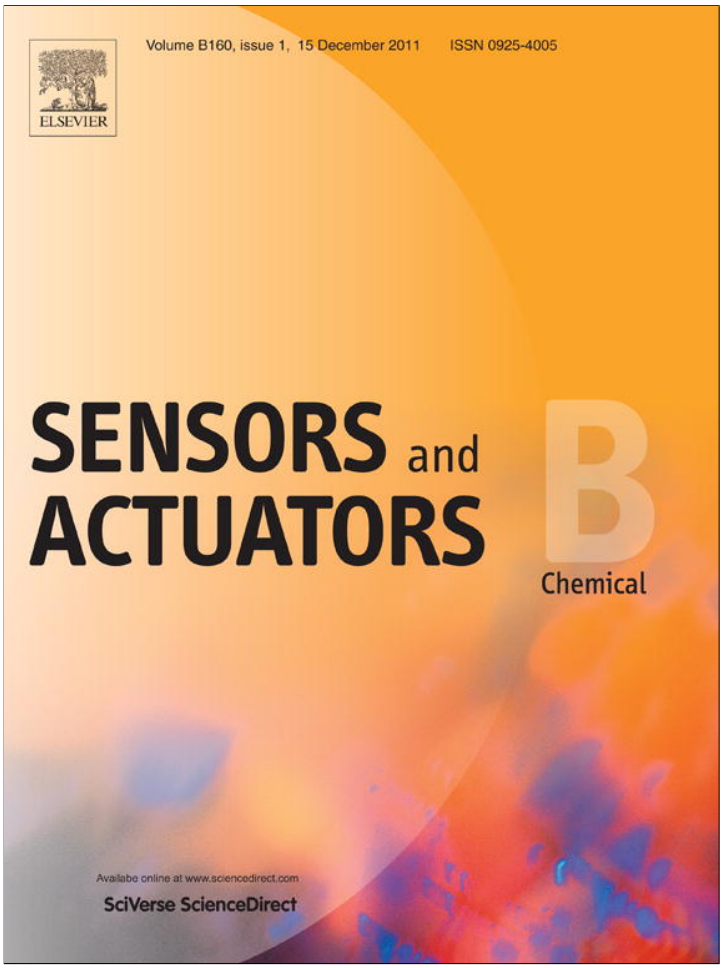

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A fluoride-selective colorimetric and fluorescent chemosensor and its use for the design of molecular-scale logic devices

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ABSTRACT

A simple and efficient chemosensor **1** was synthesized by reacting thiosemicarbazide with 1-naphthaldehyde. It was found that the sensor is highly selective toward fluoride anions in both UV–vis and fluorescence channels in DMSO solution. Especially, the spectral responses of the sensor along with a visible color change can be switched back and forth by successively adding F^- and HSO_4^- anions into the DMSO solution, which may be represented by a complementary "IMPLICATION/INHIBIT" logic gate at molecular level employing both the ions as the chemical inputs. Based on such a reversible and reproducible sensing system, we designed a molecular-scale sequential memory unit displaying "Writing-Reading-Erasing-Reading" and "Multi-write" functions in the form of binary logic.

1. Introduction

These days, real-time monitoring and accurate detection of fluoride anions are attracting increasing attention owing to its extremely important role in health and environmental science. Numerous artificial neutral receptors containing urea [1], thiourea [2], phenolic units [3], amide [4], or pyrrole fragments [5] have been reported and exhibited a perceived color change or fluorescence response upon fluoride binding due to the deprotonation mechanism [6]. Of particular interest are systems that can signal the presence of F^- through the changes in both color and fluorescence for two reasons: (i) an easy-to-detect color change allows rapid visual sensing; (ii) it is possible to accurately determine the concentration for fluoride ion, because fluorescence is a more sensitive means of molecular detection compared with such modes as UV–vis, NMR or transmittance [7]. However, most of the previously reported fluoride-receptors are single responsive (i.e. *via* fluorescence or UV–vis channel only). Although limited systems have been found to be highly selective for fluoride over other halide anions in both fluorescence and UV–vis channels, there remain some important open questions. For example, they are unable to discriminate F^- and AcO^- ions, because these two species have a similar basicity (acetate is actually slightly more basic than fluoride) [8,9].

Owing to the increasing demands of information technology for miniaturization, a noteworthy trend in the molecular recognition investigations is to design molecular logic devices, such as logic gates [10–14], molecular keypad locks [15], information storage devices [16] and so on. Such logic instruments are believed to transfer the molecular-level information to the observable optical signal [13]. Since the pioneering work by de Silva et al. [10] in 1993, diverse digital functionalities (AND [10], OR [11], XOR [12], INHIBIT [13], etc.) have been realized by a large amount of organic molecule-based logic gates. Recently, various complex logic functions such as multivalued logic [14b] and Fuzzy logic [14c] are also achieved through simple organic systems. However, there is quite few currently reported combinatorial molecular logic gates displaying complementary IMPLICATION/INHIBIT (IMP/INH) logic functions [17]. As a basic unit, this kind of IMP/INH logic gate is very important for the design of many complex logic systems, such as half-adders and subtractors [18], multiplexers [19], encoders [20], and molecular keypad locks [21].

The outputs of the combinatorial logic systems, like the aforementioned logic gates or adders–subtractors, are exclusively a Boolean function of the current inputs. In contrast, for sequential logic circuits such as molecular keypad locks, the outputs are actually determined by the current state of the systems, which is usually a function of the previous inputs and the present inputs [16e]. In the latter case, the molecular-level systems that remember and store information about the previous inputs are required. Thus, the construction of sequential logic circuits having memory function has become another new research hotspot in the field of

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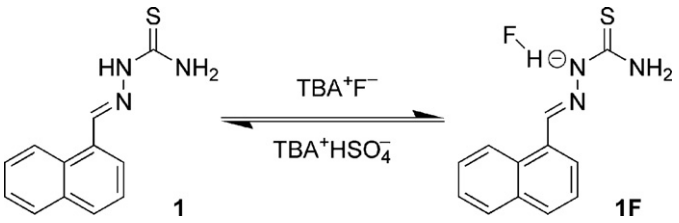


Scheme 1. Structure of receptor **1** and its deprotonation/protonation process.

molecular information technology. However, such sequential information memory or storage systems, especially those based on the synthetic receptor molecules capable of performing multiple logic operations, are relatively rare [16].

In this paper, we report a simple but efficient chemosensor **1**, which was prepared by reacting thiosemicarbazide with 1-naphthaldehyde (see Supporting information and Scheme 1). This sensor shows high selectivity toward fluoride anions in both absorption and fluorescence modes; especially, the fluoride-induced chromogenic process can be totally reversed with addition of HSO_4^-. In this way, depending on the inputs of these two ions, F^- and HSO_4^-, the receptor **1** works as a reversible colorimetric and fluorescent switch with complementary "IMP/INH" logic function, which allows the design of a molecular-scale sequential memory unit displaying "Writing-Reading-Erasing- Reading" behavior and "Multi-write" functions.

2. Experimental

2.1. Instruments

^{1}H- and ^{13}C NMR spectra were recorded with Bruker Advance AMX-400 and DMX-500 spectrometers, respectively. Elemental analysis was performed on a ThermoFinnigan Flash EA 1112 analyzer. A Bruker Vector 22 Fourier Transform Infrared spectrometer was applied for recording IR spectra in KBr pellets. UV–vis spectra were obtained in DMSO at 25 °C using a quartz cell of 1 cm on MOS-450 (Biologic Company, France). Steady-state fluorescence spectra were recorded on a PerkinElmer LS-55 fluorescence spectrometer in the right-angle geometry (90° collecting optics, λ_{ex} = 348 nm).

2.2. Materials

1-Naphthaldehyde, thiosemicarbazide, and all of tetrabutylammonium (TBA^+) salts were purchased from Aladdin Shanghai Reagent Company. DMSO was distilled in the presence of CaH_2 under reduced pressure before use. Other reagents were used without further purification.

2.3. Synthesis and characterization of sensor *1*

The sensor **1**, (2*E*)-2-(naphthylmethylene)hydrazinecarbothioamide, was synthesized according to the reported method [22]. To a solution of 1-naphthaldehyde (6.00 g, 38.5 mmol) in CH_3OH (500 mL) was added thiosemicarbazide (3.55 g, 39 mmol). After refluxing at 70 °C for 12 h, the solvent was removed under reduced pressure giving the crude product. The product was recrystallized from ethyl acetate to give a pale yellow solid (7.83 g, yield 82%). ^{1}H NMR (400 MHz, DMSO-d_6, 25 °C, TMS): δ = 11.46 (s, 1H), 8.89 (s, 1H), 8.32 (d, 1H), 8.30 (bs, 1H), 8.21(d, 1H), 7.98 (m, 3H), 7.62 (m, 1H), 7.54 ppm (m, 2H). ^{13}C NMR (125 MHz, DMSO-d_6, δ): 178.3, 141.6, 133.9, 131.0, 130.8, 129.8, 129.3, 127.8, 126.7, 126.3, 126.1, 123.4. Elemental analysis calcd (%) for $C_{12}H_{11}N_3S$: C 62.36, H 4.84, N 18.33; found: C 62.09, H 4.91, N 18.01.

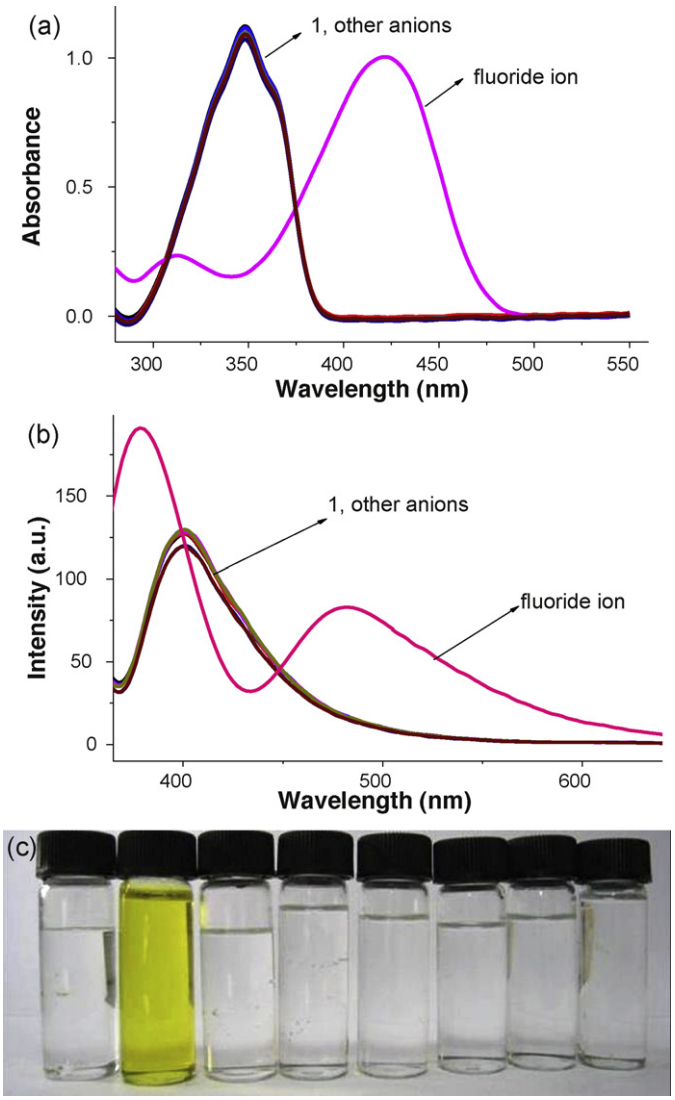


Fig. 1. (a) UV–vis spectra, (b) fluorescence spectra (λ_{ex} = 348 nm) of **1** (5×10^{-5} mol/L) upon addition of 50 equiv of various anions (as TBA^+ salts) in dry DMSO solution, and (c) corresponding color change of the DMSO solution of **1** induced by various anions (from left to right: **1** only, F^-, $H_2PO_4^-$, AcO^-, Cl^-, Br^-, I^-, and HSO_4^-).

3. Results and discussion

3.1. The interaction between receptor *1* and various anions

The examination of the receptor's interaction with anions was carried out by both UV–vis and fluorescence spectroscopy. In a typical experiment, TBA^+ salts of various anions were slowly added to a DMSO solution of receptor **1**. As shown in Fig. 1a, upon addition of TBA^+F^- (50 equiv), the absorption maximum of the receptor solution was red-shifted from 348 nm to 421 nm accompanying with a colorless-to-yellow color change. Both the new absorption band and the color change almost appeared instantaneously after the addition of fluoride ions. In the corresponding fluorescence spectra (Fig. 1b), the addition of F^- (50 equiv) caused two new emission peaks at 364 and 483 nm, respectively, with disappearance of the original emission at 400 nm. However, other anions (Cl^-, Br^-, I^-, HSO_4^-, $H_2PO_4^-$, AcO^-) did not bring noticeable changes in both absorption and emission spectra. The perceived color variation and spectral response on the addition of fluoride ion would be useful

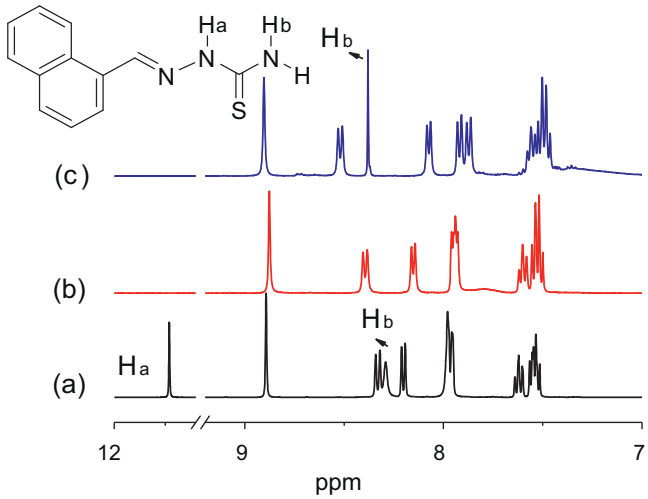


Fig. 2. ^{1}H NMR of receptor **1** in DMSO-d_6 upon addition of TBA$^+$F$^-$: (a) 0, (b) 1.0, and (c) 2.0 equiv.

not only for the ratiometric method of detection but also for rapid visual sensing (Fig. 1c).

To get insight into the binding mode of receptor **1** with F$^-$, ^{1}H NMR titration experiments were then performed in DMSO-d_6 (Fig. 2). It was found that the intensity of thiourea N–H$_a$ signal at 11.46 ppm disappears entirely upon addition of 1 equiv of fluoride. Also observed were that the N–H$_b$ signals shift downfield from 8.30 ppm to 8.38 ppm without an obvious decrease in the intensity during the course of titration. These observations indicated that the deprotonation process of thiourea N–H$_a$ segments is involved in the receptor's interaction with fluoride anions to increase the electron density on the N atom, associated with enhancement in the push-pull effect of the ICT (Internal Charge Transfer) transition [23]. A yellow color formation is visible evidence for this.

More support for the F$^-$-induced deprotonation mechanism came from colorimetric and fluorescent spectra titration (Fig. 3). In these experiments, TBA$^+$F$^-$ as a fluoride source was progressively added to the DMSO solution of **1** (5×10^{-5} mol/L). Upon fluoride addition, the absorption band at 348 nm gradually decreases, while a new band centered at 421 nm appears and increases with increasing concentrations of fluoride anion (Fig. 3a). Job's plot (Fig. S2) analysis of the colorimetric titration spectra showed a maximum at a 0.5 molar fraction of F$^-$, indicating the formation of a ratio of 1:1 complex **1F** (Scheme 1). These results together with the presence of two well-defined isosbestic points at 307 and 374 nm substantiate the existence of deprotonation effect throughout the titration. Also, based on the analysis of UV–vis titration spectra the detection limit of the sensor was determined to be 2.2×10^{-5} mol/L, indicating a high sensitivity for fluoride detection (see Fig. S3 in Supporting information).

On the other hand, spectrofluorimetric titration showed that upon successive addition of TBA$^+$F$^-$, the receptor produces two well separated emission bands at 364 and 483 nm, respectively, and the former gradually buried the original emission at 400 nm (Fig. 3b). As a chemosensor, this unique fluorescence response is of particular interest, since it provides a reliable ratiometric signal independent of the probe concentration [24].

Considering the possibility of the photochemical reactions involved with naphthalene moieties causing the fluorescence quenching of **1**, it appeared necessary to make a control experiment [25]. Thus, in the absence of fluoride anions, the emission intensity of the receptor solution was monitored in a continuous irradiation. The results showed that no significant change was observable in the fluorescence spectrum, suggesting that the spectral responses

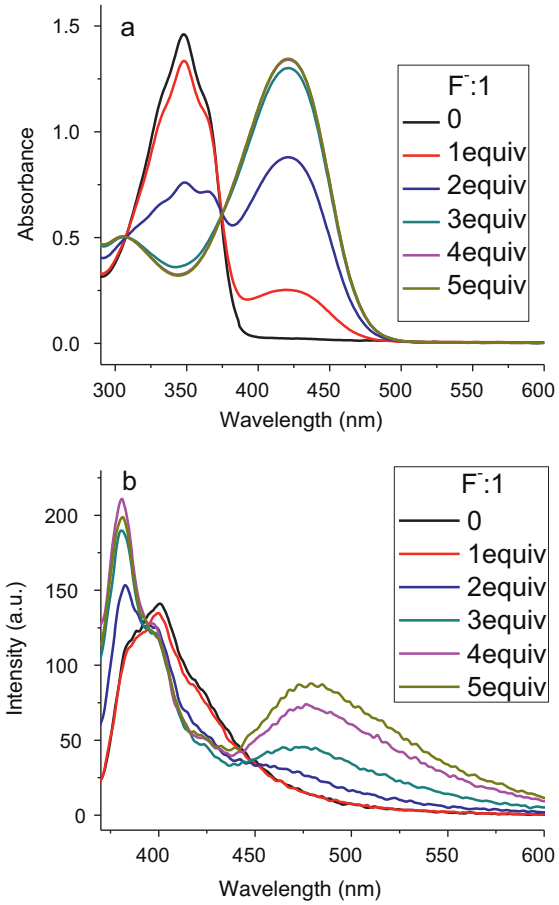


Fig. 3. (a) UV–vis titration spectra and (b) fluorescence titration spectra of **1** (5×10^{-5} mol/L) with TBA$^+$F$^-$ in absolute DMSO solution. (F$^-$: **1** represents the molar ratio of fluoride to the receptor.)

of the receptor **1** toward F$^-$ should be attributed to the enhanced ICT effect induced by fluoride ion binding.

It is well known that the basicity difference is a crucial factor for anion recognition [26]. Therefore, it is easily understandable that sensor **1** has no response to such anions as Cl$^-$, Br$^-$, I$^-$, $H_2PO_4^-$, and HSO_4^-, because their basicity is rather low. However, the observation that the sensor exhibited high selectivity of F$^-$ over AcO$^-$ seems to be surprising, since acetate is slightly more basic than fluoride [9]. This result may be explained by the idea that fluoride ion has much larger charge density than AcO$^-$ and hence strongly interacts with N–H fragments leading to the deprotonation. Also, the configuration of acetate anion most likely does not match with the acidic site of the sensor, which is probably another reason.

3.2. The reversible colorimetric switch with complementary "IMP/INH" logic functions

As discussed above, concomitant with a color change from colorless to yellow, a drastic red-shift of 73 nm was observed in the UV–vis spectra of sensor **1** in DMSO upon fluoride binding. More interestingly, we found that the F$^-$-induced chromogenic process is totally reversed with HSO_4^- anions (see Fig. 4). The eye-detected change was clearly reflected in the UV–vis spectra, in which the addition of 1.5 equiv of HSO_4^- results in vanishing of the band at 421 nm and reappearance of the absorption at 348 nm (Fig. 4). The reversible colorless–yellow–colorless cycle could be repeated for several times by alternating addition of 5 equiv of F$^-$ and 1.5 equiv of HSO_4^- salts.

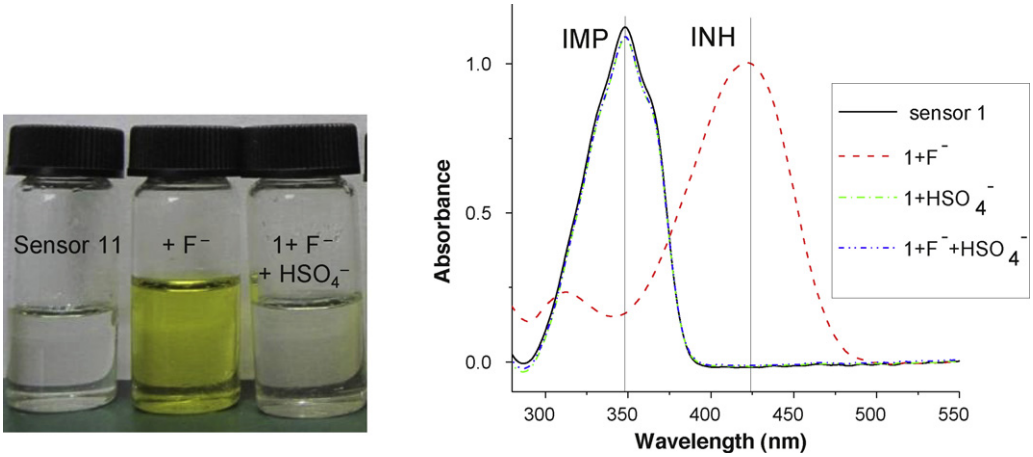
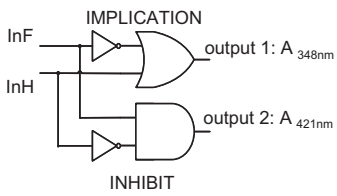


Fig. 4. Left: color change of **1** (5×10^{-5} mol/L) upon successive addition F^- (5 equiv), and HSO_4^- (1.5 equiv) in DMSO; right: UV–vis spectra of receptor **1** in DMSO with various combinations of F^- (5 equiv) and HSO_4^- (1.5 equiv), as well as the corresponding molecular logic functions IMP (348 nm) and INH (421 nm).

However, this reversible chromogenic switching process did not occur when HSO_4^-, instead of F^-, is previously added to the solution. A set of comparison experiments demonstrated that the successive addition of 1.5 equiv of HSO_4^- and 5 equiv of F^- does not produce the DMSO solution of **1** any change in both its color and absorption spectrum. The results indicate that the presence of HSO_4^- anions would inhibit the interaction of F^- with the sensor **1**, since HSO_4^- is a more acidic species compared to **1**. As elucidated in Scheme 1, the interaction of HSO_4^- with the complex **1F** reproduces the receptor; meanwhile the released F^- ions could be converted into other species such as HF and HF_2^- [27]. This also explains why the colorimetric switch can work in a reproducible manner, despite the fact that the added F^- and HSO_4^- anions are not of equal molar amount in one single cycle.

The reversible and reproducible colorimetric switching process may be represented by a molecular "INHIBIT" logic gate, employing F^- (InF) and HSO_4^- (InH) as the inputs and the absorbance at 421 nm as the output. When using the absorbance at 348 nm as another output, an "IMPLICATION" logic gate is fabricated. In this way, a complementary IMP/INH logic function can be realized based on the receptor molecule **1**, as shown in Fig. 5.

Thus, the color changes of **1** in DMSO (optical output) are controlled by the input of two anions: F^- "switches" ON the optical output, while HSO_4^- "switches" OFF the optical output. By alternately adding F^- (5 equiv) and HSO_4^- (1.5 equiv) into the receptor solution, a reversible colorimetric switch could be created in a reproducible manner (Fig. 6). To the best of our knowledge, this kind of reversible and reproducible switch is of great interest for molecular-level information processing. In the field of information technology, the switching process must be based on a reversible chemical process to perform any useful calculation [28]. However, most of newly reported logic systems are actually based on irreversible chemical processes. Although these logic devices have already found some applications in medicine as novel approaches toward anticancer therapy [29], they are not suitable for computational operations. Therefore, the present logic device has a great advantage over early reported relative systems at least in terms of the reversible and reproducible characteristics.



Truth Table

InF	InH	A_{348nm}	A_{421nm}
0	0	1	0
1	0	0	1
0	1	1	0
1	1	1	0

Fig. 5. The complementary IMP/INH logic gate and its truth table. InF and InH represent Input F^- and Input HSO_4^-, respectively.

3.3. The design of molecular memory unit

Based on the reversible and reproducible colorimetric switch, we designed a useful sequential logic circuit displaying "Writing-Reading-Erasing-Reading" behavior in the form of binary logic for molecular-level information processing. In this concrete system (Fig. 7), the ON state (Output 2 = 1) is defined as the strong absorption at 421 nm, whereas the OFF state (Output 2 = 0) corresponds to the significantly weak absorption at the identical wavelength. The inputs are constituted by F^- (InF) and HSO_4^- (InH) for the Set (S) and Reset (R), respectively. The operation of this memory unit is as follows: whenever the Set input is high (S = 1), the system writes and memorizes the binary state 1; on the other hand, when the Reset input is high (R = 1), the 1 state is erased and the 0 state is written and memorized. As shown in Fig. 6a, the reversible and reconfigurable sequences of Set/Reset logic operations in a feedback loop demonstrate the memory feature with "Writing-Reading-Erasing-Reading" functions with the absorbance at 421 nm as the output. Also, Fig. 6b defines the bistability behavior, "ON–OFF" state, of receptor **1** and reveals the non-volatile nature of the memory effect. More importantly, the "ON–OFF" states of **1** could be repeated for many times, suggesting "Writing-Reading-Erasing-Reading" cycles could be conducted.

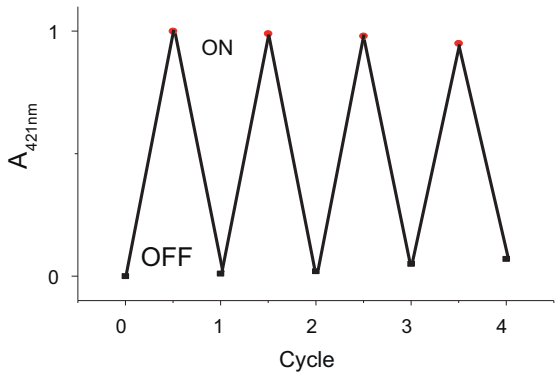


Fig. 6. The reversible and reproducible colorimetric switch controlled by alternate addition of F^- (5 equiv) and HSO_4^- (1.5 equiv) into the DMSO solution of **1** (5×10^{-5} mol/L).

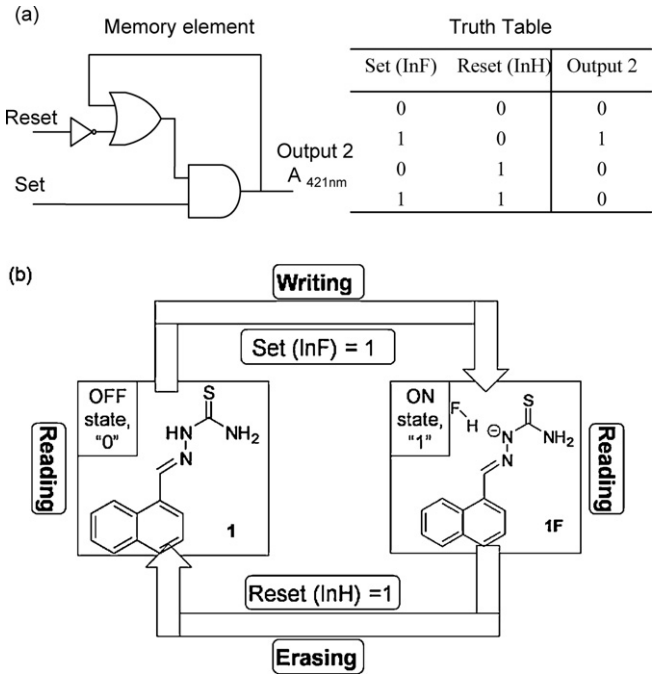


Fig. 7. (a) The sequential logic circuit of the memory machine and its truth table. (b) The feedback loop exhibiting reversible logic operations with "Writing-Reading-Erasing-Reading" behavior.

In other words, this system exhibits "Multi-write" ability without obvious degradation in its optical output (the absorbance at 421 nm). As a result, this kind of sequential logic circuits possessing the same behavior as the traditional logic devices constructed by semiconducting materials would be beneficial to the development of molecular microprocessors for memory elements of integrated logic circuits in the future.

4. Conclusion

In summary, this article describes a simple and efficient chemosensor **1** containing a naphthalene signal moiety and thiourea recognition sites to fluoride anions. The sensor has proven to be highly selective for fluoride and show a remarkable color change and fluorescence quenching upon fluoride binding in DMSO solution, which enable us to discriminate F^- ion from other anions including Cl^-, Br^-, I^-, $H_2PO_4^-$, AcO^-, and HSO_4^- by naked eye only. More interestingly, the F^--induced chromogenic process could be totally reversed by addition of HSO_4^-. This chemical switching may be represented through the molecular-level complementary "IMPLICATION/INHIBIT" logic gate employing F^- and HSO_4^- as the inputs. Based on the reversible and reproducible colorimetric switch, we has designed a molecular-scale sequential information processing circuit displaying "Writing-Reading-Erasing-Reading" behavior and "Multi-write" functions in the form of binary logic.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.snb.2011.09.018.

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