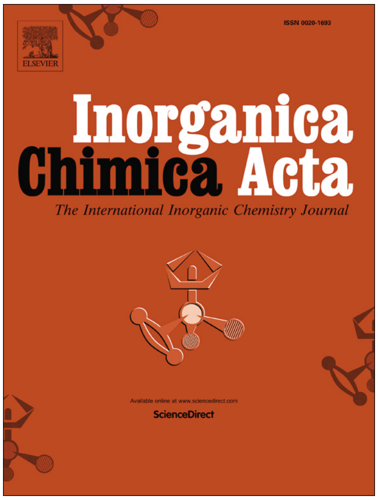

Research paper

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Probing anion and cation with novel salicylidene Schiff base receptor appended with 1, 10-phenanthroline: mimicking INHIBIT molecular logic gate

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Abstract: A novel tailor-made 1, 10-phenanthroline based Schiff base, 2-((E)-(1, 10-phenanthroline -5-ylimino) methyl) phenol, (**1**) receptor has been designed, synthesized and reported as multi-ion responsive scaffolds for the sensing of both anions and cations via chromogenic methods. In CH_3CN, receptor **1** displayed excellent selectivity for F^- ions amongst anions and for Al^{3+} and Hg^{2+} ions amongst cations. Moreover, the colorless solution of **1** turned yellow only with F^- ions due to new absorption band in visible region. Receptor **1** formed 1:1 complex with F^- ions while 1:2 with the Hg^{2+}/Al^{3+} ions as revealed by the separate Job's plot analysis. Receptor **1** can also mimic the functioning of "TRANSFER" and INHIBIT molecular logic gate with chemical inputs from Al^{3+} and F^- ions.

Keywords: 1, 10-phenanthroline, colorimetric, cation /anion recognition, Schiff base, molecular logic gates

1. Introduction

Schiff bases constitute a major category in coordination chemistry as their coordinating ability containing various donor atoms is widely reported. They have several applications in analytical chemistry, food and dye industry, catalysis and in many biological aspects [1-3]. The spectacular characteristics of Schiff bases like low cost simple synthesis coupled with synthetic tailorability, biodegradability, pronounced photophysical properties and ability to coordinate to metal ions tag Schiff bases as one of the most widely explored molecular chemosensors [4-6]. Schiff bases can be used for heavy metal sensing and removal of pollutant in the environment. They are good spectrophotometric and fluorimetric agents [2, 7]. The complexing ability of Schiff base has been attributed to the presence of azomethine linkage, which has a tendency to donate the lone pair of electrons present on the nitrogen atom of the azomethine moiety (C=N-). The structure of Schiff bases is responsible for geometric and cavity control of host guest complexation and produce remarkable selectivity and sensitivity for specific ion. Consequently, Schiff base complexes have attracted increasing attention in the area of ionic binding. [8-11].

Development of 2-Hydroxy Schiff base receptors for anion recognition process is commonly based on the fact that strongly basic anions can easily deprotonate the –OH proton or promote the formation of keto-tautomer or inhibit the excited state proton transfer (ESIPT) phenomenon, leading to modification of spectral properties and inducing the desired chemoselectivity in the sensor [12-16]. The presence of an acidic group like a phenolic OH or another donor group close to the azomethine group increases the coordinating effect of the lone pair of electrons, thereby increasing the stability of the metal complexes.

More recently, interfacing of chemistry with electronics has attained immense significance. This interfacing is achieved by connecting microcontroller services with chemosensors [17]. The tailor-made chemosensors can be viewed as computational devices that use physical or chemical inputs to generate outputs based on a set of logical operators. The development of molecular logic gates [18-20] and their integration into working automation [21] and arithmetic systems [22-23] has brought chemists closer to the realization of a molecular scale calculator i.e. moleculator. These molecular logic gates operate in a wireless mode and thus, they have the potential for computation on a nanometer scale that silicon-based devices cannot address [24]. In addition to Boolean molecular logic gates, Fuzzy logic is playing key roles in the development of a machine intelligent quotient. Fuzzy logic is an “infinite-valued” logic that helps in making decisions based upon incomplete and vague information tainted by environmental noise i.e. in soft computing [25].

Based on the above known facts, we report the recognition studies on cation and anion sensing by novel 5-substituted-1, 10-phenanthroline based azomethine receptor **1** possessing a phenolic –OH group that helps in binding to F^- ion. The position of azo-methine (-CH=N) group in this Schiff base allows it to act as a ligand to bind with cations (Al^{3+} / Hg^{2+}). It is well documented in literature that presence of a functional group (–OH) close to the azomethine group makes the Schiff bases to bear an excellent coordinating ability to act as multidentate ligands. [26-27]. To the best of our knowledge, 1, 10-phenanthroline based Schiff bases are remotely explored for chemosensing properties. The receptor **1** displayed selectivity for most basic F^- ions accompanied by colorimetric response of **1** for F^- ions; while UV-vis spectral changes in **1** were observed with only Al^{3+} and Hg^{2+} ions.

2. Experimental

2.1 Methods and Instrumentation

All reagents and chemicals were purchased from Aldrich and used without further purification. Solvents used for spectroscopic studies were purified by standard procedures before use. ^{1}H NMR spectra were recorded on a Bruker Advance II 400 at 400 MHz from solutions in DMSO-d$_6$. ^{13}C NMR spectra were recorded at 100 MHz and values are reported relative to $CDCl_3$ signal at δ 77.0. Absorption spectra were recorded on Shimadzu UV-240 spectrophotometer.

2.2 General procedure for Spectroscopic experiments

Stock solutions of the receptor **1** (10^{-2} M), different anions and metal ions (both 10^{-1} M) were prepared in CH_3CN. These stock solutions were further diluted with CH_3CN and used further for different spectroscopic experiments (UV-vis). All the anions (F^-, Cl^-, Br^-, I^-, AcO^-, $H_2PO_4^-$ and HSO_4) were added as their tetrabutylammonium salts; while cations (Na^+, K^+, Mg^{2+}, Al^{3+}, Mn^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Cd^{2+} and Hg^{2+}) were added as their perchlorate salts for the absorption experiments. Aliquots of ions under investigation were then injected into the sample solution through a rubber septum in the cap. The solutions were allowed to get stabilized after each addition, and were then scanned.

2.3 *^{1}H NMR Titrations*

To the 4 × 10^{-2} M solution of **1** in DMSO-d$_6$, the varying equivalents of TBAF were added and ^{1}H NMR spectra were recorded after each addition.

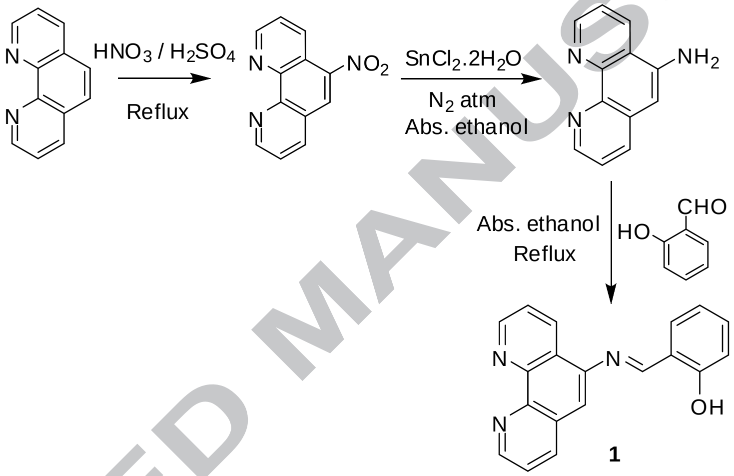
*2.4 Synthesis of 2-((E)-(1, 10-phenanthroline -5-ylimino) methyl) phenol (**1**)*

5-amino-1, 10-phenanthroline has been synthesized from 1, 10-phenathroline using the known literature methods [28]. 5-amino-1, 10-phenanthroline (0.5 g, 2.56 mmol) was dissolved in 5 ml of hot absolute ethanol. To this hot stirring solution of 5-amino-1, 10-phenanthroline, salicyldehyde (0.312 g, 2.56 mmol) was added and the contents were heated at 70 °C till the TLC indicated completion of the reaction [29]. Yellow colored solid separated at the completion of the reaction. The yellow residue was filtered and collected as crude product. Recrystallization of crude solid from $CHCl_3$ / CH_3OH resulted in the formation of pure product **1**.Yellow solid, 85%

Yield; M.P: 196 ºC. Selected FT-IR peaks (cm^{-1}): 3411 (υ_{O-H}), 3060 ($\upsilon_{C-Hstr.,}$ aromatic), 1617 ($\upsilon_{C=NH}$) ^{1}H NMR (DMSO, 400 MHz) δ: 7.07-7.01 (m, 2H, Ar-H), 7.49 (t, *J* = 7.08 Hz, 1H, Ar-H), 7.89-7.75 (m, 4H, Ar-H), 8.48 (d, *J* = 7.92, 2H, Ar-H), 8.67 (d, *J* = 7.08 Hz, 1H, Ar-H), 9.05 (s, 1H, Ar-H), 9.17 (s, 2H, Ar-H);^{13}C NMR (DMSO, 100 MHz) δ: 112.61, 117.45, 119.16, 119.59, 123.29, 123.49, 125.21, 128.57, 132.06, 132.82, 134.18, 135.88, 145.07, 145.38, 146.34, 149.87, 150.94, 161.25, 164.99. ESI-MS: m/z= 300.14 $(M+1)^+$.

3. Results and Discussions

Scheme-1 outlines the synthesis of receptor **1**, whose structure has been characterized by ^{1}H NMR, ^{13}C NMR and Mass Spectra (Figs. S1-S3).



Scheme 1: Synthesis of receptor **1**

*3.1. Optical changes of **1** with addition of various anions*

The recognition and sensing ability of receptor **1** towards various anions (F^-, Cl^-, Br^-, I^-, AcO^-, $H_2PO_4^-$ and HSO_4^- (tetrabutyl ammonium as a counterion) was determined by naked eye and UV-vis titration spectra. With immediate addition of 100 equiv. of all the anions, the colorless solution of **1** turned to yellow with only F^- ions in CH_3CN. All the other added anions did not produce any color change that could be detected with naked eye.

In the absence of anions, receptor **1** (20 μM in CH_3CN) displayed absorption bands at 270 nm and 343 nm. Here, the low energy band at 343 nm may occur due to intramolecular charge transfer (ICT) transitions within the whole structure of the Schiff base [30-31]. The ICT band at 343 nm indicates the presence of strong intramolecular hydrogen bond between the hydroxyl group and the azomethine nitrogen that causes planarity of the molecules and facilitates the charge transfer to take place within the whole molecule [32]. Amongst all the added anions, significant changes in the absorption spectra of **1** were observed only with the addition of F^- ions, where a new band at longer wavelength was observed in case of **1**, responsible for naked eye color change. The UV-vis spectrum of **1** was unaltered with addition of all other anions (Fig. 1).

Along with CH_3CN, the photophysical properties of receptor **1** has also been evaluated in other solvents like pure MeOH, DMSO as well as in pure aqueous media. The receptor **1** showed maximum selectivity and sensitivity towards F^-, Al^{3+} and Hg^{2+} ions only in CH_3CN. In all other solvents, non-selective behavior of the receptor as well as precipitation in case of pure H_2O system was observed.

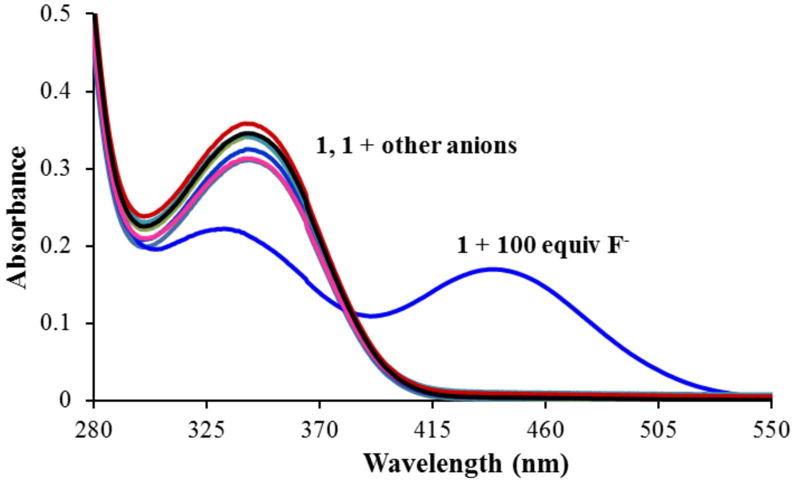


Fig. 1: UV-vis spectral changes of **1** (20 μM) upon addition of 100 equiv. of various anions.

To have a better insight into the response and recognition phenomenon, UV-vis titrations were performed with F^- ions. With the addition of F^- to solution of **1** (20 μM), the absorbance of 343 nm band decreased; while a new band emerged in the visible region at 436 nm. With the progressive addition of F^-, the intensity of 436 nm band increased with concomitant color change from colorless to yellow. At the same time, 343 nm band was blue shifted to 332 nm. With the addition of F^-, the intramolecular hydrogen bond between the –OH group and the –HC=N nitrogen might have broken, leading to new hydrogen bonding interactions between the –OH groups of the phenyl ring of salicylaldimine with F^- ions and causing the color transition from colorless to yellow.

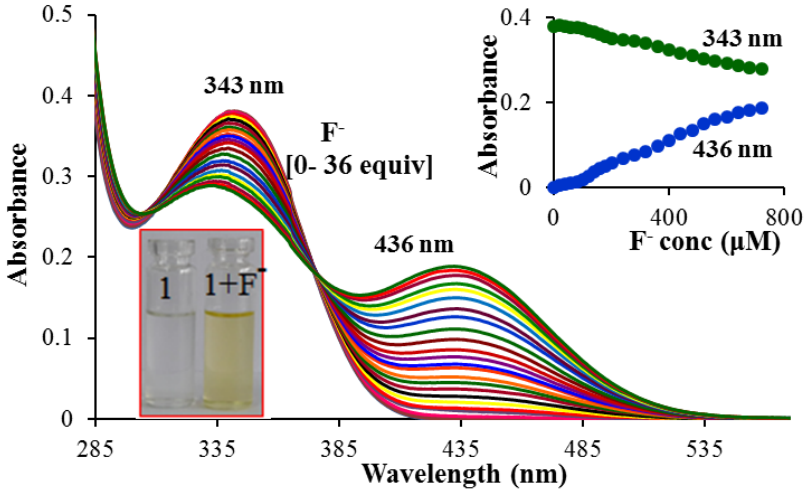


Fig. 2: Family of UV-vis spectra of **1** with F$^-$ ions in CH_3CN recorded during the titration Inset: Plot of absorbances at 343 and 436 nm versus the concentration of F$^-$ ions added along with naked eye color change in **1** with F$^-$ ions.

The Job's plot experiment was performed which pointed towards 1:1 binding stoichiometry of **1** with F$^-$ ions (Fig. S4). The binding constant of **1** with F$^-$ was calculated based on the UV-vis titration through the Benesi-Hildebrand equation [33], which was given as follows;

$$\frac{1}{A-A_o} = \frac{1}{A_{max}-A_o} + \frac{1}{[A_{max}-A_o]\, K\, [C]}$$

Here, A_o, A, and A_{max} is the absorbance of free **1**, measured with F$^-$ and measured with excess amount of F$^-$ at 436 nm, respectively and K is the association constant and [C] is the concentration of F$^-$ ion added. Plotting of $1/(A-A_0)$ versus $1/[F^-]$ showed a linear relationship, which indicates that **1** associates with F$^-$ in a 1:1 stoichiometry. The binding constant (K) between **1** and F$^-$ is determined from the ratio of intercept/slope to be 5.4×10^4 M^{-1}.

From the changes in F$^-$ dependent absorption intensity, the detection limit was calculated using equation LOD = $3\sigma/\rho$. So, to calculate limit of detection, standard deviation is divided by slope of line followed by multiply it with 3 [34]. The detection limit is reasonably estimated to be 5.2 µM.

*3.2 Optical changes of **1** with addition of various metal ions*

The binding interaction studies of **1** were conducted in CH_3CN against various cations like Na^+, K^+, Mg^{2+}, Al^{3+}, Mn^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Cd^{2+}, Hg^{2+} (perchlorate as the counter ion) using colorimetric and UV-vis experiments. Upon adding 100 equiv. of said cations to solution of **1**, no obvious color changes were observed in the colorless solution of **1,** excluding the possibility of colorimetric detection. However, the absorption spectrum of **1** was perturbed mainly with Hg^{2+} and Al^{3+} ions (Fig. 3). Different literature reports highlight similar type of absorption changes with addition of Al^{3+} to azomethine receptors, where low absorption intensity changes have been observed [8, 35]. All the other cations exhibited negligible changes in the absorption spectrum. The increased absorbance due to Cu^{2+} is because of its own absorbance in this region, so any actual changes on interaction of **1** with Cu^{2+} cannot be measured in this region (Fig. S5).

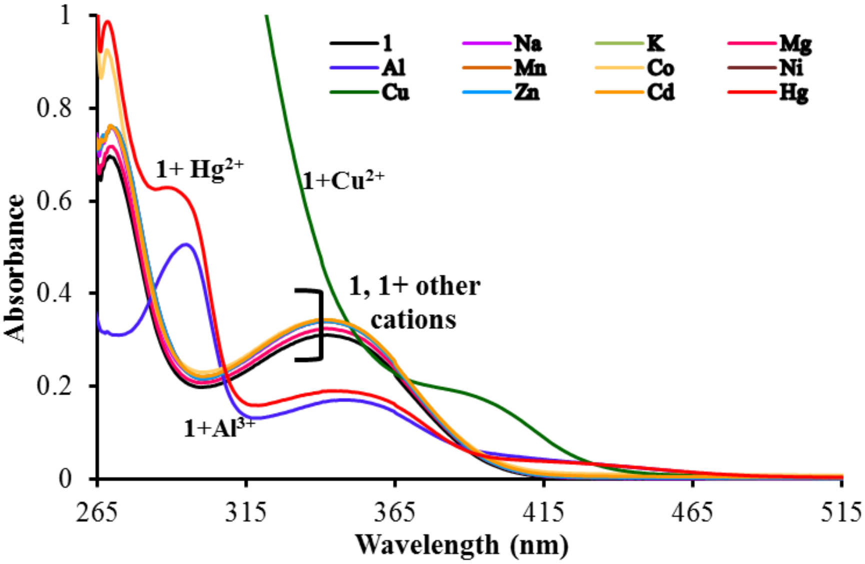


Fig. 3: UV-vis spectral response of **1** (20 μM) with 100 equiv. of various cations in CH$_3$CN.

The UV-vis titrations of **1** were carried out with Al^{3+} and Hg^{2+} ions in CH$_3$CN to deduce the detailed complexation characteristics (Fig. 4). With immediate addition of Al^{3+} ions, 270 and 343 nm bands were red shifted to 294 and 348 nm, respectively, accompanied by gradual decrease in the absorbance (Fig. 4a). The absorbance at both the wavelengths reached the limiting values when 70 equiv. of Al^{3+} ions have been added.

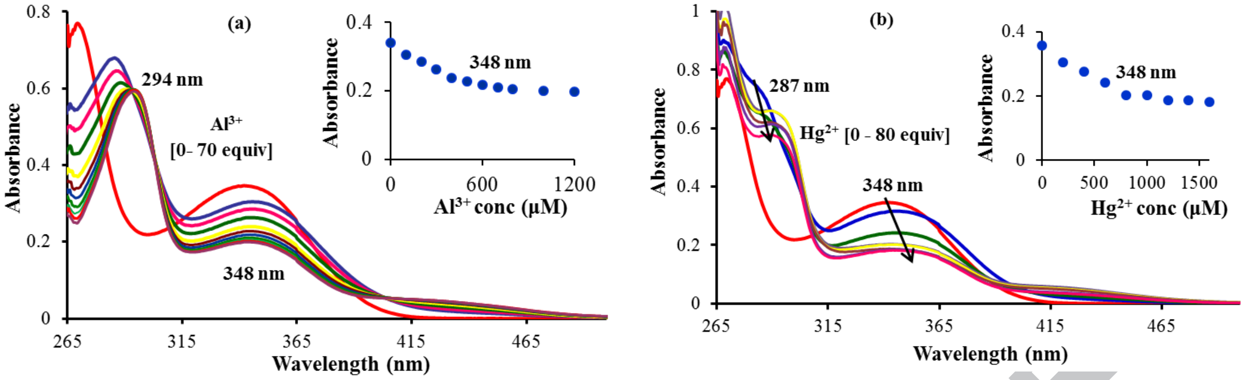


Fig. 4: Family of UV-vis spectra of **1** (20 µM) (a) with Al^{3+} ions (b) Hg^{2+} ions during the course of titration in CH_3CN. Inset: Plot of absorbances at 348 nm versus the concentration of Al^{3+} and Hg^{2+} ions.

Gradual addition of Hg^{2+} ions displayed the similar behavior as Al^{3+} ions at both the wavelengths except that the absorbance value reached saturation at 80 equiv. of Hg^{2+} ions (Fig. 4b). The Job's plot analysis pointed towards 1:2 (**1**:M^{n+}) stoichiometry of **1** with M^{n+} (M^{n+} = Al^{3+}, Hg^{2+}) ions (Fig. S6). The phenanthroline N's are known to offer good binding affinity towards cations [36]. Moreover, the presence of hydroxyl and imine groups in the same receptor molecule confers the coordination capacity required to coordinate Al^{3+} and Hg^{2+} ions [37]. Therefore, one M^{n+} ion may bind to the two phenanthroline N's; while the other to the hydroxyl and imine group simultaneously.

The respective binding constants calculated using Benesi-Hildebrand equation (K) [33] and LOD values calculated using equation LOD = $3\sigma/\rho$ [34] have been given in table 1. The higher binding constant and lower detection limit for Al^{3+} ions are indicative of better binding of **1** with Al^{3+} ions. According to United Nations Environment Report (UNEP), exposure to mercury vapour of level ~ 20 µM or higher have subtle effects on the central nervous system and this lower value is still higher than the LOD value calculated for Hg^{2+} by this method. Moreover, the literature reports reflect that Al^{3+} levels higher than 2.4 µM may cause encephalopathy [38], which is comparable to the LOD value for Al^{3+} calculated by this method.

Table 1. Binding constants (K in M^{-2}) of receptor **1** with Al^{3+} and Hg^{2+} and corresponding limit of detection (LOD in µM) values in CH_3CN.

Receptor	Metal ion	K (M^{-2})	LOD (µM)
1	Al^{3+}	8.6×10^9	3.2
	Hg^{2+}	4.14×10^9	4.9

*3.3 ^{1}H NMR Titration studies for the complexation of **1** with F^{-}*

To further understand the binding behavior of receptor **1** with F^{-}, ^{1}H NMR titration experiment of **1** (4 × 10^{-2} M) was performed in DMSO-d$_{6}$ with varying equivalents of TBAF (Fig. 5). The distinct signal of OH did not appear for **1** in its ^{1}H NMR spectrum in DMSO-d$_{6}$. With the addition of F^{-}, the protons of benzene ring adjacent to imine showed spectral pattern change and an upfield shift, probably due to increased electron density owing to H-bonding interactions of F^{-} ion with OH [39-40]. Since the signal of OH did not appear for receptor **1**, deprotonation followed by hydrogen bonding could not be ascertained.

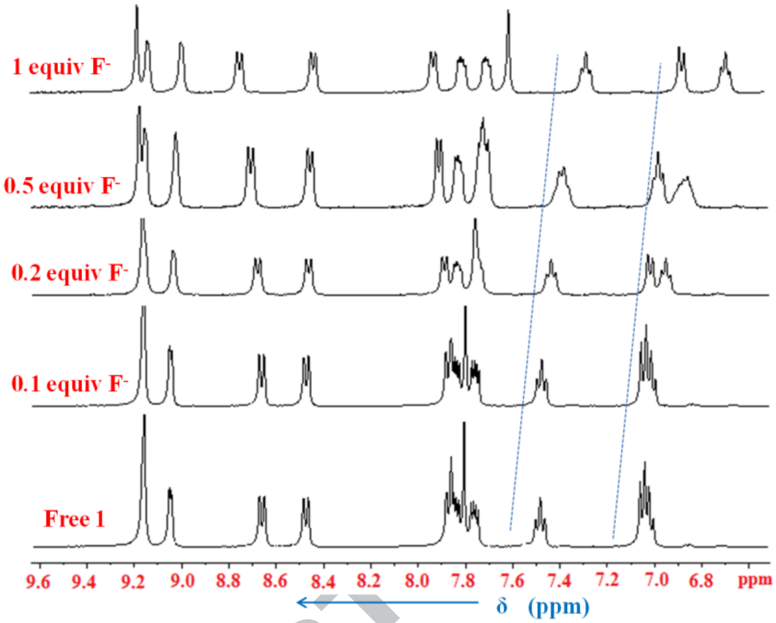
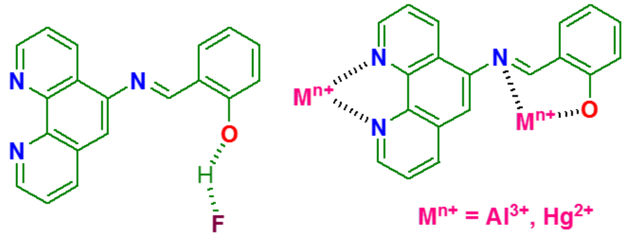


Fig. 5: ^{1}H–NMR titration of **1** (4 × 10^{-2} M) with incremental addition of TBAF in DMSO.

3.4 Plausible sensing mechanism

Scheme 2 demonstrates the plausible sensing mechanism of receptor **1** with F- and Al^{3+}/Hg^{2+} ions on the basis of Job's plot and ^{1}H NMR titrations. As suggested by the ^{1}H NMR titration experiments, the -OH of receptor **1** might be due to hydrogen bonding interactions with F^{-} ion. The selectivity of the receptor **1** for F^{-} ions may be justified on the basis of its characteristics like high basicity, small size and high charge density which makes it better fit for hydrogen bonding and deprotonation interactions. On the other hand, the active participation of phen N's and cooperative participation of salicylaldimine oxygen and imine nitrogen might be contributing in Al^{3+}/Hg^{2+} ion binding corresponding to 1:2 stoichiometry (Scheme 2).

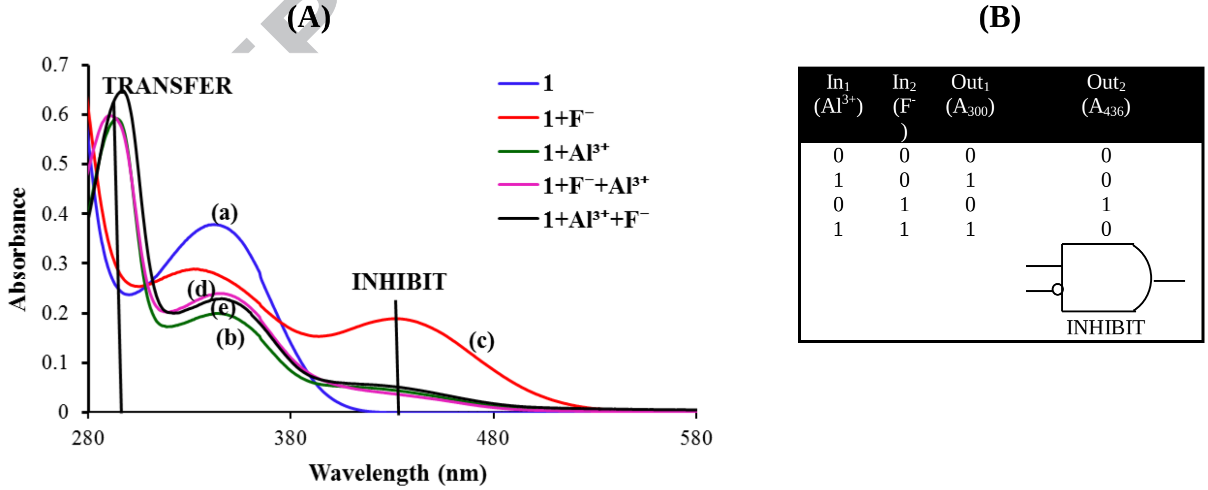


Scheme 2. Plausible sensing mechanism of interaction of receptor **1** with F^-, Al^{3+}/Hg^{2+} ions.

3.5 Elaboration of molecular photonic logic systems

Careful analysis of absorption changes observed with individual addition of Al^{3+} and F^- ions and their binding constant values reveals that different logic operations can be viewed in receptor **1** by observing different output values. Recently, the conversion of chemically encoded information into optical (fluorescence / absorbance) signals for the development of sequential logic devices, has attracted tremendous attention for unconvential computing.

The molecular switching behavior of receptor **1** with two chemical inputs (Al^{3+} and F^-) can be demonstrated with the help of binary logic. Depending upon the two chemical inputs Al^{3+} and F^- (400 μM), the receptor **1** gave different absorption bands that helps in elaboration of multi logic operations within single molecule. There are two input signals, viz. In_1 (Al^{3+}) and In_2 (F^-), whereas the output signals are, Out_1 ($A_{300\ nm}$) and out_2 ($A_{436\ nm}$). Figure 6A displayed different absorption spectra observed with addition of two inputs. Monitoring the absorption changes at 300 nm and 436 nm i.e. the addition of Al^{3+}, F^- and an equimolar mixture of Al^{3+} and F^-, led to "TRANSFER" and "INHIBIT" logic gates, respectively as represented in truth table (fig. 6B).



In_1 (Al^{3+})	In_2 (F^-)	Out_1 (A_{300})	Out_2 (A_{436})
0	0	0	0
1	0	1	0
0	1	0	1
1	1	1	0

Figure 4. (A) Molecular scale implementation of different logic gates; **(a).**blank; **(b).1** + Al^{3+}; **(c).1** + F^-; **(d).1**+ F^- + Al^{3+} and (e). **1** + Al^{3+}+ F^- (B) Truth table for "NOR" and "TRANSFER" gates.

"INHIBIT" is neither communicative nor associative and it can be viewed as two input AND gates, one of whose input lines contain an inverter [41]. Here, addition of F^- induced the appearance of the absorption band at 436 nm, acting as ON behavior, whereas Hg^{2+} addition led to only small absorption intensity at 436 nm, acting as the OFF switch. Receptor **1** (20 µM) gave "0" output at 436 nm with addition of Al^{3+} also, which on addition of F^- was converted to high output at 436 nm. But addition of both Al^{3+} (400 µM) and F^- (400 µM) resulted in low output at 436 nm and thus Al^{3+} inhibited the effect of F^- and is termed as 'INHIBIT' logic gate (Fig. 6).

Moreover, by changing the observation wavelength from 436 nm to 300 nm, the spectral changes led to "TRANSFER" logic gate [42]. The output of TRANSFER gate is high when either one of the inputs or both of the inputs are high.

Conclusions:

The present work reports the new salicylidene Schiff base (**1**) receptor possessing 1, 10-phenanthroline for selective recognition of F^- ions and sensing of Al^{3+} and Hg^{2+} ions. The hydrogen bonding interactions between the –OH groups of the phenyl ring of salicylaldimine and F^- ions are responsible for the recognition event. On the contrary, the phenanthroline N's and the availability of hydroxyl and imine groups in the same receptor molecule (**1)** account for the 1:2 stoichiometry with Al^{3+} and Hg^{2+} ions. The differential outputs observed by sequential addition of Al^{3+} and F^- ions to solution of **1** led to elaboration of "TRANSFER" and "INHIBIT" molecular logic gates at 300 nm and 436 nm, respectively.

Acknowledgements

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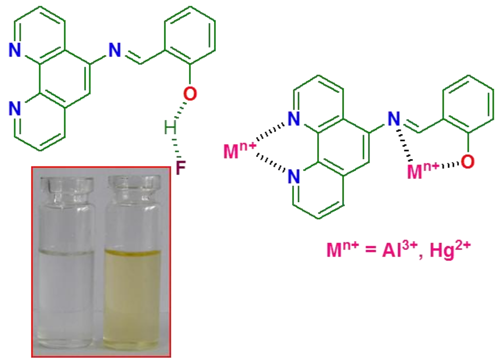
- Novel salicylidene Schiff base (**1**) receptor appended with 1, 10-phenanthroline for selectively sensing F^- ions.
- Colorless solution of **1** turns yellow with F^- ions.
- Receptor **1** recognizes Al^{3+} and Hg^{2+} ions with 1:2 stoichiometry.
- Receptor **1** mimics the functioning of “TRANSFER” and “INHIBIT” molecular logic gates.


N
N
N
O
H
F
M^{n+}
N
N
N
M^{n+}
O
M^{n+} = Al^{3+}, Hg^{2+}