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A dual colorimetric and fluorescent sensor for lead ion based on naphthalene hydrazone derivative

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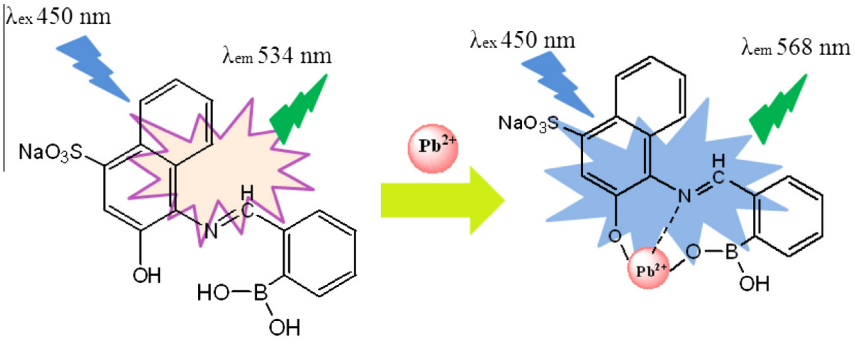
HIGHLIGHTS

- An easy-to-make colorimetric and fluorometric sensor **1** for Pb^{2+} was obtained.
- The assay carried in aqueous solution with low limit detection as 37 nM mol L^{-1}.
- The high selectivity and sensitivity of **1** for Pb^{2+} was for **1**'s distinctive structure.
- **1**–Pb^{2+} shows longer wavelength absorption and emission and strong fluorescence.

GRAPHICAL ABSTRACT

A 1:1 metal-ligand complex between lead ion and compound 1 was formed which resulted in color change from red to blue and enhancement of fluorescence at 568 nm.



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ABSTRACT

A new compound, 2-boronobenzaldehyde-(2′-hydroxyl-4′-sulfonic acid) naphthalene hydrazone (**1**), was synthesized and its structure was characterized by proton nuclear magnetic resonance, mass and element analyses. The presence of Pb^{2+} led **1** to undergo colorimetric and fluorescent changes, which were detectable with the naked eye. Thus, a dual spectral response for Pb^{2+} detection was introduced. In KH_2PO_4–NaOH buffer aqueous solution (pH 6.0), **1** exhibited fluorescence enhancement at 568 nm and hyperchromicity at 595 nm upon the addition of Pb^{2+}. The fluorescent intensity change was proportionate to the concentration of Pb^{2+} with a dynamic working range of 5.0×10^{-7} mol L^{-1} to 1.0×10^{-4} mol L^{-1} and a detection limit of 3.7×10^{-8} mol L^{-1}. The fluorometric method was successfully applied for the detection of Pb^{2+} water of Qianhu Lake and soil in Nanchang university campus. The recoveries were 111–116% for water and 97.6% for soil respectively, determined via the standard addition method.

Introduction

Among the metal ions, lead (Pb) is a highly toxic heavy metal ion that causes various adverse health problems, such as high blood pressure; nerve disorders; and reproductive, cardiovascular, muscular, and joint pains in adults and children [1–4]. Lead also causes environmental pollution. The main sources of lead pollution are mining, metal smelting, coal combustion, and the use of Pb-based paint, leaded gasoline, and Pb-containing pipes in water supply systems [5,6]. Thus, the concentration of Pb^{2+} in environmental is restricted. The U.S. Environmental Protection Agency sets the maximum contamination level for lead in drinking water at

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15 μg L^{-1} (75 nM) [7]. The US Center for Disease Control set the standard contamination level for Pb at 10–19 μg L^{-1} (50–95 nM) in blood [8]. Many instrumentally intensive methods for the detection of lead have been developed to protect human health and the environment, including inductively coupled plasma mass spectrometry [9,10], atomic absorption spectrometry [11,12], and anodic stripping voltammetry [13,14]. However, these techniques have some limitation, such as cumbersome sample preparation, expensive instruments, and costly maintenance, which often limit their use to the laboratory [15]. Therefore, the development of alternative assays with easy operation and low cost has sparked great interest. Fluorescent assays possess some advantages, such as easy-to-operate, low cost, and sensitivity, which have been widely used in the detection of metal ions analysis [16]. Some research groups have exerted great effort in the design and synthesis of many excellent fluorescent and colorimetric sensors for the detection of Pb^{2+} [17,18]. However, only a few sensors in which the binding of Pb^{2+} increases the fluorescence intensity have been reported [19–22]. Colorimetric sensors are among the Pb^{2+}-detecting sensors that have received considerable attention [23–25]. However, major improvements can still be made in terms of developing suitable sensors that can function in aqueous systems.

With these considerations in mind, we extend our earlier investigations [26] and report a new successful development of a 2-boronobenzaldehyde-(2′-hydroxyl-4′-sulfonic acid) naphthalene hydrazone (**1**), which exhibits fluorescent and colorimetric responses to Pb^{2+} in aqueous solution. Compound **1** contains boric acid and hydroxyl groups that are capable of chelating Pb^{2+} via coordination. Meanwhile, **1** has good solubility in water due to the sulfonic acid and boric acid groups. As described in our previous work, the quinoline group in compound **2** was replaced by the naphthalene group in **1**. **1**−Pb^{2+} showed more superior characteristics, such as longer wavelength of emission and absorption and lower detection limit compared with **2**−Pb^{2+}.

Experimental

Materials and reagents

2-Formylbenzeneboronic acid, 1-amino-2-naphthol-4-sufonic acid, and $Pb(NO_3)_2$ were purchased from Sigma–Aldrich (USA, www.sigmaaldrich.com). Other metal salts were the products of the Shanghai Qingxi Technology Co., Ltd. (Shanghai, China, www.ce-r.cn/sites/qingxi/). All reagents and solvents were of analytical grade and used without further purification. The solution of **1** (5.0×10^{-4} mol L^{-1}) was prepared with double-distilled water. The metal ion solutions of Na^+, K^+, Mg^{2+}, Ca^{2+}, Mn^{2+}, Fe^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Cd^{2+}, Cr^{3+}, Hg^{2+}, and Ag^+ (0.01 mol L^{-1}) were prepared from their chlorides or nitrates. The buffer solution (pH 6.0) was prepared using NaOH (0.1 mol L^{-1}) and KH_2PO_4 (0.2 mol L^{-1}).

Apparatus

Absorption spectra were collected on a Shimadzu-2550 ultraviolet–visible spectrophotometer (Japan,) using a 1.0 cm quartz cell. All fluorescence measurements were performed with a scan speed of 1200 nm min^{-1} on a Hitachi F-4600 spectrofluorimeter (Japan) equipped with a xenon lamp source and a 1.0 cm quartz cell. Proton nuclear magnetic resonance (^{1}H NMR) spectra were obtained with D_2O on a Bruker Advance 600 MHz NMR spectrometer (Switzerland) using tetramethylsilane as the internal standard. Electrospray ionization (ESI) mass spectrum was recorded using a Waters ZQ4000/2695 LC–MS spectrometer (USA). The element analysis data were obtained on Perkin-Elmer-240(II) (USA). All measurements were operated at room temperature (about 298 K).

*Synthesis of compound **1***

2-Formylbenzeneboronic acid (0.15 g, 0.001 mol) was dissolved with the appropriate amount of ethanol. 1-Amino-2-naphthol-4-sufonic acid (0.24 g, 0.001 mol) was dissolved with NaOH solution (0.1 mol L^{-1}), and the pH was adjusted to 7.0 by adding acetic acid. Above two kinds solution were mixed and refluxed for about 12 h at 80 °C. The resulting mixture was cooled, filtered, and washed with methanol and double-distilled water three times, and the product was dried in a vacuum-drying oven to obtain 0.79 g in 20% yield, which was confirmed by the data. ^{1}H NMR (D_2O, 600 MHz) (ppm): 5.568 (s, 1H, —CH=N—), 7.068 (t, 3H, Ar—H), 7.187–7.242 (m, 1H, Ar—H), 7.355 (s, 1H, Ar—H), 7.355 (s, 1H, Ar—H), 7.468 (t, 1H, Ar—H), 7.561 (d, 1H, Ar—H), 7.710 (d, 1H, Ar—H), 8.354 (d, 1H, Ar—H);ESI mass: m/e calcd. for $C_{17}H_{13}BNNaO_6S$ [M—H^-] 392.05, found [M—H^-] 392.50; Anal.: calcd. for $C_{17}H_{13}BNNaO_6S$: C, 51.93; H, 3.33; N, 3.56; O, 24.42; S, 8.16, found C, 51.63; H, 3.43; N, 3.76; O, 24.48; S, 8.06. (See Scheme 1)

Results and discussion

*Fluorescent spectral responses of compound **1** to different metal ions*

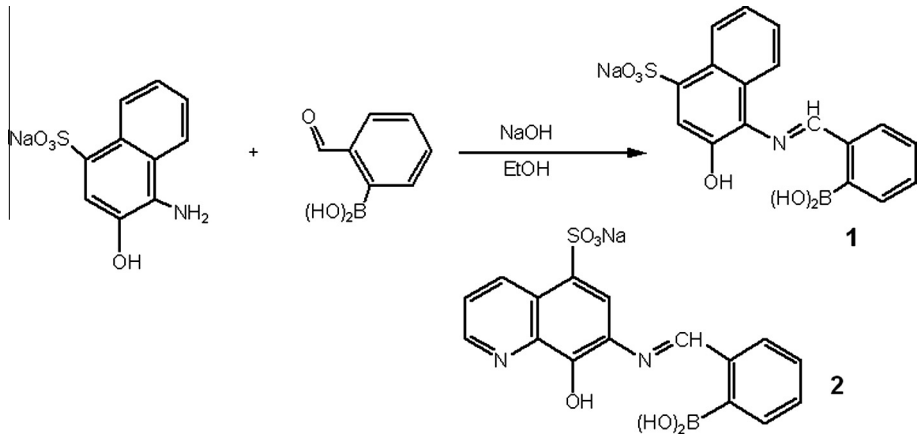
Compound **1** exhibits good solubility and unique fluorescent stability in water. Fig. 1 displayed the spectral change and intensity change of **1** in the presence and absence of various metal ions. In pH 6.0 buffer KH_2PO_4–NaOH aqueous solution, compound **1** emitted weak fluorescence at 534 nm. Upon the addition of various metal ions, such as Na^+, K^+, Mg^+, Ca^{2+}, Fe^{3+}, Pb^{2+}, Zn^{2+}, Cu^{2+}, Co^{3+}, Hg^{2+}, Ag^+, Mn^{2+}, and Ni^{2+}, only the presence of Pb^{2+} resulted in a remarkable spectral change. The emission intensity at 568 nm was enhanced by about eightfold upon the addition of Pb^{2+} (20 equiv.). However, the other metal ions showed little fluorescence enhancement. Thus, compound **1** could be used to detect Pb^{2+} with high selectivity.

Fluorescent titration of **1** against Pb^{2+} was performed, and the spectral changes are presented in Fig. 2a. The intensity change at 568 nm was proportionate to the Pb^{2+} concentration in the range of 0.005 mol L^{-1} to 1.0×10^{-4} mol L^{-1}. The linear regression equation was C (μmol/L) = 0.5291ΔF−1.046 (n = 17, R = 0.9983). The detection limit was calculated to be 3.7×10^{-8} mol L^{-1}. The enhancement of fluorescent intensity was presumably due to **1**−Pb^{2+} complex formation. Consequently, the binding ratio between **1** and Pb^{2+} was estimated as 1:1 via Job plot curve according to the fluorescent intensity change (Fig. 2b).

Some similar methods for determination Pb^{2+} were listed in Table 1. It is clear that the determination wavelength and linear range of proposed method are better compared with the other methods. Although the detection limit is not the best, it is fulfill the requirements because the detecting limit is lower than the maximum permitted amount of Pb^{2+} in drinking water defined by the World Health Organization [7]. It is worth to point that compound 1 as organic fluorescent probe is more easily obtained than the others and possesses good water solubility.

*3.2 Absorption spectral change for **1** upon the addition Pb^{2+}*

Compound **1** exhibits a red color in aqueous solution. As shown in Fig. 3, a solution of 5.0×10^{-4} mol L^{-1} **1** in aqueous solution at pH 6.0 KH_2PO_4–NaOH buffer solution exhibited an absorption band centered at 528 nm ($\varepsilon = 2.03 \times 10^3$ mol^{-1} L cm^{-1}). With increasing Pb^{2+}concentration, the absorbance of the maximum wavelength at 528 nm decreased, and a new absorption band centered at 603 nm emerged. Three isobestic points appeared at 419, 450, and 578 nm, which also suggested a stoichiometric complex formation between **1** and Pb^{2+}. Simultaneously, the change in intensity ratio (A_{603nm}/



Scheme 1. Syntheses of compound **1** and control compound 2.

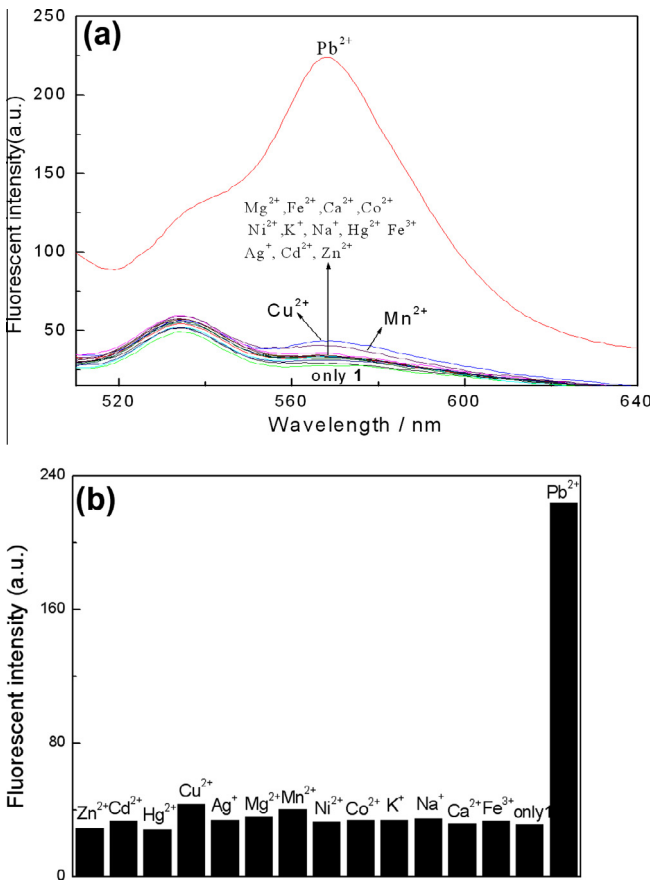


Fig. 1. (a) The fluorescence spectra of **1** (5.0×10^{-6} mol L^{-1}) and upon addition of various metal ions (1.0×10^{-4} mol L^{-1}) with an excitation at 450 nm. (b) Fluorescence density at 568 nm of **1** upon the addition of 1 equive. of different metal ions.

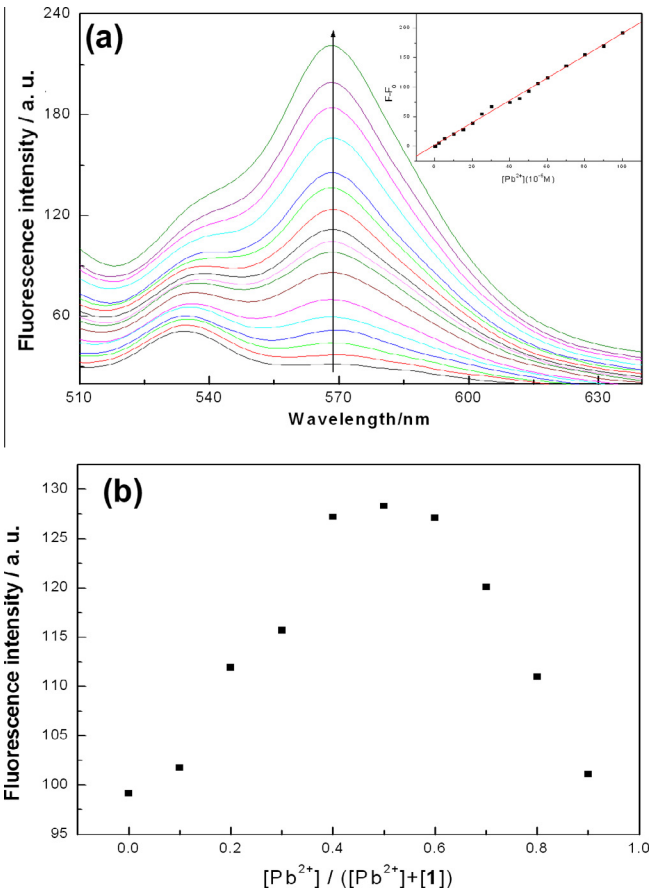


Fig. 2. (a) Fluorescence emission spectra of **1** (5.0×10^{-6} mol L^{-1}) in aqueous solution upon addition of Pb^{2+} in concentrations of 0, 2.0, 5.0, 10.0, 15.0, 20.0, 25.0, 30.0, 40.0, 45.0, 50.0, 55.0, 60.0, 70.0, 80.0, 90.0, 100.0 $\times 10^{-6}$ mol L^{-1}. (b) The Job-plot for the complex of **1** and Pb^{2+}, the total concentration of **1** and Pb^{2+} was 5.0×10^{-5} mol L^{-1}.

A_{528nm}) was proportionate to the Pb^{2+} concentration in the range of 0.05 mol L^{-1} to 8.0×10^{-6} mol L^{-1}. The linear regression equation was C (μmol L^{-1}) = $7.87A_{603nm}/A_{528nm}$ + 13.2, $R = -0.9859$, $n = 11$. The linear dynamic range of absorption spectrometry was narrower than that of fluorimetry.

Determining the binding constant of ***1***$-Pb^{2+}$

The binding constant (K_s) and the binding site numbers (n) can be calculated using the Benesi–Hildebrand equation [27] according to the absorption spectral change, as follows:

$$\ln[(A_0 - A_e)/(A_e - A_\infty)] = n\ln C_0 + \ln K_s$$

where A_0 is the absorbance intensity of **1**, A_e is the absorbance intensity of **1**$-Pb^{2+}$, and C_0 is the initial molar concentration of Pb^{2+}.

K_S and n can be derived from the intercept and slope obtained through the plot of $\ln[(A_0-A_e)/(A_e-A_\infty)]$ against $\ln C_0$. The fitted curve is shown in Fig. 4. The binding constant and the number of binding sites were 43028.16 and 1, respectively. The value of n was approximately equal to 1, indicating the existence of one

Table 1
Comparison of fluorescence enhancement sensor for Pb^{2+}.

Material	Monitor wavelengths (nm)	Conditions	Linear range	Detection limit (nm)	Reference
DNA device originates	594	pH 6.5, MOPS-NH_2OH buffer	0.02–10 μM	20	[21]
1,8-Naphthalimide	485	CH_3CN–H_2O	0–6 μM	50	[29]
DNA-based biosensor	587	Aqueous solution	0–0.96 μM	6	[30]
Peptide Tryptophan-Dansyl	495	Aqueous solution	0–4 μg/L	50	[31]
Bis(2-pyridylmethyl)amine	560	Aqueous solution	0–8.8 μM	19	[32]
Naphthalene hydrazone derivative	568	Aqueous solution	0.5–100 μM	38	This method

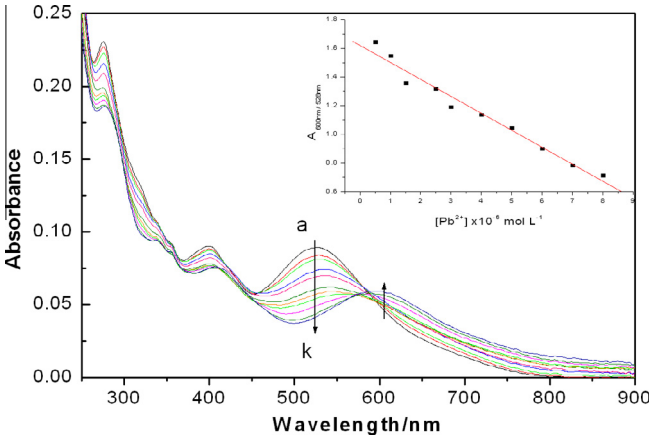


Fig. 3. UV–vis spectra of **1** (5.0×10^{-5} mol L^{-1}) in different concentrations of Pb^{2+} (a) 0, (b) 0.05, (c) 0.10, (d) 0.15, (e) 0.25, (f) 0.30, (g) 0.40, (h) 0.50, (i) 0.60, (j) 0.70, (k) 0.80×10^{-5} mol L^{-1}.

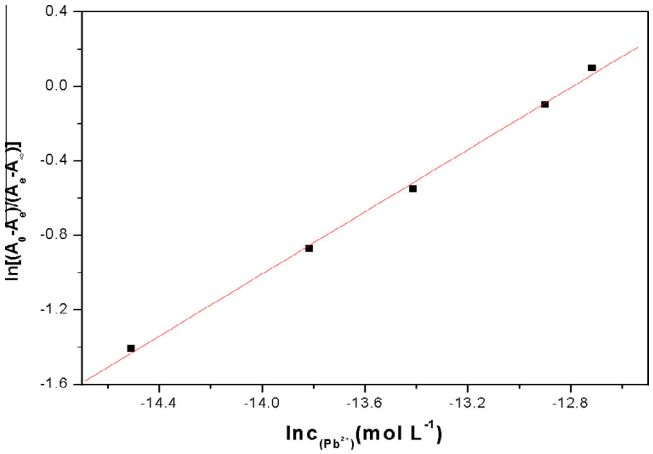


Fig. 4. Plot of fluorescence intensity change against the concentration of Pb^{2+}.

binding site for **1**, which is in agreement with the results of the Job plot. The obtained binding constant further suggested a strong binding force existing between **1** and Pb^{2+}, possibly from the strong force of the boric acid group. The binding parameters of **1**–Pb^{2+} and control complex of **2**–Pb^{2+} [26] are listed in Table 2 to explain the binding model. Compound **1** exhibits a highly sensitive spectral response to Pb^{2+} compared with control compound **2**.

Conclusions were drawn based on the structures of compounds **1** and **2**. First, the N atom in the quinoline group did not participate in the coordination with Pb^{2+} because the binding constant of **1**–Pb^{2+} was much larger than that of **2**–Pb^{2+}. Second, the position of the sulfonic acid groups affects the wavelength change of the Pb complex. The sulfonic acid group in compound **1** containing naphthalene is positioned at the para-position of oxime, which is beneficial for charge transfer, resulting in longer wavelength absorption and emission. Thus, according to the obtained results, chemosensor **1** is the most likely to chelate with Pb^{2+} via its O and N atoms and the —OH of the boric acid group (Scheme 2). The electron density of **1** was changed due to chelation with Pb^{2+}, which resulted in a remarkable color change from red to blue. The combination of atoms and space position maybe lead to the high selectively integration with Pb^{2+} over the other heavy metal ions like Hg^{2+}, Cd^{2+}, Cu^{2+}, and Ag^{+}.

Analytical application

Optimal experimental conditions

The effects of pH, reaction time, reaction temperature, and volume of ligand on the detection for Pb^{2+} were investigated. Optimal experimental conditions were obtained under room temperature in pH 6.0 KH_2PO_4–NaOH buffer solution and maintained for 5 min reaction time. The concentrations of compound **1** were 5.0×10^{-5} and 5.0×10^{-6} mol L^{-1} for absorption and emission, respectively. The fluorescent intensity and absorbance of **1**–Pb^{2+} in testing condition kept constant for at least 8 h.

Effect of foreign substances

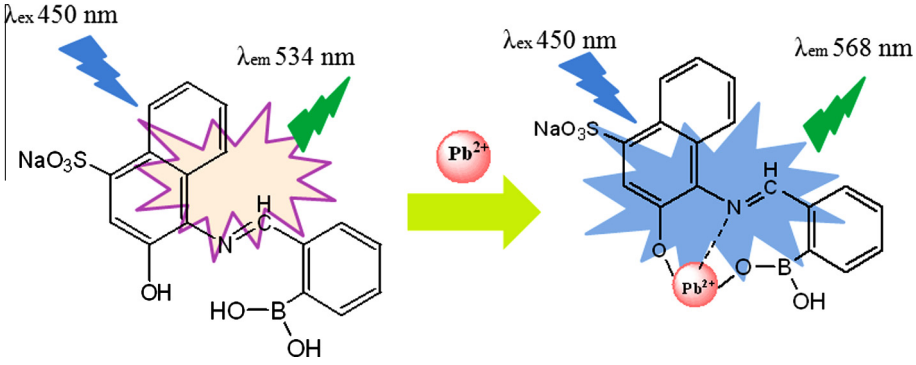
Under optimal experimental conditions, the presence of the following amounts of foreign substances in the mixture solution of 5.0×10^{-6} mol L^{-1} Pb^{2+} resulted in less than ±5% error compared with the Pb^{2+}concentration: 1000-fold Na^{+}, K^{+}, Cl^{-}, NO_3^{-}, SO_4^{2-}, and CO_3^{2-}, 100-fold Ca^{2+}, Mg^{2+}, Co^{2+}, Ni^{2+}, and $CH_3CO_2^{-}$, 50-fold Hg^{2+}, Mn^{2+} and Zn^{2+}, 10-fold Cu^{2+} and Fe^{3+}, 5-fold Cd^{2+} and Ag^{+}.

Analysis of real samples

The preliminary analytical application of this method was evaluated by determining Pb^{2+} in Qian Lake water and soil samples in Nanchang university campus. The results obtained are given in Table 3. The lake water samples were used without any treatment. The soil was treated as reference.[28] The recovery range was between 97.6% and 116%, which was in good agreement with Pb^{2+} obtained between spike and measured Pb^{2+}. The concentration of Pb^{2+} in soil obtained by our method is accordant with the determination value from atom absorption spectrum.

Table 2
Parameters of complexes of **1** and **2** with Pb^{2+}.

Complex	λax (nm)	λex/λem (nm/nm)	*Ka* (M^{-1})	Detection limit (M)	Linear range (M)
1–Pb	595	450/568	43 028	9.7×10^{-8}	$0.50–100 \times 10^{-6}$
2–Pb	590	360/477	1089	2.3×10^{-7}	$0.36–10 \times 10^{-6}$



Scheme 2. Proposed mechanism for the fluorescence changes of **1** upon addition of Pb^{2+}.

Table 3
Results for determination of Pb^{2+} in lake water and soil samples[a].

Sample	Pb^{2+} found (10^{-6} mol L^{-1})	Pb^{2+} added (10^{-6} mol L^{-1})	Total Pb^{2+} (10^{-6} mol L^{-1})			Average (10^{-6} mol L^{-1})	RSD (%)	Recovery (%)
	2.409	1.500	4.478	4.049	4.615	4.381	0.295	112
Lake water	2.409	2.500	6.186	5.832	5.134	5.718	0.535	116
	2.409	5.000	8.187	8.160	8.346	8.231	0.101	111
Soil	6.620[b]	5.000	10.82	11.05	12.16	11.34	0.72	97.6

[a] The determination results of Pb^{2+} were obtained by fluorometric method.
[b] The concentration of Pb^{2+} in soil was 6.793×10^{-6} mol L^{-1} obtained via atom absorption spectrum.

4. Conclusions

A simple-structured and fluorescence-activating sensor **1**,2-boronobenzaldehyde-(2′-hydroxyl-4′-sulfonic acid) naphthalene hydrazone, was easily synthesized through a one-step reaction. However, the structure was distinctive. The boronic acid group guaranteed good water solubility and a binding site in the coordination of **1**−Pb^{2+}. The sulfonic acid group and its position play key functions. The sulfonic acid group in compound **1** was placed at the para-position of oxime, which enhanced charge transfer, resulting in longer wavelength absorption and emission. Meanwhile, the sulfonic acid group is also hydrophilic. Therefore, a novel Pb^{2+} sensor based on dual spectral response was introduced, and emission and color changes were easily detected with the naked eye in aqueous solutions.

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