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Research Article

A Novel Thiourea-Based Sensor: Synthesis and Recognition for Acetate Anion

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Abstract

Here in a series of novel and colorimetric chemosensors based on (trans)-1.2-cyclohexanediamine and thiourea group were designed and synthesized, and their application in anion recognition was investigated. The chemsensor 4b showed higher selectivity for the detection of acetate ion than other anion such F^-, Br^-, I^-, ClO_4^-, $H_2PO_4^-$ in DMSO. The chemsensor 4b displayed immediate visible changes from nearly colorless toyellow and yellow-green upon the addition of acetate anion.

Keywords: Anion Sensor; Thiourea; Acetate Anion; Colorimetry; Trans-1.2-Cyclohexanediamine

Introduction

The development of colorimetric and fluorescent chemosensors for the detection of various anions has attracted much attention due to the importance of anions in biological, industrial, food and environmental process [1-8]. Among the chemosensors, colorimetric sensors could be close to be applied in practical project, in which the detection of various anions by color change with those receptors needed less instruments and costed less [9], especially for real-time and online analysis of analytes. Most of that artificial receptor for the recognition of anions were mainly based on the combined nonconvalent interactions by using the dominant N-H function groups and neutral and cationic C-H hydrogen bond donors [10]. As a result, those receptors incorporating groups such as amide [11-16], urea [17-18], pyrrole [19-20] and thiourea [21-23] as binding units were designed and reported. Some of these colorimetric receptors for acetate ion in organic media such as DMSO have been reported [24-26]. On the other hand, among various anions, acetate ion has been found to be a possible tracer for maligences and have been extensively investigated in prostate cancer and metastases [27]. Moreover the rate of the acetate ion production and oxidation has been proved to be vital as an indicator of organic eletrocomposition in marine sediments [28], in which context, the development a novel colorimetric and fluorescent chemosensor for the rapid, convenient and low-cost detection of biologically important anions such as acetate was required.

While during the last few years, a large number of sensors for acetate anion have been reported, it was still a challenge to find easily synthesized, highly sensitive and selective to acetate anion and novel receptor.

Experimental

Materials

All reagents for synthesis were obtained commercially and used without further purification. In the titration experiments, all the anions were added in the form of tetrabutylammonium (TBA) salts, which were also commercially available, and stored in vacuum desiccators containing self-indicating silica and dried fully before use. DMSO was dried with CaH_2 and then distilled under reduced pressure.

General method

^{1}H NMR spectra were recorded on a Bruker AV-300 spectrometer or a Bruker AV-500 spectrometer at room temperature. Chemical shifts (in ppm) were referenced to tetramethylsilane (δ = 0 ppm) in $CDCl_3$ or DMSO-d_6 as an internal standard. ^{13}C NMR spectra were obtained by the same NMR spectrometer and were calibrated with DMSO-d_6 (δ =39.00ppm). Data for ^{1}H NMR were reported as follows: chemical shifts (δ ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet or unresolved, br s = broad singlet), coupling constant (Hz) and integration. Data for ^{13}C NMR were reported in terms of chemical shift and multiplicity where appropriate. Mass spectra were performed on an Aglient 6530 Q-TOF for HRMS. The yields were determined on a METTLER TOLEDO ME 104 balance (accuracy: 0.1mg): Flash column chromatography was carried out on silica gel (200-300 mesh). UV-vis spectra were recorded on UV-2450.

Synthesis

Synthesis of 1a: 1a-1 was prepared according to the literature [29].

To a solution of 1a-1 (0.492g 2.4mmol) in anhydrous DMF, were added sequentially at room temperature: N_2H_4-2HCl (0.106g, 1mmol) HOBt (0.35g, 2.4mmol), DIPEA (1.4ml, 8.0mmol), EDCI (0.46g, 2.4mmol). The mixture was stirred at room temperature for 24 hours and diluted with EtOAc (40ml). The mixture was washed with water, 1% HCl and saturated sodium chloride and dried with Na_2SO_4. After filtration, evaporation of the solvent gave and the crude product which was further purified by silica gel (eluent: petrol: EA = 2:1) to afford the white solid (yield: 68%)

1a ^{1}H-NMR (DMSO-d_6, 500MHz) δ 9.74-9.5(2H, s, NH); 7.45-7.40(4H, d, J =7.43 Hz, ArH); 7.36-7.32(4H, t, J = 7.34 Hz, ArH); 7.29-7.25(2H, t, J=7.27 Hz, ArH); 4.03-3.97(2H, d, J = 4.0Hz, CH); 3.46-3.41(2H, d, J = 3.44Hz, CH); 3.22-3.16(2H, m, CH); 2.90-2.85 (2H, m, CH); 2.31-2.23(2H, m, CH); 2.13-2.05(2H, m, CH); 1.90-1.82(2H, m, CH), 1,80-1.71(4H, m, CH_2); ^{13}C-NMR(300 MHz, DMSO-d_6) δ

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170.72, 138.75, 128.34, 128.05, 126.864, 67.76, 58.078, 52.480, 29.36, 23.02; ESI-MS: calculated for $C_{24}H_{30}N_4O_2$ [M+Na]+: 429.2 ; observed 429.3.

Synthesis of 2a, 3a: 2a and 3a were prepared according to the literature [30-31].

2a, white solid, yield: 98%:

^{1}H-NMR (300MHz, DMSO-d6): δ 8.55-8.47 (2H, d, J = 8.59Hz, ArH), 8.26-8.16 (2H, br s, CONH), 8.14-8.00 (4H, d, J = 8.06, ArH), 7.84-7.66 (2H, t, J = 7.73, ArH), 7.40-7.28 (2H, t, J = 7.33Hz, ArH) , 4.17-3.90 (2H, s, CH), 2.27-2.18 (2H, m, CH), 1.89-1.79 (2H, m, CH_2), 1.52-1.43 (4H, m CH_2); ^{13}C-NMR (300MHz, DMSO-d_6): δ 163.6, 149.7, 148.2, 137.5, 126.3, 121.7, 52.3, 31.6, 24.4; ESI-MS: calculated for $C_{18}H_{20}O_2N_4$ [M+Na]$^+$ 347.1, found 347.1.

3a**,** white solid, yield: 90%:

^{1}H-NMR($CDCl_3$ 500 MHz):δ 8.65-8.47 (2H, br s, CONH), 8.28-8.14 (6H, m, ArH), 7.85-7.79 (2H, d, J = 7.82Hz, ArH) 7.79-7.74(2H, t, J = 7.77 Hz, ArH), 7.63-7.56(2H, t, J = 7.69 Hz, ArH), 4.66-3.98 (2H, s, CH), 2.41-2.32(2H, s, CH), 2.00-1.87(2H, s, CH), 1.72-1.49 (4H, m, CH_2); ^{13}C-NMR($CDCl_3$ 300MHz):δ 149.8,145.8,137.6,130.4, 129.0, 128.6, 127.9, 118.4, 52.8, 31.6, 24.5, ESI-MS: calculated for $C_{26}H_{24}N_4O_2$ [M+Na]$^+$ 447.1, found 447.2

Synthesis of 2b and 3b: To a solution of 2a or 3a (1mmol) in CH_2Cl_2 (10mL) was added m-CPBA (383mg, 2.2mmol) at 0°C under stirring. After the reaction was completed, the mixture was concentrated and purified by the basic aluminum oxide column chromatography (eluent: EtOAc/MeOH = 3:1) to give a white powder 2b or 3b.

2b white solid, yield: 65%:

^{1}H-NMR(300MHz,$CDCl_3$) δ 11.62-11.22(2H, br s, CONH), 8.49-8.34(2H, d, J = 8.40Hz, ArH), 8.28-8.12(2H, d, J = 8.19Hz, ArH), 7.45-7.26 (4H, m, ArH), 4,42-3.98 (2H, s, CH), 2.30-2.16 (2H, s, CH), 1.87-1.73(2H, s, CH), 1.58-1.40(4H, m, CH_2); ^{13}C NMR δ 159.4, 140.7, 140.4, 128.9, 127.0, 126.7, 52.5, 31.7,24.2. ESI-MS: calculated for $C_{18}H_{20}N_4O_4$ [M+Na]$^+$: 379.1; observed 379.1.

3b white solid, yield: 57%:

^{1}H-NMR(DMSO-d_6, 300MHz) δ 11.78-11.49 (2H, s, CONH), 8.81-8.63 (2H, m, NH), 8.40-8.33 (2H, m, ArH), 7.86-7.79 (6H, m, ArH); 7.74-7.65(2H, m, ArH), 4.51-4.25 (2H, s, CH), 2.42-2.17 (2H, m, CH_2), 1.89-1.80 (2H, m, CH_2), 1.66-1.57 (2H, m, CH_2); 1.41-1.23 (2H, m, CH_2); ^{13}C NMR δ160.3, 141.7, 137.4, 130.7, 130.5, 129.5, 127.9, 125.8, 122.7, 120.2, 52.6, 31.9, 24.3; ESI-MS: calculated for $C_{26}H_{24}N_4O_4$ [M+Na]$^+$: 479.1; observed 479.0

Synthesis of 4a and 4b: 4a-1 and 4b-1 were preparation according to literature [32].

To a solution of 4a-1 or 4b-1 (1mmol) in anhydrous THF (5ml) was added (1R,2R)-1,2-cyclohenxanediamine (0.5mmol). The resulting mixture was stirred at room temperature for 12 hours. Evaporation of the solvent gave the crude product, which was purified by flash column chromatography.

4a, white solid, yield: 70%:

^{1}H-NMR (500MHz, DMSO-d_6) δ12.02-11.83(2H, s, NH), 10.48-10.33(2H, s, NH), 8.25-8.18(2H, d, ArH, J = 8.25 Hz), 7.75-7.66(2H, t, ArH, J = 7.71 Hz), 7.10-7.03(2H, d, ArH, J = 7.07 Hz), 7.03-6.95(2H, t, ArH, J = 7.00Hz), 4.59-4.39(2H, m, NCH), 2,32-2.22(2H, m, CH), 1.76-1.70(2H, m, CH_2), 1.48-1.39(4H, m, CH_2); ^{13}C-NMR(300 MHz, DMSO-d6) δ 179.35, 183.66, 145.55, 138.20, 117.52, 112.28, 63.76, 28.74, 23.49; ESI-MS: calculated $C_{18}H_{22}N_6S_2$ [M+Na]$^+$ 409.1, observed 409.1.

4b, yellow-white solid, yield: 69%:

^{1}H-NMR (500MHz,DMSO-d_6): δ 11.87-11.19(2H, s, NH), 10.93-10.34(2H, s, NH) 8.52- 8.04(2H, d, ArH, J = 8.25Hz), 7.98-7.88(2H, dd, ArH, J = 7.83 Hz), 7.41-6.90(2H, d, ArH, J = 7.11Hz), 4.77-4,34(2H, s, NCH), 2.38 2.19(2H, s, CH), 1.81-1.69(2H, s, CH_2), 1.47-1.32(4H, m, CH_2); 13C-NMR(500 MHz, DMSO-d_6) δ 179.1, 151.9, 143.7, 138.6, 123.5,113.8, 57.7, 31.2, 24.0; ESI-MS: calculated for $C_{18}H_{20}Cl_2N_6S_2$ [M+H]$^+$ 455.0; observed 455.1 (Figure 1).

Result and Discussion

UV–vis spectral observations and Colorimetric studies

To determine the selectivity of those receptors to various anion, UV-vis spectral experiment were conducted using standard solution of the receptors 1a, 2a, 2b, 3a,3b,4a and 4b in dry DMSO by adding up to 10 equiv of tetrabutylammonium salts as acetate anion and recording the absorbance changes.

As was showed in Figure 2, receptors 1a, 2a, 2b, 3a, 3b, 4a in DMSO with AcO^-, had only one absorption band, while the receptor 4b with AcO^- had two absorption bands, which one absorption band is at 337nm, the other is at 444nm. Additionally, the colorimetric properties of those receptors were explored to establish if there were changes could be observed by naked eye and the findings were depicted in Figure 3. It was observed that receptor 4b with AcO^- displayed a distinct color change, which the solution changed from colorless to yellow or yellow-green.

Based on receptor 4b with AcO^- exhibiting distinct color changes, further experiments about receptor 4b were conducted.

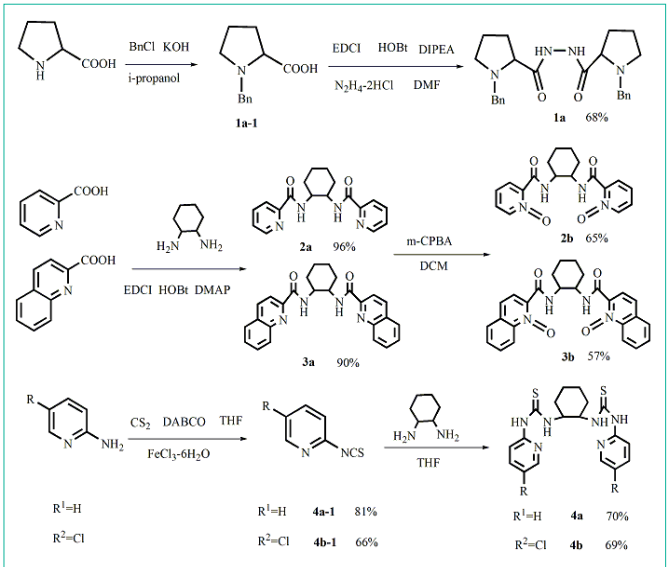

Figure 1: synthesis of 1a, 2a, 2b, 3a, 3b, 4a, 4b.

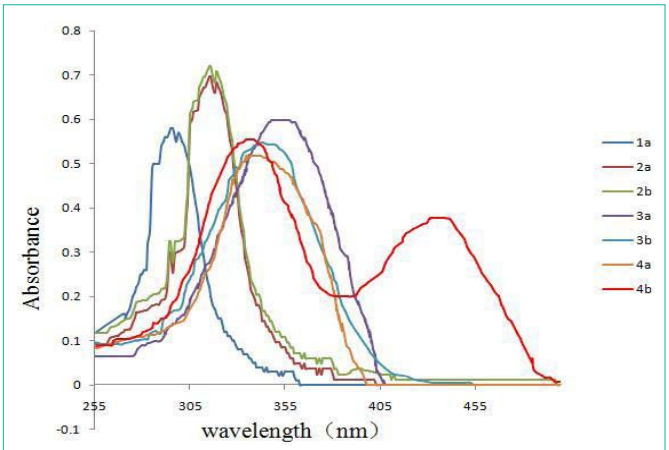

Figure 2: UV-vis absorption spectra of receptors (1a,2a,2b,3a,3b,4a,4b) (5×10^{-5} M in DMSO) with 8 equiv of TBA-AcO.

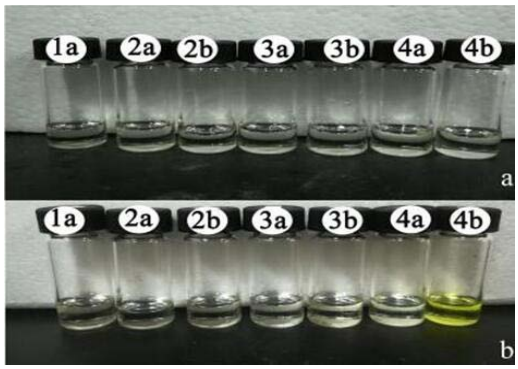

Figure 3: (a) color changes observed for receptors (1a,2a,2b,3a,3b,4a,4b) (5×10^{-3} M in DMSO) (b) color changes observed for receptors (1a,2a,2b,3a,3b,4a,4b) (5×10^{-3} M in DMSO) upon addition of 1.0 equiv of TBA-AcO at room temperature.

It was observed in Figure 4 that receptor 4b, as the free receptor molecular, exhibited one main absorption band at 337nm. Upon the addition of 10 equiv of the various anions to the receptor 4b solution, the absorption at 337nm decreased gradually and a new absorption band at 444 nm emerged, especially upon the addition of the acetate ion. Other anions ($ClO4^-$, I^-, Br^-) were faintly responsive at 444nm, while F^- and $H_2PO_4^-$ did not show noticeable decrease in absorbance at 337nm.

Moreover, as was shown in Figure 5, when addition of 0.01 equiv of F^-, it did not exhibit a distinct color changes while AcO^- still had noticeable color change. Those result showed that 4b was able to detect acetate ion in the range of 10^{-5}-10^{-3} M. Also, 4b with acetate exhibited yellow, while other anions with 4b did show noticeable color change at 365nm.

On the other hand, on addition of increasing amounts of tetrabutylammmonium salts of acetate ion to the solution of receptor 4b, the absorption band at 337nm decreased while formation a distinct isosbestic point at 361 nm (Figure 6). The observation of an isosbestic point in the UV-vis spectra indicated the formation of a stable complex between the host and guest.

To further study the complex of the host and guest, Job spot was also done. The Job plot showed that the ratio of receptor 4b with acetate ion is 1:1 (Figure 7).

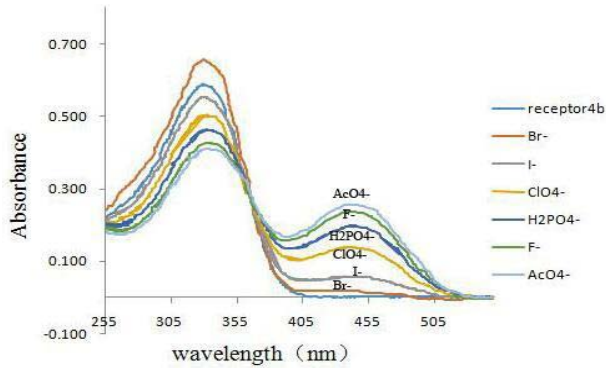

Figure 4: UV-vis spectra of 4b (2.0×10^{-5} M solution in DMSO) upon the addition of 8 equiv of each tetrabutylammonium salts (TBA-X) (X= F^-, Br^-, I^-, AcO^-, $H_2PO_4^-$, ClO_4^-).

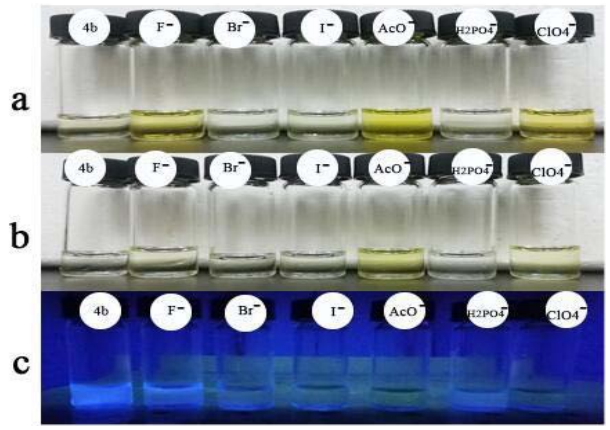

Figure 5: Color observed 4b (Y M solution in DMSO) upon the addition of 1.0 equiv of various anions as TBA-X(X=F^-, Br^-, I^-, AcO^-, ClO_4^-, $H_2PO_4^-$), and the first 4b solution was observed without addition of any anions. (a) Y= 5×10^{-3} ; (b) Y= 5×10^{-5} ; (c) Y= 5×10^{-3} , detected at 365nm.

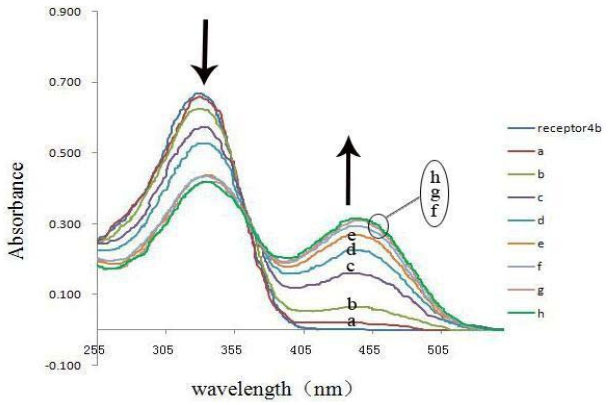

Figure 6: UV-vis absorption spectra titration of receptor 4b (5×10^{-5} M in DMSO) with x equiv of TBA-AcO at room temperature: (a) x= 1.0; (b) x=2.0; (c) x= 3.0; (d) x=4.0; (e) x= 5.0; (f) x= 6.0; (g) x=7.0; (h) x= 8.0.

^{1}H-NMR titration experiments

The interaction of the anion with the receptor NH protons

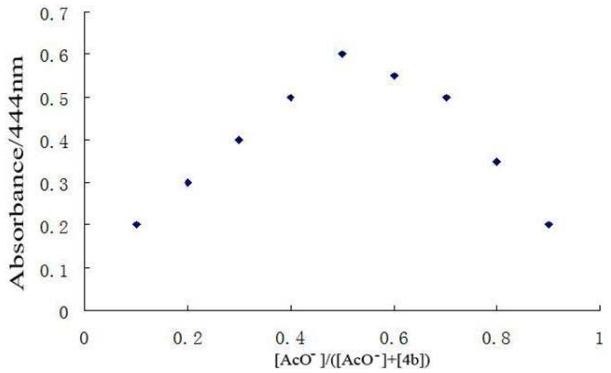

Figure 7: The stoichiometry analysis of the complex [4b-AcO$^-$] in DMSO by Job plot analysis; [4b]+[AcO$^-$]= 2.0×10^{-5} M.

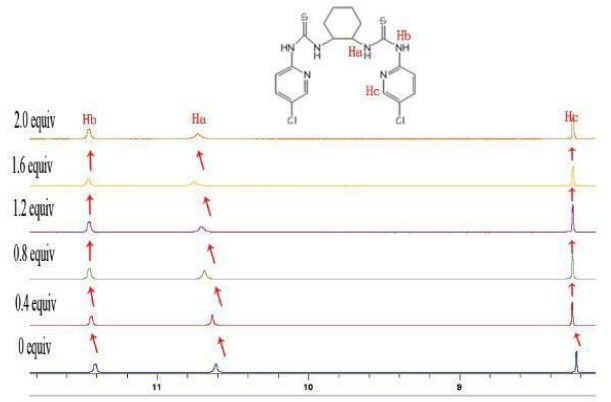

Figure 8: ^{1}H-NMR titration spectra of receptor 4b in DMSO-d6 upon addition of x equiv of TBA-AcO; x= 0; 0.4; 0.8; 1.2; 1.6; 2.0.

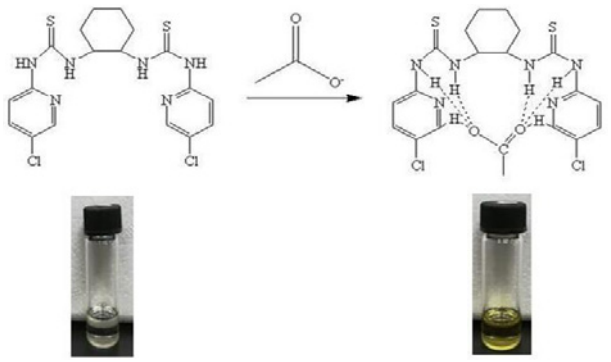

Figure 9: Proposed mechanism of formation the complex of 4b and AcO$^-$.

through hydrogen bond interaction is known to result in line broadening of the concerned ^{1}H-NMR signals, and is usually followed by a downfield shift of the resonances of these protons with increasing concentrations of anions [33]. As was shown in Figure 8, in order to gain an insight into the interaction details of receptor 4b with guest anion, the ^{1}H-NMR titration of 4b with AcO$^-$ were conducted. Free receptor 4b exhibited three signal peaks at 11.40ppm, 10.61ppm and 8.22ppm in the far downfield region, attributed to Ha, Hb, Hc respectively. Upon addition of 1.0 equiv of AcO$^-$, clear downshifts (0.05ppm, 0.11ppm, 0.03ppm) for Ha, Hb, Hc respectively were observed. Hence, the added AcO$^-$ was bound by 4b, through hydrogen bond interactions, from thiourea and aromatic hydrogen.

Based on the facts above, we considered the stable complex might attribute to the six hydrogen-binding with the acetate anion (Figure 9).

Conclusion

In summary, a novel colorimetric anion sensor which allowed for "naked eye" detection and UV-VIS detection of acetate anion with high sensitivity and selectivity has been designed and synthesized. The urea group was exploited as an anion binding site in 4b, and it utilizes hydrogen-binding to form a 1:1 host and guest complex as a mode of recognition AcO$^-$, which could be concluded by the Job plot analysis. And ^{1}H-NMR titration of receptor 4b with AcO$^-$ confirmed the N-H hydrogen bonding interaction mode.

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