



# A fluorescence turn-on sensor for aluminum ion by a naphthaldehyde derivative



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## ARTICLE INFO

### Article history:

Received 5 May 2015

Received in revised form

27 December 2015

Accepted 28 December 2015

Available online 30 December 2015

### Keywords:

Al<sup>3+</sup> ion

Chemosensor

Naphthaldehyde derivative

Detection limit

Turn-on

## ABSTRACT

A new sensor, 1-((2-hydroxynaphthalen-1-yl)methylene)thiourea (**NT**) was synthesized and characterized by infrared spectra (FI-IR), elemental analysis (EAs), Electrospray ionization mass spectra (ESI-MS). The fluorescent sensing behaviors of the **NT** toward different metal ions, anions and amino acids were determined by fluorescence spectroscopy. The receptor **NT** exhibited 73-fold increase in fluorescence intensity in the presence of Al<sup>3+</sup> over most other competitive metal ions, anions and amino acids. Moreover, the probe is very sensitive to detect Al<sup>3+</sup> with a detection limit 0.11 μM.

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## 1. Introduction

Aluminum (Al<sup>3+</sup>) is a polyvalent cation and the third abundant metal in the earth's crust and is widespread in air, water and soil due to various sources, such as water treatment, food additives, medicines and the production of light alloys in modern society [1–7]. Once absorbed in the human body, the accumulation of aluminum in human body can damage normal tissues and cells and result in human health problems, such as Alzheimer's disease [6,8–10], Parkinson's disease [11], lung cancer, breast cancer, and bladder cancer [10,12]. Thus, Al<sup>3+</sup> detection has attracted considerable attention and a variety of detection strategies have been developed for Al<sup>3+</sup>, such as chromatography [13], graphite furnace atomic absorption spectrometry (GFAAS) [14], neutron activation analysis (NAA) [15], inductively coupled plasma-mass spectrometry (ICP-MS) [16], ablation microprobe mass analysis (LAMMA) [17], etc.

However, these methods are time-consuming, require highly trained personnel and sophisticated instrumentation, especially not suitable to real-time monitoring. So it is meaningfully to develop a simple and an inexpensive method to detect Al<sup>3+</sup> with desirable for real-time monitoring of environmental, biological, and industrial samples.

Accordingly, during the past decade, considerable efforts have been devoted to developing fluorescent probes for Al<sup>3+</sup>, because of fluorescent detection is the most efficient method, operational simplicity, low cost, real time monitoring and high sensitivity. Among various fluorescent probes, the fluorophores employed as signal reporters of fluorescent probes mainly focused on Rhodamine [18–24], Thiazole [25–27], Quinolone [28–34], AZO dyes [35], Curcumin [36,37], Macrocyclic (C<sub>34</sub>H<sub>35</sub>N<sub>3</sub>O<sub>2</sub>) [38], Chromone [39,40], Tetrazole [41], Juloidine-imine [42], coumarin [43], pyridoxal [44,45], etc. there are also two naphthalene derivatives were reported to detect Al<sup>3+</sup> [46–49], but the selectivity is poor.

Therefore, we designed and synthesized a kind of 1-((2-hydroxynaphthalen-1-yl)methylene)thiourea (**NT**) in this work. And the recognition ability of **NT** on various metal ions, anions and amino acids were studied by fluorescence and UV–vis spectroscopies firstly. The result showed that **NT** was a high selective fluorescence probe for Al<sup>3+</sup> over other metal ions, anion ions and

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amino acids.

## 2. Experimental

### 2.1. Materials and instrumentation

Melting point was determined on a WRX-4 apparatus in a sealed capillary. FT-IR spectra ( $4000\text{--}400\text{ cm}^{-1}$ ) were obtained on NICO-LET380 FT-IR spectrometer in KBr disks. The fluorescence spectra were obtained on Varian Cary ESe-NipSe-N fluorescence spectrometer with the excitation and emission slits are 2.5 nm. Elemental analyses (EAs) were carried out with a VARI-EL elemental analyzer. Electrospray ionization mass spectra (ESI-MS) were measured on a Finnigan LCQ system in methanol/water solution. All chemicals used were of analytical grade and were used as received without any further purification and were obtained from Sigma–Aldrich and Alfa Aesar Chemical Reagent Co. All metal salts were introduced as their chlorate salts. Anions were purchased as sodium salts or potassium salts.

### 2.2. General fluorescence spectra measurements

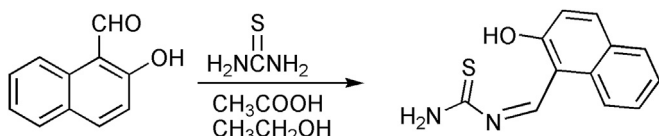
Fluorescence measurements were measured from 360 to 600 nm with slit width of 2.5/5 nm ( $\lambda_{\text{ex}} = 390\text{ nm}$ ). The solutions of metal ions  $\text{Zn}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Na}^+$ ,  $\text{Mg}^{2+}$ ,  $\text{K}^+$ ,  $\text{Hg}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Co}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Bi}^{3+}$ ,  $\text{Ba}^{2+}$ ,  $\text{Al}^{3+}$ ,  $\text{Ag}^+$  were prepared from their chlorate salts. The solutions of anions  $\text{SO}_4^{2-}$ ,  $\text{SO}_3^{2-}$ ,  $\text{PO}_4^{3-}$ ,  $\text{NS}^-$ ,  $\text{NO}_3^-$ ,  $\text{MnO}_4^-$ ,  $\text{IO}_3^-$ ,  $\text{HPO}_4^-$ ,  $\text{HCO}_3^-$ ,  $\text{H}_2\text{PO}_4^-$ ,  $\text{F}^-$ ,  $\text{CrO}_4^{2-}$ ,  $\text{Cr}_2\text{O}_7^{2-}$ ,  $\text{CO}_3^{2-}$ ,  $\text{CNS}^-$ ,  $\text{ClO}_3^-$ ,  $\text{Cl}^-$ ,  $\text{C}_2\text{O}_4^{2-}$ ,  $\text{Br}^-$ ,  $\text{B}_4\text{O}_7^{2-}$  were prepared from their sodium salts or potassium. The amino acids Ile, Val, Asp, Ser, Pro, Tyr, Lys, Arg, Met, Gln, Glu, Gly, Ala, Phe, Leu were purchased from Sigma–Aldrich. All of the metal ions, anions and amino acids were dissolved in water.

### 2.3. Synthesis of probe NT

0.344 g (0.002 mol) 2-hydroxy naphthalene formaldehyde was added to 6.00 mL ethanol with heating until it was dissolved. Then 0.152 g (0.002 mol) of thiourea in 7.00 mL water and 0.4 mL acetic acid was added drop-wise into the mentioned solution above. After the resulting solution was refluxed for 15 min with a constant stirring, light yellow solid was obtained and washed with ethanol and ether, respectively, recrystallized from dimethylbenzene, dried in vacuo. Yield, 60.9%. mp.:  $118\text{--}120\text{ }^\circ\text{C}$ . Elemental Analysis Calcd for  $\text{C}_{12}\text{H}_{10}\text{N}_2\text{OS}$ : C%: 62.59; H%: 4.38; N%: 12.16. Found: C%: 62.66; H%: 4.36; N%: 12.13. IR ( $\text{cm}^{-1}$ , s strong, m medium, w weak): 3440 m,  $\nu(\text{O-H})$ ; 3320 m,  $\nu(\text{N-H})$ ; 1678s,  $\nu(\text{C-H})$ ; 1623 m,  $\nu(\text{C=N})$ . Exact mass for **NS**: 230.29, ESI-MS:  $[\text{NT} + \text{H}]^+$  ( $m/z$ , 231.3463) (Scheme 1).

### 2.4. Selectivity over metal ions

The fluorescence spectrum of **NT** not exhibited remarkable emission at 438 nm when the excitation wavelength is at 390 nm in  $\text{C}_2\text{H}_5\text{OH}/\text{H}_2\text{O}$  (v/v, 1:1). No obvious fluorescence intensity changes were observed in emission spectra of **NT** after addition of a wide range of environmentally and physiologically active metal ions,



Scheme 1. Synthesis of 1-((2-hydroxynaphthalen-1-yl)methylene)thiourea (**NT**).

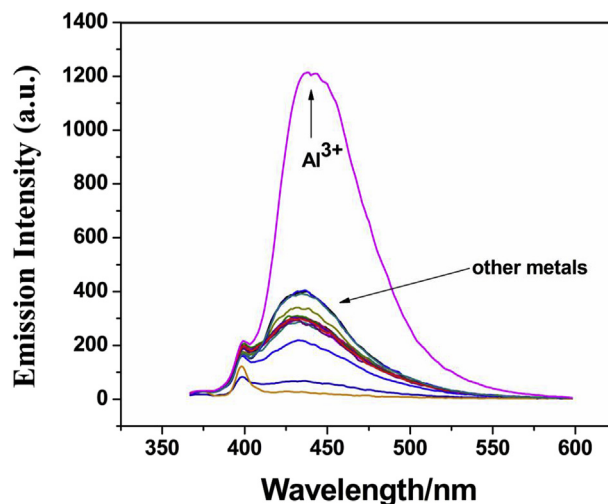


Fig. 1. Fluorescence spectra of **NT** ( $\lambda_{\text{ex}} = 390\text{ nm}$ ) with 80 equiv. of various metal ions (including  $\text{Al}^{3+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Na}^+$ ,  $\text{Mg}^{2+}$ ,  $\text{K}^+$ ,  $\text{Hg}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Co}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Bi}^{3+}$ ,  $\text{Ba}^{2+}$ ,  $\text{Ag}^+$ ) in  $\text{C}_2\text{H}_5\text{OH}/\text{H}_2\text{O}$  (v/v, 1:1).

such as  $\text{Zn}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Na}^+$ ,  $\text{Mg}^{2+}$ ,  $\text{K}^+$ ,  $\text{Hg}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Co}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Bi}^{3+}$ ,  $\text{Ba}^{2+}$ ,  $\text{Ag}^+$  (80 equiv.), except for enhanced by  $\text{Al}^{3+}$ . Fig. 1 shows fluorescence optical density of **NT** at 438 nm when various metal ions are added.

High selectivity toward the target analyte over the other potentially competitive species is a very important parameter to evaluate the performance of a probe. Therefore, the competition experiments in the presence of potentially competitive metal ions were also conducted by monitoring the change in fluorescence intensity at 438 nm upon addition of  $\text{Al}^{3+}$  ion to a solution of **NT** and different metal ions in  $\text{C}_2\text{H}_5\text{OH}/\text{H}_2\text{O}$  (v/v, 1:1). As shown in Fig. 2, it can be observed that none of the metal ions exerts virtually any effect on the fluorescence intensity. It is exhibited 73-fold increase in fluorescence intensity in the presence of  $\text{Al}^{3+}$  over most other competitive metals in  $\text{C}_2\text{H}_5\text{OH}/\text{H}_2\text{O}$  (v/v, 1:1), the result is better than early reported [50]. Therefore, the **NT** is capable of

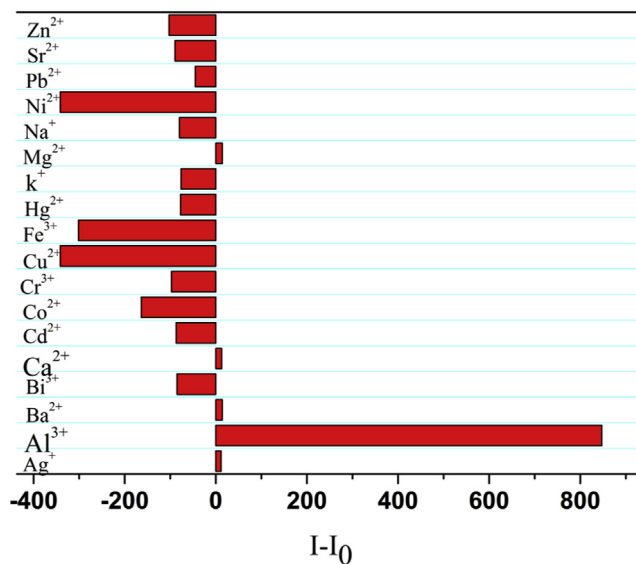


Fig. 2. Optical density two-dimensional graph of **NT** at 438 nm, respectively upon the addition of several metal ions (including  $\text{Al}^{3+}$ ,  $\text{Zn}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Ni}^{2+}$ ,  $\text{Na}^+$ ,  $\text{Mg}^{2+}$ ,  $\text{K}^+$ ,  $\text{Hg}^{2+}$ ,  $\text{Fe}^{3+}$ ,  $\text{Cu}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Co}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Bi}^{3+}$ ,  $\text{Ba}^{2+}$ ,  $\text{Ag}^+$ ),  $\lambda_{\text{ex}} = 390\text{ nm}$ .

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