**3**), respectively. The red shift of absorption and fluorescence peak of **1** relative to that of **2** and **3** may be derived from the stronger electron donating ability of terminate dimethylamine group, which make the molecule polarity increase and energy gaps decrease. The strong electron-donating ability of dimethylamino is beneficial to charge transfer and then **1** exhibits the strongest linear absorption and fluorescence emission.

Spectral response to cyanide anions

As a general adapted method, n-Bu₄NCN (TBACN) as cyanide source was progressively added to solutions of compounds 1-3 in CH₃CN or CH₃CN-H₂O solution. The linear absorption and steady fluorescence emission of solutions containing increasing cyanide anions were recorded to examine the complexation between 1-3 and cyanide anions. The changes in UV-vis absorption spectra of the compounds in acetonitrile upon the addition of different equivalents of TBACN are shown in Fig. 3 (1 and 2) and Fig. S1 (3). Compounds 1-3 show obvious spectral response to the cyanide anions. As shown in Fig. 3(a), we can see that the characteristic intense charge transfer absorption band of compound 1 at 449 nm gradually decreases and is red shift to 470 nm upon the addition of cyanide anions, at the same time, a new weak absorption band at 378 nm appears. When 1.4 equiv. TBACN was added, the complexation between compound 1 and cyanide anions is almost saturated. The absorbance at 449 nm decreases from 0.373 to 0.221 and the peak at this absorption band has a 20 nm red shift. At the same time, the absorbance at 378 nm increase from 0.049 to 0.132. And the solution color changed from light yellow to orange.

Compounds **2** and **3** exhibit similar absorption spectra response to cyanide anions (Fig. 3(b) and Fig. S1). The original absorption band gradually decreases, at the same time, a new absorption band appears. When 1.6 equiv. cyanide anions were added, the reaction is almost saturated. The original absorption peak disappeared completely. The compounds show a good sensitivity for cyanide anion.



Fig. 3. Changes in UV-vis absorption spectra of compounds **1** (a) and **2** (b) in acetonitrile upon the addition of different concentration of TBACN. Insert: absorbance at 378 nm and 449 nm (a) or absorbance at 389 nm and 469 nm (b) as a function of concentration of TBACN.

The solution color of compounds **2** and **3** in acetonitrile changed from colorless to orange. Job's plots (Fig. S2) were carried out and the complexation between the compounds and cyanide anions was observed to be 1:1.

The compounds exhibit similar spectra response in aqueous solution. The changes in UV–vis absorption spectra of the compounds in CH_3CN-H_2O (1:4, v/v) upon the addition of different equivalents of TBACN are shown in Fig. 4 (2) and Fig. S3 (1 and 3). The original absorption peak of compound 2 at 399 nm gradually decreases, at the same time, a new absorption peak at 440 nm appears. When 7.0 equiv. cyanide anions were added, the reaction is almost saturated.

The compounds also exhibit fluorescence spectral response to cyanide anions. From Fig. 5, we can see that the fluorescence spectra at 552 nm of compound **1** has a decrease and a 23 nm red shift with excitation at 450 nm when cyanide anion was added. Original fluorescence intensity of compounds **2** and **3** also decreases with excitation at 389 nm (Fig. S4 (a) and (b)) upon the addition of cyanide anions.

There is an interesting phenomenon, compounds **2** and **3** exhibit new absorption peak at 469 nm (**2**) or 482 nm (**3**) when cyanide anions were added. Fluorescence spectra change of compound **2** upon the addition of cyanide anions with the new absorption peak at 469 nm as the excitation wavelength was also examined (Fig. 6). To the compound **2**, there is a new fluorescence peak at 608 nm appears with 469 nm excitation wavelength and this fluorescence



Fig. 4. Changes in UV-vis absorption spectra of **2** in $CH_3CN-H_2O(1:4, v/v)$ upon the addition of different concentration of TBACN. Insert: absorbance at 399 nm and 440 nm as a function of concentration of TBACN.



Fig. 5. Changes in fluorescence spectra of compound **1** at 450 nm in acetonitrile upon the addition of TBACN.



Fig. 6. Changes in fluorescence spectra of compound **2** at 469 nm in acetonitrile upon the addition of TBACN.

band intensity increases with the addition of cyanide anions. That means compound **2** exhibits turn-on fluorescence response with 469 nm as the excitation wavelength.

In order to examine the binding mechanism of **1–3** with cyanide anions, ¹H NMR spectra upon the addition of cyanide anions to the



Fig. 7. UV–vis spectral changes of 1 (10 $\mu M)$ in acetonitrile observed upon the addition of various anions.

compounds in CD₃CN solvent were monitored. For the sake of brevity, compound **1** was taken as an example. As shown in Fig. S5, upon the addition of TBACN in CD₃CN, the proton signal on –OH in **1** at 8.06 ppm disappeared with the upfield shift of aromatic protons in benzofuran group (H1: from 6.58 ppm to 5.89 ppm; H2: from 7.52 ppm to 7.05 ppm; H3 from 6.86 to 6.00), which corresponds to the occurrence of hydrogen bonding reaction between CN^- and the O–H moiety of the sensor [23]. The possible binding mechanism is shown in Scheme S2. At the first time, hydroxyl group in the compounds reacts with cyanide anion by intermolecular hydrogen bonding interaction and then deprotonation occurs, which can be directly verified by the disappearance of proton signal.

The pH dependence of the absorption spectra of the compounds is also investigated. The absorbance of the compounds at 388 nm (1), 440 nm (2) and 454 nm (3) were monitored as a function of pH. As shown in Fig. S6, the compounds remain almost stable with pH value changing from 2.0 to 6.0, but when pH value is larger than 6.0, the absorbance increase smartly and exhibit similar spectra change with the addition of cyanide anions. pKa values are obtained, which are 10.2 (1), 7.58(2) and 6.86(3), respectively.

The detection limits [25,31,32] in acetonitrile for CN⁻ with fluorescence as detected signal are also investigated (Fig. S7). The concentration of the compounds is 10 μ M. The detection limits of compounds **1–3** in acetonitrile are ca. 0.01 μ M (**1**), 0.058 μ M(**2**) and 0.02 μ M (**3**), respectively, which are smartly lower than 1.9 μ M. The detection limits of the compounds in aqueous solution with absorption as detected signal (Fig. S8) are ca. 4.39 μ M (**1**, CH₃-CN-H₂O(4:1, v/v)), 2.83 μ M (**2**, CH₃CN-H₂O(1:4, v/v)) and 3.44 μ M(**3**, CH₃CN-H₂O(1:4, v/v)), respectively.

It is generally accepted that high selectivity is a necessary criterion for a desirable chemosensor. Thus, the influence of other anions on the compounds was also examined. As shown in Fig. 7, S9 and S10 in contrast to the cyanide anion, other anions such as F^- , Cl^- , Br^- , I^- , $H_2PO_4^-$, and HSO_4^- induce negligible absorption changes, just SCN⁻ induces a small absorption change.

The color and fluorescence changes of 1-3 in CH₃CN upon addition of various anions were also checked. As shown in Fig. S11, cyanide induced compound 1 a color change from light yellow to orange and fluorescence change from yellow–green to orange. In contrast, other anions (in addition to fluoride anions) caused only neglectable color and fluorescence change. Although fluoride induced a similar fluorescence change but there is not obvious color change compared to CN⁻. Based on these observations, the color changes and fluorescence change can be employed to distinguish cyanide from other anions by naked eye. Similar results were

observed for **2** and **3** (Figs. S12 and S13). To compounds **2** and **3**, cyanide anions induced an obvious color change from colorless to orange and fluorescence quench. Based on these observations, the compounds show a highly selective sensing of CN^- .

Conclusion

In summary, three 4-hydroxy aurone receptors 1-3, which allow for 'naked-eye' detection of cyanide anions with high selectivity and sensitivity have been developed. The addition of CN^- to acetonitrile solutions of 1-3 induced obvious color, UV-vis and fluorescence change.

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Appendix A. Supplementary matrial

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.saa.2013.07.041.

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