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# A highly selective fluorescent probe for Al<sup>3+</sup> based on quinoline derivative



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## HIGHLIGHTS

- The receptor HL shows 286-fold enhancement of fluorescence intensity in the present of Al<sup>3+</sup>
- The detection limit of HL for Al<sup>3+</sup> could reach at  $10^{-7}$  M level.
- A new chemosensor for Al<sup>3+</sup> via the photo-induced electron transfer effect have been reported.

## G R A P H I C A L A B S T R A C T

Possible PET process of HL.



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## Introduction

Aluminum is the third most abundant element in the Earth's crust (approximately 8% of total mineral components) [1]. Its compounds are widely used in food additives, cookware, drinking water supplies, antiperspirants, deodorants, bleached flour, and antacids [2,3]. Nevertheless, as a non-essential element for living system, the extensive use of aluminum not only are deadly to growing plants but could induce many diseases, such as

ABSTRACT

A novel Schiff base fluorescent probe, 1-phenyl-3-methyl-5-hydroxypyrazole-4-carbaldehyde (2'-methyl quinoline-4'-formyl) hydrazone (PMHCH), for selective detection of Al<sup>3+</sup> has been designed and synthesized. Upon addition of various metal ions, the receptor only shows 286-fold enhancement of fluorescence intensity which might be attributed to a 1:1 stoichiometry between PMHCH and Al<sup>3+</sup> and the photo-induced electron transfer progress in the present of Al<sup>3+</sup> at 505 nm. More importantly, the detection limit of PMHCH for Al<sup>3+</sup> could reach at 10<sup>-7</sup> M level.

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Parkinson's disease and Alzheimer's disease [4-8]. According to The World Health Organization (WHO), the average daily human intake of Al<sup>3+</sup> of around 3–10 mg and the weekly tolerable dietary intake of 7 mg kg<sup>-1</sup> body weight [9]. The determination of very low levels of aluminum has become increasingly very important in environmental and clinical chemistry since its negative role in the human life. Therefore, it is desirable to develop some new analytical methods for detecting Al<sup>3+</sup>.

Among all detection techniques [10–13], fluorescent chemosensors have attracted considerable attention due to its easily detectable signals upon recognition of metal ions with high sensitivity and selectivity [14–17]. But the poor coordination ability, strong

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hydration ability, and the lack of spectroscopic characteristics of Al<sup>3+</sup> have hindered development of a suitable fluorescence sensor compared to other metal ions [18]. Previous reports indicate that structures of most fluorescent probes for Al<sup>3+</sup> contain nitrogen and oxygen-rich coordination environments providing a hard base environment for the hard acid Al<sup>3+</sup> [19]. Therefore, design of selective and sensitive chemo-sensors for Al<sup>3+</sup> remains desirable. Schiff bases are known to form stable complexes with metal ions and act as ion carriers. The structures of Schiff bases contain nitrogen-oxygen-rich coordination environments which provide a hard-base environment for the hard-acid Al<sup>3+</sup>. The resulting Schiff base complexes have attracted increasing attention in the area of ionic binding due to their unique properties and reactivity [20,21].

Here, we have reported a Schiff base ligand 1-phenyl-3-me thyl-5-hydroxypyrazole-4-carbaldehyde (2'-methylquinoline-4'- f ormyl) hydrazone (PMHCH) as a chemosensor for Al<sup>3+</sup> via the photo-induced electron transfer (PET) effect. The free receptor PMHCH almost did not show fluorescence emission at 515 nm when it was excited at 375 nm. The intensity dramatically enhanced (286-fold) in the present of Al<sup>3+</sup>, other ions such as K<sup>+</sup>, Na<sup>+</sup>, Mg<sup>2+</sup>, Ca<sup>2+</sup>, Cr<sup>2+</sup>, Ba<sup>2+</sup>, Mn<sup>2+</sup>, Cu<sup>2+</sup>, Co<sup>2+</sup>, Pb<sup>2+</sup>, Zn<sup>2+</sup>, Hg<sup>2+</sup>, Fe<sup>2+</sup> and Fe<sup>3+</sup> did not have much influence. Moreover, the detection limit of PMHCH for Al<sup>3+</sup> could be as low as  $2.2 \times 10^{-7}$  M in ethanol.

## Experimental

#### Materials and instrumentation

All chemicals were of reagent grade quality, obtained from commercial sources, and were used as received without further purification. Melting points were determined using a Beijing XT4-100x microscopic melting point apparatus. <sup>1</sup>H-NMR spectra were recorded on JNM-ECS 400 MHz instruments spectrometers with TMS (tetramethylsilane) as internal standard and DMSO-d<sub>6</sub> as solvent. Mass spectra were recorded in methanol solvent on a Bruker Esquire 6000 spectrometer. UV-vis absorption spectra were obtained with a Shimadzu UV-240 spectrophotometer and recorded in quartz cells with 1 cm optical path length. Emission spectra were measured using a Hitachi RF-5301 fluorimeter.

## Synthesis of the probe PMHCH

1-Phenyl-3-methyl-4-formyl-pyrazolone-5 (PMFP) was synthesized as reported procedure [22]. Ethyl 2-methyl quinoline -4-carboxylate was synthesized according to the method reported [23]. The synthetic route of PMHCH is shown in Scheme 1.

#### *Synthesis of 2-methyl quinoline-4-carboxylic hydrazide* [24]

To a solution of ethyl 2-methyl quinoline-4-carboxylate (5 mmol, 1.015 g) in ethanol (40 mL) was added hydrazine hydrate (80%, 4 mL) dropwise. Then the reaction mixture was stirred at 80 °C for 12 h under reflux. After evaporating the solvent in a vacuum, the precipitate was filtered and stand overnight in refrigerator. A white needle crystalline solid was obtained and then recrystallized with ethanol to get the final product. Yield: 53%; m.p.: 178–179 °C. <sup>1</sup>H NMR (Fig. S1): (400 MHz; CDCl<sub>3</sub>):  $\delta$  = 2.71 (s, 3H<sub>e</sub>), 4.25 (s, 2H<sub>h</sub>), 7.29 (s, 1H<sub>f</sub>), 7.54 (m, 1H<sub>b</sub>), 7.59 (s, 1H<sub>g</sub>), 7.73 (m, 1H<sub>c</sub>), 8.05 (d, J = 8.25 Hz, 1H<sub>d</sub>), 8.14 (d, J = 8.25 Hz, 1H<sub>a</sub>).

## Synthesis of PMHCH

An ethanol solution (30 mL) of 2-methyl quinoline-4-carboxylic hydrazide (0.402 g, 2 mmol) was added dropwise to a solution (30 mL) of 1-phenyl-3-methyl-4-formyl-pyrazolone-5(PMFP) (0.404 g, 2 mmol) in ethanol. The reaction mixture was heated to reflux for 8 h with stirring, during which time a green precipitated



Scheme 1. Synthetic route of PMHCH.

formed immediately. Following reaction, the mixture was allowed to cool to room temperature. The precipitated was filtered, washed with cold absolute ethanol. The crude product was purified by recrystallization from ethanol to give 0.484 g of PMHCH as a light-green solid. Yield: 62%; m.p.: 253–255 °C. IR (KBr, cm<sup>-1</sup>): 3118, 1583, 1387, 1281 (Fig. S2). <sup>1</sup>H NMR (Fig. S3): (400 MHz; DMSO-d<sub>6</sub>):  $\delta$  = 8.26 (m, 1H), 8.14 (s, 1H), 8.00 (d, J = 7.4 Hz, 1H), 7.96 (d, J = 7.4 Hz, 1H), 7.75 (m, 2H), 7.61 (m, 2H), 7.370 (m, 2H), 7.305 (m, 1H), 7.09 (t, J = 7.4 Hz, 1H), 4.44 (s, 1H), 2.69 (s, 3H), 2.20 (d, J = 7.4 Hz, 3H).  $^{13}$ C NMR (Fig. S4): (400 MHz; DMSO-d<sub>6</sub>):  $\delta = 163.23, 161.17, 159.12, 158.78, 151.17, 140.00, 139.95,$ 139.09, 138.43, 130.60, 129.28, 127.11, 122.86, 121.03, 119.60, 118.11, 100.00, 98.55, 25.21, 13.23. ESI-MS (Fig. S5): m/z calcd for C<sub>22</sub>H<sub>19</sub>N<sub>5</sub>O<sub>2</sub> + H<sup>+</sup>: 386.15 [M+H<sup>+</sup>]: found, 386.11. Anal. Calcd for C<sub>22</sub>H<sub>19</sub>N<sub>5</sub>O<sub>2</sub>: C, 68.56; H, 4.97; N, 18.17. Found: C, 68.14; H, 4.24; N, 18.13.

## **Results and discussion**

#### General information

Solutions of metal ions were prepared from the corresponding metal nitrate salts. Stock solutions of metal ions were prepared by dissolving the desired amount of material in ethanol. Test solutions were prepared by placing 20  $\mu$ L of the probe stock solution into cuvettes, adding an appropriate aliquot of each ions stock, and diluting the solution to 2 mL with ethanol solutions. For fluorescence measurements, the excitation and emission slit widths were 5 nm and 3 nm, respectively.

The association constant for PMHCH–Al<sup>3+</sup> complex is further estimated on the basis of the nonlinear filtting of the fluorescence titration curve assuming a 1:1 stoichiometry by the Benesi–Hildebrand method [25,26].

$$\frac{1}{F - F_{\min}} = \frac{1}{K(F_{\max} - F_{\min})\left[\mathsf{Al}^{3+}\right]} + \frac{1}{F_{\max} - F_{\min}}$$

 $F_{\min}$ , F, and  $F_{\max}$  are the emission intensities of the organic moiety considered in the absence of Al (III) ions, at an intermediate aluminum concentration, and at a concentration of complete interaction, respectively. And K is the binding constant concentration.

The detection limit is estimated from the fluorescence titration. The emission intensity of the complex (PMHCH–Al<sup>3+</sup>) without any anion is measured to determine the S/N ratio [27,28]. And the standard deviation of blank measurements is calculated. The detection limit is calculated based on  $3 \times \delta_{\text{blank}}/k$ . Where  $\delta_{\text{blank}}$  is the

standard deviation of the blank solution and k is the slope of the calibration plot.

### UV-vis analysis

We have carried out UV–vis titration experiments in ethanol at room temperature to understand the nature of binding of PMHCH to Al<sup>3+</sup>. As shown in Fig. 1, upon gradual addition of Al<sup>3+</sup>, a concomitant 16 nm red shift in the spectral position at 375 nm was observed along with an increase in the absorption intensity. A well-defined isosbestic point could be clearly observed at 368 nm, indicating a balance between PMHCH and PMHCH–Al<sup>3+</sup>. These phenomenons indicated the formation of a new complex between ligand PMHCH and Al<sup>3+</sup> [29].

## Fluorescence study

The effect of reaction time on the fluorescence spectra was studied and the results were shown in Fig. S6. Adding 1 equiv. of  $Al^{3+}$  to ethanol solution of PMHCH, 1 min later the emission at 505 nm of the system got to maximum and did not increase with more reaction time. It means PMHCH could be used as a real-time  $Al^{3+}$  chemosensor.

Fluorescence titration experiments were also performed in ethanol. As shown in Fig. 2, the free receptor PMHCH almost not show fluorescence emission at 515 nm when it was excited at 375 nm ( $\Phi_0$  = 1.82). Upon successive addition of Al<sup>3+</sup> ion to the solution of receptor PMHCH, a new emission band with a maximum at 505 nm appeared and the intensity of this band gradually increased with an increasing concentration of  $Al^{3+}$  ( $\Phi_{Al} = 4.62$ ). The response of the probe toward the addition of  $Al^{3+}$  was immediate and the intensity dramatically enhanced (286-fold) which can be attributed to the decrease of PET effect and the chelation-enhanced fluorescence (CHEF) effect [30,31]. More specifically, the free receptor displayed very weak fluorescence band at 515 nm which was due to quenching by the carbonyl group through a photo-induced electron transfer (PET), induced by a lone pair electron from the Schiff base. Upon the addition of Al<sup>3+</sup>, the chelation of the oxygen and nitrogen atom with Al<sup>3+</sup> resulted in the efficient inhibition for the PET process of the -C=N group as shown in Scheme 2.

The Job's plot obtained for PMHCH-Al<sup>3+</sup> system in ethanol clearly suggests a formation of 1:1 metal-ligand stoichiometry [32] (Fig. 3). To confirm further the stoichiometry between PMHCH and the Al<sup>3+</sup>, mass spectra analysis was conducted



Fig. 1. Changes in the absorption spectra of PMHCH (10  $\mu M)$  in ethanol after addition of  $Al^{3*}$  (1–9 equiv.).



**Fig. 2.** Fluorescence spectra of PMHCH (10  $\mu$ M) with addition of increasing amount of Al<sup>3+</sup> (0–19  $\mu$ M) in ethanol. Excitation wavelength was 375 nm, and emission was at 505 nm. Inset: color of PMHCH (left) and PMHCH + Al<sup>3+</sup> (right) under UV lamp. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)



Scheme 2. Possible PET process of PMHCH.



Fig. 3. Job's plot of PMHCH with  $Al^{3\ast}$  in ethanol solution. Total concentration of PMHCH +  $Al^{3\ast}$  was kept constant at 10  $\mu$ M, the emission at 505 nm was used.

(Fig. S7). A mass peak at m/z 473.07 corresponding to [PMHCH + Al<sup>3+</sup> + NO<sub>3</sub><sup>-</sup> - H<sup>+</sup>]<sup>+</sup> is indicative of the formation of a 1:1 complex. According to linear Benesi-Hildebrand expression,



**Fig. 4.** Emission spectra of PMHCH (10  $\mu$ M) upon addition of different mental ions (1 equiv. K<sup>+</sup>, Na<sup>+</sup>, Mg<sup>2+</sup>, Ca<sup>2+</sup>, Cr<sup>2+</sup>, Ba<sup>2+</sup>, Mn<sup>2+</sup>, Cu<sup>2+</sup>, Co<sup>2+</sup>, Pb<sup>2+</sup>, Zn<sup>2+</sup>, Hg<sup>2+</sup>, Fe<sup>2+</sup>, Fe<sup>3+</sup>and Al<sup>3+</sup>) in ethanol.



**Fig. 5.** Changes in fluorescence emission intensity at 505 nm of PMHCH (10.0  $\mu$ M) in the presence of various metal ions in ethanol. Black bars represent the addition of metal ions written below the bars to a 10  $\mu$ M solution of PMHCH. Red bars represent emission intensity of a mixture of PMHCH (10  $\mu$ M) with the metal ions written below the bars (10  $\mu$ M) following addition of 10  $\mu$ M Al<sup>3+</sup> to the solutions. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

the association constant between PMHCH and  $Al^{3^{+}}$  was calculated from the fluorescence titration result and was found to be  $1.89\times10^{4}\,M^{-1}$  [33,34] (Fig. S8). For practical applications, the detection limit of PMHCH was also estimated from the titration experiment (Fig. S9). The detection limit for  $Al^{3^{+}}$  was calculated to  $2.2\times10^{-7}\,M$ , which is far below the WHO acceptable limit (0.05 mg  $L^{-1}$  or  $1.85\,\mu M$  of  $Al^{3^{+}}$ ) in drinking water.

We also investigated the selectivity profile of PMHCH in response to other metal ions (Fig. 4). Upon the addition of other metal ions, such as K<sup>+</sup>, Na<sup>+</sup>, Mg<sup>2+</sup>, Ca<sup>2+</sup>, Cr<sup>2+</sup>, Ba<sup>2+</sup>, Mn<sup>2+</sup>, Cu<sup>2+</sup>, Co<sup>2+</sup>, Pb<sup>2+</sup>, Zn<sup>2+</sup>, Hg<sup>2+</sup>, Fe<sup>2+</sup> and Fe<sup>3+</sup>, no detectable change in the emission intensity of PMHCH was found except Mg<sup>2+</sup>. Only Mg<sup>2+</sup> can enhance the fluorescent intensity, yet the intensity was far less than that of Al<sup>3+</sup>. This means that the ligand PMHCH has excellent fluorescence selectivity toward Al<sup>3+</sup> over all other metal ions.

To check the practical ability of chemosensor PMHCH as an effective  $Al^{3+}$  ion fluorescent chemosensor, competitive experiments in the presence of  $Al^{3+}$  ions mixed with other different metal

ions have been carried out (Fig. 5). PMHCH responding for  $Al^{3+}$  in the presence of  $Mg^{2+}$ ,  $Cd^{2+}$  and  $Zn^{2+}$  are relatively low but clearly detectable. As for as  $Fe^{2+}$ ,  $Cu^{2+}$ , those ions are typical ions that have a pronounced quenching effect on fluorophores by mechanisms inherent to the paramagnetic species [35,36]. This result indicated that PMHCH is a selective fluorescent sensor toward  $Al^{3+}$  over most competitive cations.

Furthermore, the reversibility nature of chemosensor PMHCH was measured by the titration of EDTA with fluorescent probe (PMHCH +  $Al^{3+}$ ) (Fig. S10). The addition of 1 equiv. of EDTA to a solution of PMHCH– $Al^{3+}$  complex resulted in its emission intensity decreased dramatically, which indicates the regeneration of the free PMHCH. The result indicated that sensor PMHCH could be a reversible chemosensor for  $Al^{3+}$ .

To understand better the complexation behavior of PMHCH with Al<sup>3+</sup>, <sup>1</sup>H NMR experiments were carried out in d<sub>6</sub>-DMSO (Fig. S11). Upon addition of Al<sup>3+</sup>, the NH proton of PMHCH at around 7.75 ppm was shifted downfield toward 7.87 ppm. At the same time, the signal for -CH=N- was shifted downfield by 0.08 and the hydrogens of quinoline were shifted downfield slightly followed the addition of Al<sup>3+</sup>. The results suggested that the binding of probe PMHCH to Al<sup>3+</sup> forms a rigid system by a chelation with the nitrogen of imine-nitrogen and the oxygen atom of carbonyl group.

## Conclusion

In conclusion, we have presented the synthesis, characterization, and spectral properties of PMHCH, a new fluorescent sensor for  $AI^{3+}$  with high sensitivity. This phenomenon was attributed to forms 1:1 complex which inhibit photo-induced electron transfer process and chelation-enhanced fluorescence precess. The detection limit for  $AI^{3+}$  can reach at  $10^{-7}$  M level. Further study will focus on enhancing the water solubility of the receptor and its potential applications in biological chemistry. The design strategy and remarkable photo physical properties of the sensor would help to extend the development of fluorescent sensors for other ions.

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## Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.saa.2015.05.041.

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