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A highly selective quinoline-based fluorescent sensor for Zn(II)

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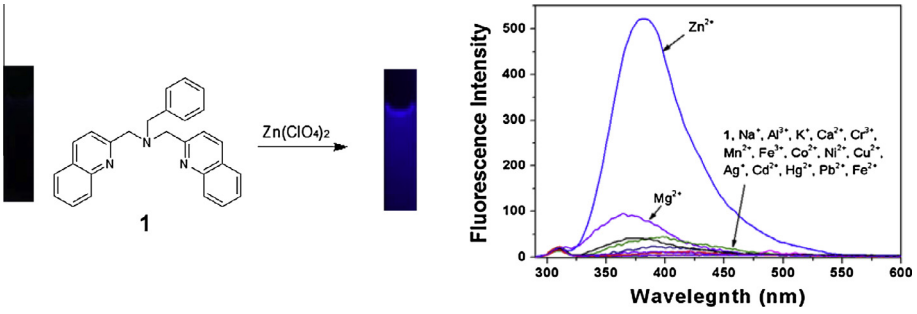
HIGHLIGHTS

- Quinoline-based receptor **1** was synthesized.
- The receptor **1** showed high selectivity for Zn^{2+} over other various cations.
- The system exhibited turn-on fluorescence via PET and CHEF.

GRAPHICAL ABSTRACT



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ABSTRACT

A quinoline-based simple receptor (bis(2-quinolinylmethyl)benzylamine = **1**) as a Zn^{2+} selective fluorescent chemosensor showed a large fluorescent enhancement with a blue shift in the presence of Zn^{2+} which is attributed to a chelation enhanced fluorescence (CHEF) effect with inhibition of a photoinduced electron transfer (PET) process of **1**. In particular, this receptor could clearly distinguish Zn^{2+} from Cd^{2+}. The binding mode of **1** and Zn^{2+} was found to be a 1:1 and confirmed by Job plot, ^1H NMR titration and ESI-mass spectrometry analysis.

Introduction

As the second most abundant transition metal ion after iron in the human body, zinc is believed to be an essential factor in many biological processes such as brain function and pathology, gene transcription, immune function, and mammalian reproduction [1–4]. In addition, Zn^{2+} is indispensable for mediating many enzyme-catalyzed reactions and therefore plays a very important role in a wide variety of physiological and pathological processes [5,6]. It is also an essential nutrient and strongly influences cell division and differentiation and hence is very important for the growth and development of all forms of life [7]. Recent studies indicate that elevated levels of Zn^{2+} in the human body have been implicated in neurodegenerative disorders among others [8,9]. Disruption of Zn^{2+} concentration in cells may be associated with a variety of diseases including Alzheimer's disease, epilepsy and infantile diarrhea [10–14]. Therefore, detection of Zn^{2+} is crucial in controlling its concentration levels in the biosphere and its direct impact on human health.

Fluorescent zinc sensors include fluorescein [15,16], coumarin [17,18], dansylamide [19,20], BODIPY [21], quinoline [22–25], and other fluorophores [7,26–30]. In particular, much attention has been recently paid to quinoline-based fluorescent zinc sensors, because the quinoline moiety acts as both metal binding ligand and fluorophore by means of CHEF [31].

It is a huge challenge to discriminate Zn^{2+} from Cd^{2+} through convenient methods with a simple probe, because Zn^{2+} and Cd^{2+} with similar physical properties often respond together with similar spectral changes [32,33]. Recently, we have showed that a

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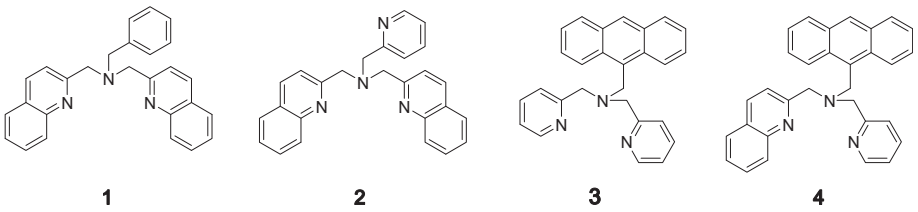


Scheme 1. Structures of receptors **1**, **2**, **3**, and **4**.

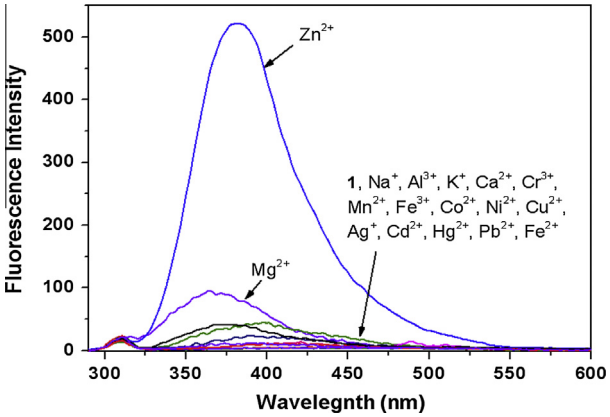


Fig. 1. Fluorescence spectra of **1** (20 μM) upon addition of 1 equiv of Na^+, Mg^{2+}, Al^{3+}, K^+, Ca^{2+}, Cr^{3+}, Mn^{2+}, Fe^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Ag^+, Cd^{2+}, Hg^{2+}, Pb^{2+} and Fe^{2+} in CH_3CN (λ_{ex} = 311 nm).

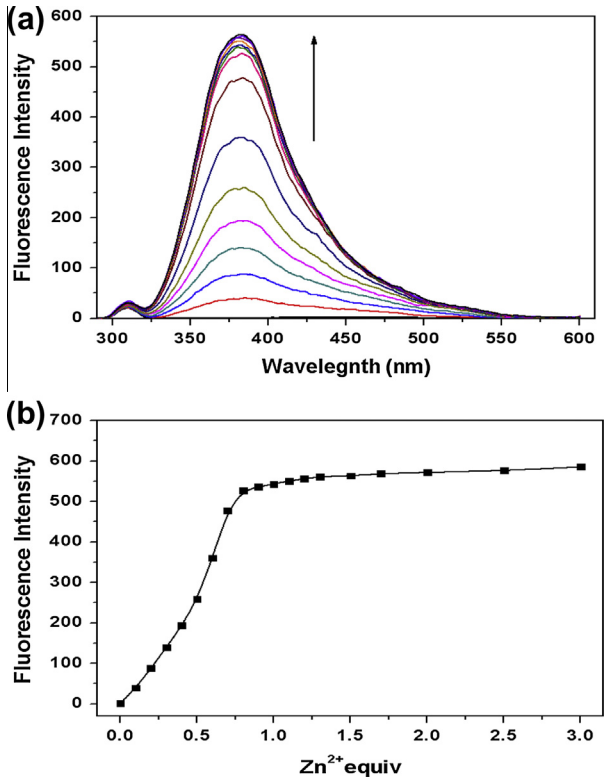


Fig. 2. (a) Fluorescence spectra of **1** (20 μM) in the presence of different concentrations of Zn^{2+}. (b) Fluorescence intensity at 382 nm of **1** as a function of the Zn^{2+} concentration.

small change of the substituent on a receptor afforded a very different sensing property. For example, the receptor **3** did not completely distinguish Zn^{2+} from Cd^{2+} [34], while the receptor **4** synthesized by displacement of a pyridine of **3** with a quinoline could clearly discriminate Zn^{2+} from Cd^{2+} (Scheme 1) [35]. As an another trial, therefore, we chose the receptor **2**, which was used as a Zn^{2+} sensor but could not completely discriminate Zn^{2+} from Cd^{2+} [36]. We planned to switch the pyridyl group of **2** with the phenyl group in order to observe the degree of the discrimination of Zn^{2+} from Cd^{2+}. Interestingly, the resulting receptor **1** clearly distinguished Zn^{2+} from Cd^{2+}.

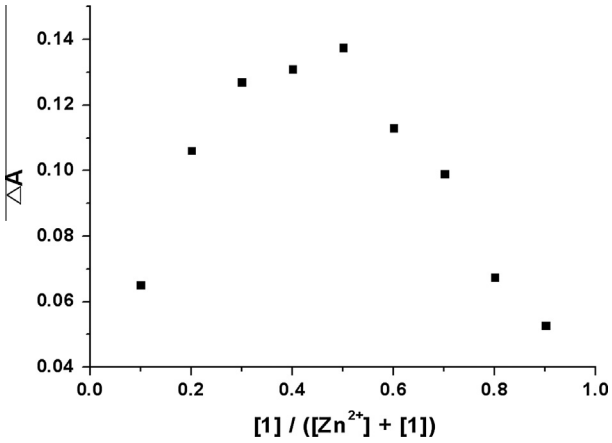


Fig. 3. Job plot for the binding of **1** and Zn^{2+}. The total concentration of **1** with Zn^{2+} was 10 μM.

Herein, we report on a highly selective chemosensor **1**, which recognizes Zn^{2+}. The treatment of Zn^{2+} to the solution of **1** induced highly turn-on fluorescence with a blue shift. In particular, **1** could discriminate Zn^{2+} from Cd^{2+}. Moreover, **1** could detect Zn^{2+} without any significant inhibition in the presence of competing metal ions.

Experimental

Reagents

All the solvents and reagents (analytical grade and spectroscopic grade) were obtained commercially and used as received.

Instrumentation

NMR spectra were recorded on a Varian 400 spectrometer. Chemical shifts (δ) are reported in ppm, relative to tetramethylsilane $Si(CH_3)_4$. Absorption spectra were recorded at room temperature using a Perkin Elmer model Lambda 2S UV/Vis spectrometer. The emission spectra were recorded on a Perkin–Elmer LS45 fluorescence spectrometer. Electrospray ionization mass spectra (ESI-MS) were collected on a Thermo Finnigan (San Jose, CA, USA) LCQTM Advantage MAX quadrupole ion trap instrument.

*Synthesis of bis(2-quinolinylmethyl)benzylamine (**1**)*

1 was synthesized by coupling of 2-(chloromethyl)quinoline hydrochloride and benzylamine via one step according to the liter-

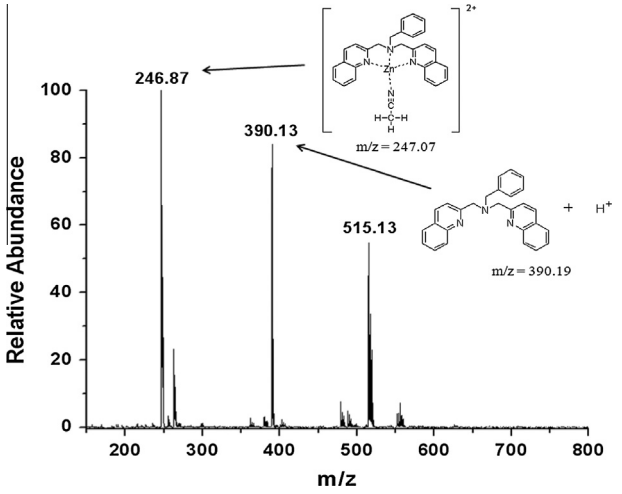
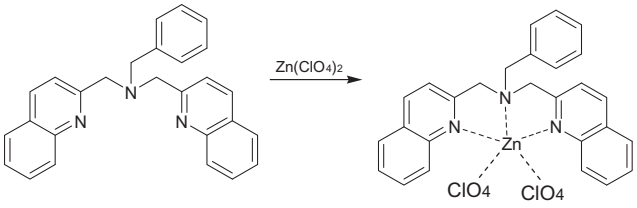


Fig. 4. Positive-ion electrospray ionization mass spectrum of **1** (1.0×10^{-4} M) upon addition of 1 equiv of Zn^{2+} in CH_3CN.



Scheme 2. Proposed structure of **1**-Zn^{2+} complex.

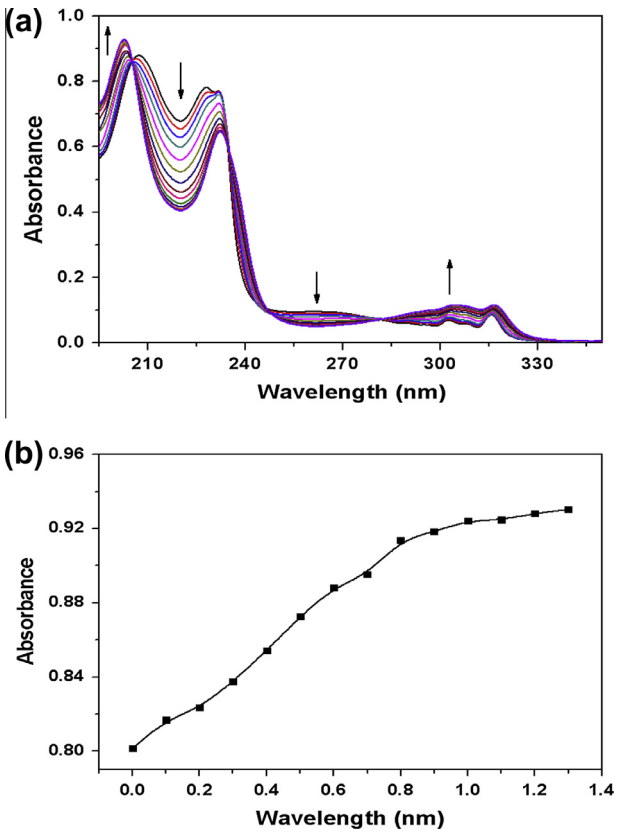


Fig. 5. (a) UV–vis absorption spectra of **1** (20 μM) upon addition of Zn^{2+} in CH_3CN. (b) Absorption at 203 nm as a function of Zn^{2+} concentration.

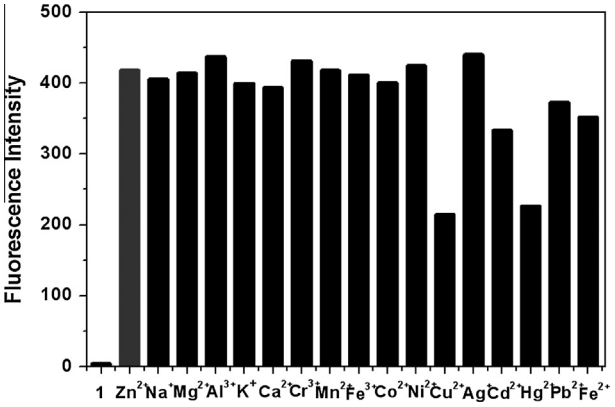


Fig. 6. Fluorescence intensity of **1** (20 μM) induced by various metal ions. Gray bar represents emission intensity of **1** in the presence of 1 equiv of Zn^{2+}. Black bars stand for fluorescence change that occurs upon addition of 1 equiv of Na^+, Mg^{2+}, Al^{3+}, K^+, Ca^{2+}, Cr^{3+}, Mn^{2+}, Fe^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Ag^+, Cd^{2+}, Hg^{2+}, Pb^{2+} and Fe^{2+} in the presence of Zn^{2+}.

ature method [37]. 2-(Chloromethyl)quinoline hydrochloride (0.46 g, 2.1 mmol) and benzylamine (0.11 mL, 1 mmol) were dissolved in 10 mL of water and heated to 70 °C. To this solution 5 mL of aqueous NaOH (0.17 g, 4.2 mmol) were added dropwise over a period of 30 min and the resulting mixture was stirred for an additional 2 h. The cooled solution was extracted three times with 20 mL of ethyl acetate, the combined organic layers were dried with Na_2SO_4, filtered and the solvent was removed in vacuo. The product was obtained as yellowish brown oil. Yield: 0.3753 g (96.4 %). ^{1}H NMR (DMSO-d_6, 400 MHz): δ 8.35 (d, J = 8.4 Hz, 2H), 7.97 (d, J = 8.4 Hz, 2H), 7.93 (d, J = 8.0 Hz, 2H), 7.78 (d, J = 8.8 Hz, 2H), 7.72 (t, J = 8.0 Hz, 2H), 7.55 (t, J = 7.60 Hz, 2H), 7.45 (d, J = 7.6 Hz, 2H), 7.34 (t, J = 7.6 Hz, 2H), 7.24 (t, J = 7.2 Hz, 1H), 3.89 (s, 4H), 3.67 (s, 2H); HRMS (ESI) m/z calcd for $C_{28}H_{24}N_2O + H^+$: 390.20 $[M + H]^+$; Found: 390.13.

Fluorescence titration

Receptor **1** (7.78 mg, 0.04 mmol) was dissolved in CH_3CN (2 mL) and 3 μL of the receptor **1** (20 mM) was diluted in 2.997 mL CH_3CN to make the final concentration of 20 μM. $Zn(ClO_4)_2$ (0.08 mmol) was dissolved in CH_3CN (4 mL) and 0.3–9 μL of the Zn^{2+} solution (20 mM) was transferred to receptor **1** solution (20 μM) prepared above. After mixing the solution for a minute, fluorescence spectra were obtained at room temperature.

UV–vis titration

Receptor **1** (72.8 mg, 0.02 mmol) was dissolved in CH_3CN (2 mL) and 3 μL of the receptor **1** (10 mM) was diluted in 2.997 mL CH_3CN to make the final concentration of 10 μM. $Zn(ClO_4)_2$ (0.04 mmol) was dissolved in CH_3CN (4 mL) and 0.3–6 μL of the Zn^{2+} solution (10 mM) was transferred to receptor **1** solution (10 μM) prepared above. After mixing the solution for a minute, UV–vis spectra were obtained at room temperature.

Competition with other metal ions

Receptor **1** (7.78 mg, 0.04 mmol) was dissolved in CH_3CN (2 mL) and 3 μL of the receptor **1** (20 mM) was diluted in 2.997 mL CH_3CN to make the final concentration of 20 μM. Na^+, Mg^{2+}, Al^{3+}, K^+, Ca^{2+}, Cr^{3+}, Mn^{2+}, Fe^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Ag^+, Cd^{2+}, Hg^{2+}, Pb^{2+} and Fe^{2+} (0.08 mmol) were dissolved in CH_3CN (4 mL), respectively. 0.3 μL of each metal solution (20 mM) was taken and added into 3 mL of each receptor **1** solution (20 μM) prepared above to make 1 equiv. Then, 3 μL of Zn^{2+} solution (20 mM) was added into the mixed solution of each metal ion and receptor **1** to make 1 equiv.

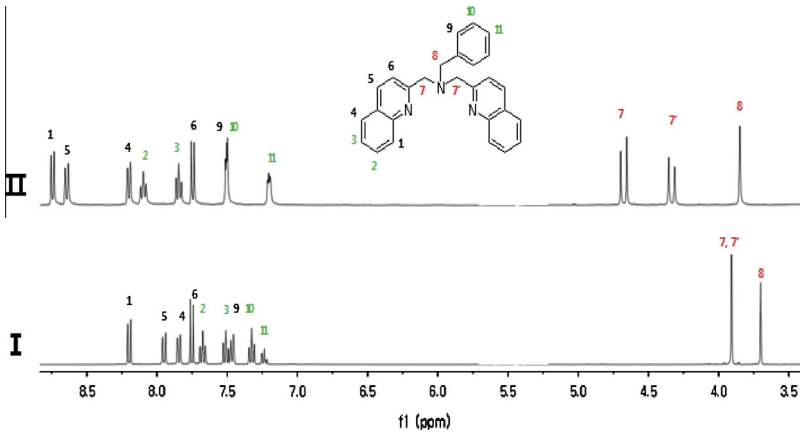


Fig. 7. ^{1}H NMR spectra of **1** with Zn^{2+} in CD_3CN: (I) **1**; (II) **1** with 1 equiv of Zn^{2+}.

After mixing them for a minute, fluorescence spectra were taken at room temperature.

Job plot measurement

Receptor **1** (72.8 mg, 0.02 mmol) was dissolved in CH_3CN (2 mL). 5, 4.5, 4, 3.5, 3, 2.5, 2, 1.5, 1, and 0.5 μL of the receptor **1** solution were taken and transferred to vials. Each vial was diluted with CH_3CN to make a total volume of 2.995 mL. $Zn(ClO_4)_2$ (0.04 mmol) was dissolved in CH_3CN (4 mL). 0, 0.5, 1, 1.5, 2, 2.5, 3, 3.5, 4, 4.5, and 5 μL of the Zn^{2+} solution were added to each diluted receptor **1** solution. Each vial had a total volume of 3 mL. After shaking the vials for a few minutes, UV–vis spectra were taken at room temperature.

NMR titration

For ^{1}H NMR titration of receptor **1** with Zn^{2+}, seven NMR tubes of **1** (38.9 mg, 0.1 mmol) dissolved in CD_3CN (0.5 mL) were prepared, and six different concentrations (0.02, 0.04, 0.06, 0.08, 0.1, and 0.2 mmol) of $Zn(ClO_4)_2$ dissolved in CD_3CN (0.5 mL) were added to each solution of **1**. After shaking them for a few minutes, ^{1}H NMR spectra were taken at room temperature.

Results and discussion

1 was synthesized by coupling of 2-(chloromethyl)quinoline hydrochloride and benzylamine via one step according to the literature method [37] and characterized by ^{1}H NMR and ESI-mass analysis.

*Selective optical response of **1** toward various metal ions*

To examine an insight into the fluorescent properties of chemosensor **1** toward various metal ions, the emission changes were measured with various metal ions such as Na^+, Mg^{2+}, Al^{3+}, K^+, Ca^{2+}, Cr^{3+}, Mn^{2+}, Fe^{3+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Ag^+, Cd^{2+}, Hg^{2+}, Pb^{2+} and Fe^{2+} in CH_3CN, which was conducted upon excitation at 311 nm (Fig. 1). The receptor **1** exhibited no fluorescence, but it became highly fluorescent on adding of Zn^{2+} ion. All other metal ions under investigation did not show any change of fluorescence of **1**, whereas Mg^{2+} responded with a slight increase in the fluorescence intensity. However, the intensity was significantly lower than that obtained with Zn^{2+} alone under the same conditions. Hence, these results suggest that **1** could be a good sensor for Zn^{2+}. For a practical purpose, we increased an amount of a buffer solution in CH_3CN. Identical results were obtained in only 1% aqueous solution.

*Fluorescence spectroscopy of **1**–Zn^{2+}*

The binding property of **1** to Zn^{2+} was investigated by fluorescence titration, as shown in Fig. 2a. The fluorescence spectrum of receptor **1** (20 μM) exhibited very weak emission at 426 nm (Φ_{free} = 0.008) due to the well-known PET fluorescence quenching process. However, with the increase in concentration of Zn^{2+} ion, the steady and smooth fluorometric spectrum curve was observed at 382 nm with the appearance of blue colored fluorescence; the quantum yield (Φ = 0.305) resulted in about 34-fold increase. The significant blue shift along with a large enhancement of emission intensity might be attributed to the Zn^{2+}-induced CHEF process [38]. After addition of **1** equiv of Zn^{2+}, it reached a saturation level. The saturation behavior of **1** with Zn^{2+} suggested that the **1**–Zn^{2+} complex has a 1:1 stoichiometry (Fig. 2b). Job plot also showed the 1:1 stoichiometry (Fig. 3), which was further confirmed by ESI-mass spectrometry analysis (Fig. 4). The $[1 + Zn + CH3CN]^{2+}$ complex was calculated to be m/z 247.07 and measured to be m/z 246.87. Based on Job plot and ESI-mass spectrometry analysis, we propose the structure of **1**–Zn^{2+} complex, as shown in Scheme 2. From the fluorescence titration experiment, the association constant (log K_a) of **1** for Zn^{2+} was determined as 4.52 (Fig. S1), using the Benesi–Hildebrand equations. The detection limit of **1** for Zn^{2+} was estimated to be 1.2×10^{-6} M by using the method of a signal-to-background (S/B) ratio (Fig. S2) [39].

*Absorption spectroscopy of **1**–Zn^{2+}*

The photophysical properties of **1** were also examined using UV–vis spectrometry. UV–vis spectrum of **1** exhibited absorption bands at 207 and 228 nm and a broad band around 264 nm (Fig. 5a). Upon the addition of Zn^{2+} to the solution of **1**, a new absorption band with a maximum at 203 nm appeared and its intensity increased gradually. The well-defined isosbestic points at 207, 235, 246 and 282 nm indicate the clean conversion of **1** into **1**–Zn^{2+} complex. When **1** was continuously titrated with Zn^{2+}, it reached a maximum at 1.0 equiv of Zn^{2+}, again, indicating a 1:1 stoichiometry for sensor **1** and Zn^{2+} (Fig. 5b).

Competition experiments

To further check the practical applicability as a Zn^{2+}-selective fluorescent sensor, we carried out competition experiments. **1** was treated with 1 equiv of Zn^{2+} in the presence of 1 equiv of competing metal ions. As shown in Fig. 6, the presence of other background metal ions showed no or a little change of fluorescence intensity, except for Cu^{2+} and Hg^{2+}, which inhibited about 50% of the fluorescence obtained with Zn^{2+} alone. Nevertheless, the recep-

tor **1** still had a sufficient turn-on ratio for the detection of Zn^{2+} in the presence of Cu^{2+} and Hg^{2+}. In particular, cadmium ion which has similar properties hardly inhibited the fluorescence intensity of **1**–Zn^{2+}. Thus, **1** could be used as a selective fluorescent sensor for Zn^{2+} detection in the presence of most competing metal ions.

^{1}H NMR titration experiments

To understand the nature of the binding interaction between receptor **1** and Zn^{2+}, ^{1}H NMR titration experiments were performed (Fig. 7). The complexation of receptor **1** with Zn^{2+} might be expected to reduce the electron density of the coordination sites, resulting in a down field shift of the nearby proton signals. As expected, the most of quinoline and benzyl hydrogens at 7.3–8.2 ppm were shifted to 7.3–8.6 ppm. The methylene protons were also shifted to the down field. Among the methylene protons (3.70 and 3.91 ppm), singlet H_7 and $H_{7'}$ signals split into two doublets upon addition of Zn^{2+}, indicating that the four protons of H_7 and $H_{7'}$ are in different chemical environment with the formation of the **1**-Zn^{2+} complex. Such a splitting of methylene protons was also previously reported in the literature [40–45]. These obvious changes of the chemical shifts indicate that **1** could form a stable complex with Zn^{2+}. There was no shifts in the position of proton signals on further addition of Zn^{2+} ions (>1.0 equiv) which confirm a 1:1 complexation between Zn^{2+} and **1**.

Conclusion

In conclusion, we report a simple quinoline-based fluorescent probe **1** which works as a chemosensor for Zn^{2+}. Upon interaction with Zn^{2+}, **1** produced a blue fluorescence with a large enhancement (34 folds) of the emission intensity. Moreover, **1** detected selectively Zn^{2+} in the presence of most competing metal ions. In particular, **1** could clearly discriminate Zn^{2+} from Cd^{2+}. Therefore, **1** could be used as an off–on fluorescent sensor toward Zn^{2+}. The sensing mechanism of **1** for Zn^{2+} might be attributed to the combination of PET and CHEF mechanisms in **1**. Future study will be focused on developing more water-soluble probes for Zn^{2+} detection.

Acknowledgements

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Appendix A. Supplementary material

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.saa.2013.09.118.

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