

Salicylaldehyde based Schiff base as a selective and sensitive chemosensor for Cd<sup>2+</sup> and Ni<sup>2+</sup> ionsUnnati P. Patel<sup>a</sup>, Ranjitsinh C. Dabhi<sup>a</sup>, Kartik K. Vaja<sup>a</sup>, Rakesh S. Jani<sup>b</sup> and Jayesh J. Maru<sup>c\*</sup><sup>a</sup>Department of Chemistry, University School of Sciences, Gujarat University, Ahmedabad-380009, India<sup>b</sup>Department of Chemistry, R R Lalan College, Bhuj-370001, India**CHRONICLE**

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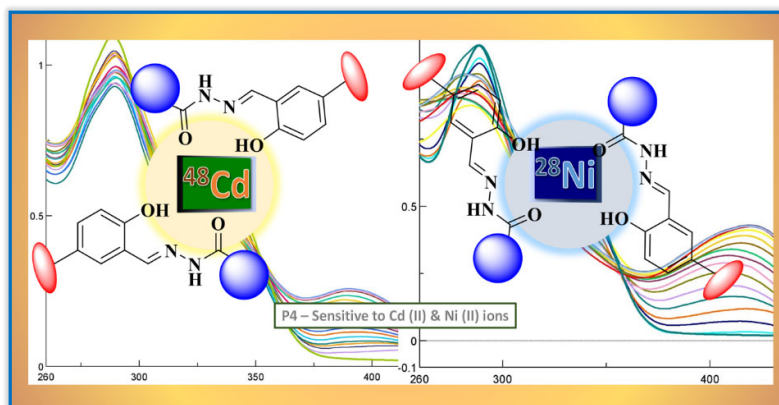
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**ABSTRACT**

A new salicylaldehyde-based sensor *N'*-(5-bromo-2-hydroxybenzylidene)-4-methylbenzohydrazide (**P4**) was designed, and its sensing ability towards various metal ions was assessed using UV-visible spectroscopy. The structure of **P4** was spectroscopically characterized by UV-Visible, Infrared, <sup>1</sup>H-NMR, <sup>13</sup>C-NMR spectroscopy and mass spectrometry. UV-Visible absorption spectral study showed selective sensing ability of **P4** for Cd<sup>2+</sup> and Ni<sup>2+</sup> ions in presence of diverse transition metal ions. The results revealed that the detection limit for Cd<sup>2+</sup> and Ni<sup>2+</sup> was 3.14 μM and 0.92 μM, respectively. Moreover, the compound **P4** was fully implemented for detection of Cd<sup>2+</sup> and Ni<sup>2+</sup> in simulated samples.

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Graphical abstract

**1. Introduction**

The development of transition metal ion sensors is one of the most essential and intriguing research domains owing to its special significance in medicine, environmental, and biological sciences. This is essentially due to their toxicity and dysfunction caused by excessive or lower concentration levels in living organisms.<sup>1-6</sup> Nickel is a key component of numerous metalloenzymes, including acireductone dioxygenases (ARDs), carbon monoxide dehydrogenases (CODHs),

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hydrogenases, and plays a vital role in biochemical processes such as biosynthesis, metabolism, and respiration.<sup>7</sup> Moreover, nickel compounds are also used in a variety of commercial and industrial applications like welding, machinery, electroplating, Ni-Cd batteries, painting pigments, and as a catalyst precursor.<sup>8,9</sup> Nickel accumulation beyond the acceptable limits has a deleterious influence on human health, resulting in pneumonitis, allergy, lung cancer, asthma, and is also responsible for disorder of respiratory system and central nervous system.<sup>10,11</sup>

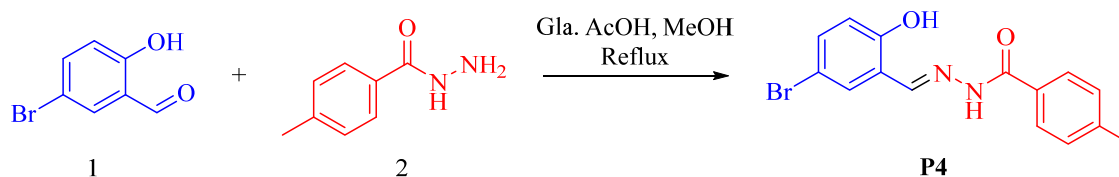
The United State Environmental Protection Agency (USEPA) has identified cadmium as a priority pollutant.<sup>12</sup> However, it is an essential element that is often used in metallurgy, electroplating, agriculture, and the military industry.<sup>4,13</sup> On the other hand, cadmium and its compounds are toxic and prolonged exposure to this metal causes cancer and is also known to target body's cardiovascular, gastrointestinal, reproductive, and respiratory systems.<sup>13,14</sup> Thus, it is essential to design and develop chemosensors, with high selectivity and sensitivity towards  $\text{Cd}^{2+}$  and  $\text{Ni}^{2+}$ . There are different applications for organic molecules, and this is very important advantage for these compounds as reported before.<sup>15-21</sup> Schiff bases are a notable class of compounds with a variety of applications in numerous fields such as medicinal, catalysis, corrosion inhibition etc.<sup>22-26</sup> The imine functional group in the Schiff base is known to bind with various metal ions and thus can induce intramolecular charge transfer or ligand-to-metal charge transfer. As a result, they are used to develop fascinating chemosensors.<sup>27-33</sup>

Based on the above facts, in the present work we synthesized a novel salicylaldehyde-based chemosensor **P4** that can efficiently sense  $\text{Cd}^{2+}$  and  $\text{Ni}^{2+}$  in presence of diverse cations as evident from UV-Visible absorption spectroscopy.

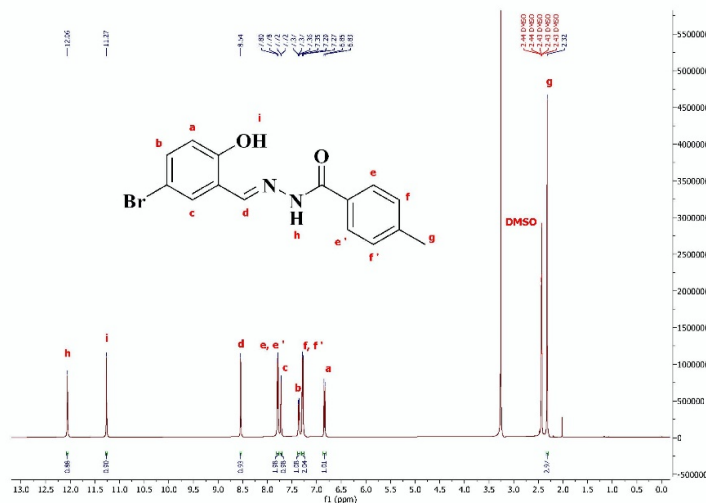
## 2. Results and Discussion

### 2.1 Synthesis and Characterization

The synthesis route of *N*-(5-bromo-2-hydroxybenzylidene)-4-methylbenzohydrazide (**P4**) is illustrated in **Scheme 1**. The compound, **P4** was characterized by Infrared, <sup>1</sup>H-NMR, <sup>13</sup>C-NMR spectroscopy, and mass spectrometry. The IR spectrum showed a sharp peak at 1644  $\text{cm}^{-1}$  assigned to (C=O) vibration. The presence of bromine has been identified by its characteristic peaks at 333.3 and 335.3 with almost equal intensity in the mass spectra. The <sup>1</sup>H-NMR spectrum showed a singlet at  $\delta$  11.27 due to amidic N-H. Another singlet peak at  $\delta$  12.06 was ascribed to the phenolic OH group. Singlet peak of one hydrogen at  $\delta$  8.54 indicated the presence of unsaturated C-H of the imine group which is directly connected to the phenyl ring (**Fig 1**).



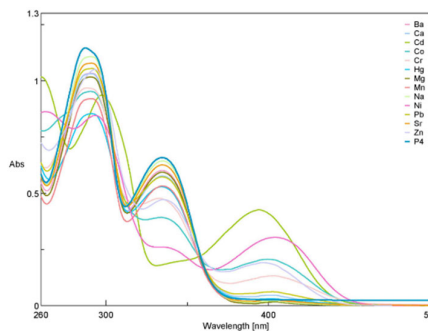
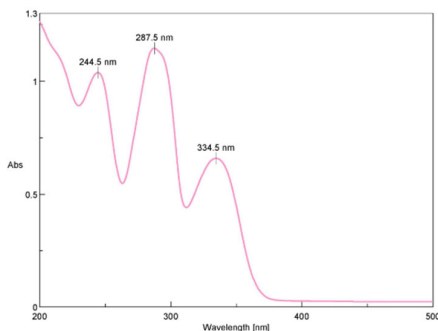
**Scheme 1.** Synthesis of chemosensor **P4**.



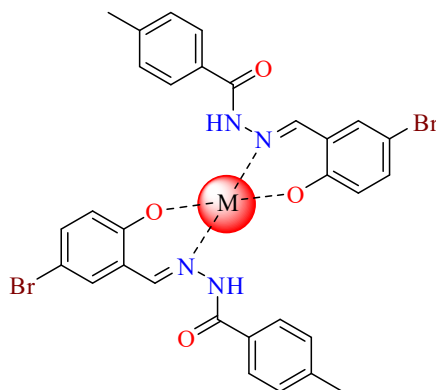
**Fig 1.** <sup>1</sup>H-NMR spectral data of **P4**.

## 2.2 Sensing Ability of P4

The compound, **P4** in acetonitrile was subjected to UV-Visible absorption studies to evaluate its sensing abilities towards several metal ions including  $\text{Ba}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Hg}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Na}^+$ ,  $\text{Ni}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Zn}^{2+}$ . Four absorbance bands with  $\lambda_{\text{max}}$  at 217nm, 244nm, 289nm, and 334nm appeared in the UV-Vis spectra of **P4**, three of which (244nm, 289nm, and 334nm) could be related to intra ligand  $\pi$  to  $\pi^*$  transitions (**Fig 2**).<sup>34</sup> There were no spectral changes observed in the  $\lambda_{\text{max}}$  of **P4** upon addition of different metal ions such as  $\text{Ba}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Hg}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Na}^+$ ,  $\text{Ni}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Zn}^{2+}$ , while a new absorbance band at 387nm and 426nm appeared with the red shift of 53 nm and 92 nm upon interactions with  $\text{Cd}^{2+}$  and  $\text{Ni}^{2+}$  ions, respectively (**Fig 3**). The sensor, **P4** coordinates with the metal ions through the nitrogen and oxygen of phenolic -OH group as illustrated in **Scheme 2**.



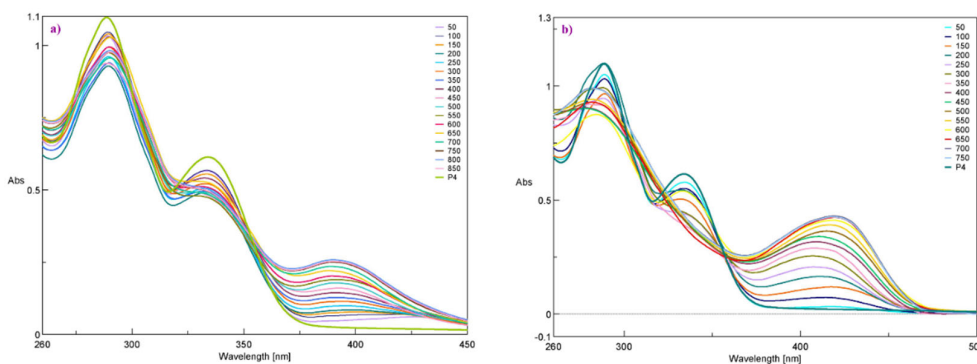
**Fig. 2.** UV-Visible absorption spectra of **P4** in acetonitrile. **Fig. 3.** UV-Visible absorption spectra of **P4** (50  $\mu\text{M}$ ) with various metal ions (200  $\mu\text{M}$ ).



**Scheme 2.** Plausible coordination sites of the probe **P4** with metal ions.

## 2.3 UV-Visible spectral study

The absorption (UV-Visible) spectral titration was performed by adding  $\text{Cd}^{2+}$  and  $\text{Ni}^{2+}$  solutions with varying concentrations from 50  $\mu\text{M}$  to 1000  $\mu\text{M}$  to **P4** in acetonitrile (**Fig 4**). The limit of detection for  $\text{Cd}^{2+}$  and  $\text{Ni}^{2+}$  ions was found to be 3.14  $\mu\text{M}$  and 0.92  $\mu\text{M}$ , respectively.



**Fig 4.** UV-Visible absorption spectra of **P4** (50  $\mu\text{M}$ ) in acetonitrile with varying concentration of a)  $\text{Cd}^{2+}$  (50, 100, 150, 200, 250, 300, 350, 400, 450, 500, 550, 600, 650, 700, 750, 800, 850  $\mu\text{M}$ ) b)  $\text{Ni}^{2+}$  (50, 100, 150, 200, 250, 300, 350, 400, 450, 500, 550, 600, 650, 700, 750  $\mu\text{M}$ )

### 3. Conclusion

In conclusion, we have synthesized a novel Schiff base derivative, **P4** for sensing  $\text{Cd}^{2+}$  and  $\text{Ni}^{2+}$  in aqueous medium. Several experiments were conducted to validate the viability of our design concept. This study emphasizes the detection of  $\text{Cd}^{2+}$  and  $\text{Ni}^{2+}$  with a lower limit of 3.14  $\mu\text{M}$  and 0.92  $\mu\text{M}$ , respectively. Furthermore, other cations showed an imperceptible change in the proposed sensing method. The simple and novel compound **P4** showed excellent sensing performance with good selectivity, lower detection limits and high sensitivity.

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### 4. Experimental

#### 4.1. Chemicals, solvents and instruments

All the required chemicals including 4-methylbenzohydrazide, 5-bromosalicylaldehyde, and metal salts were purchased either from Sigma-Aldrich or Spectrochem. The analytical grade solvents were procured from Finar and used without further rectification. The nitrate salts of various ions such as  $\text{Ba}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Hg}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Na}^+$ ,  $\text{Ni}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Zn}^{2+}$  were purchased from Finar. UV-Visible absorption spectra were performed using the JascoV700 spectrophotometer.  $^1\text{H}$  and  $^{13}\text{C}$  NMR were recorded in Bruker Ascend 500 and LC-MS Spectra were recorded on Waters using electrospray ionization in the positive as well as negative ionization mode. Infrared spectra were obtained from Bruker Alpha-II FT-IR spectrometer.

#### 4.2. Synthesis of *N'*-(5-bromo-2-hydroxybenzylidene)-4-methylbenzohydrazide (**P4**).

Equimolar quantities (0.1mmol) of 4-methylbenzohydrazide (**1**) and 5-bromosalicylaldehyde (**2**) were dissolved in 5mL methanol in a round bottom flask with vigorous stirring. The reaction mixture was refluxed for 30 min after addition of 0.5 mL of glacial acetic acid. The reaction progress was monitored by TLC. After completion of the reaction, the crude product was dumped into ice water and the precipitate formed was filtered, washed with cold water and dried.<sup>35</sup>

#### 4.3. Physical and spectral data of *N'*-(5-bromo-2-hydroxybenzylidene)-4-methylbenzohydrazide (**P4**).

Schiff base **P4**: Pale yellow; Yield: 85%; M.P.: 220° C; MS (ESI): *m/z* for  $\text{C}_{15}\text{H}_{13}\text{BrN}_2\text{O}_2$  Calculated: 333.02  $[\text{M}+\text{H}]^+$  Found: 333.3, 335.3; Calculated: 331.02  $[\text{M}-\text{H}]^-$  Found: 331.1, 333.1. FT-IR (Diamond crystal) in  $\text{cm}^{-1}$ : 1465, 1515, 1547 (C=C Stretching aromatic), 1644 (-C=O Stretching), 2910 (-C-H Stretching aromatic), 3743 (-O-H stretching).  $^1\text{H}$  NMR (500 MHz, DMSO-*d*6)  $\delta$  12.06 (s, 1H, -NH), 11.27 (s, 1H, -OH), 8.54 (s, 1H, N=CH), 7.80-7.78 (d, 2H, Ar-H), 7.72 (s, 1H, Ar-H), 7.37-7.35 (d, 1H, Ar-H), 7.29-7.27 (d, 2H, Ar-H), 6.85-6.83 (d, 1H, Ar-H), 2.32 (s, 3H, -CH<sub>3</sub>).  $^{13}\text{C}$  NMR (126 MHz, DMSO-*d*6)  $\delta$  163.26, 156.90, 145.93, 142.65, 133.97, 130.97, 130.31, 129.56, 128.19, 121.81, 119.16, 110.89, 21.54

#### 4.4. Investigation of spectral studies

The solution of Schiff base **P4** ( $10^{-3}$  mol/L) was prepared in acetonitrile. The stock solutions of various metal ions ( $2 \times 10^{-3}$  mol/L) were prepared by dissolving nitrates salts of  $\text{Ba}^{2+}$ ,  $\text{Ca}^{2+}$ ,  $\text{Cd}^{2+}$ ,  $\text{Co}^{2+}$ ,  $\text{Cr}^{3+}$ ,  $\text{Hg}^{2+}$ ,  $\text{Mg}^{2+}$ ,  $\text{Mn}^{2+}$ ,  $\text{Na}^+$ ,  $\text{Ni}^{2+}$ ,  $\text{Pb}^{2+}$ ,  $\text{Sr}^{2+}$ ,  $\text{Zn}^{2+}$  in milli-Q-water. An aliquot (200  $\mu\text{L}$ ) of stock solutions of different metal ion was mixed with 100  $\mu\text{L}$ ,  $10^{-3}$  mol/L solution of **P4** and the solution was made up to 2.0 mL with acetonitrile. The resultant change in the absorbance or shift in the wavelength was observed in the UV-Visible absorption spectra.<sup>3, 36</sup> The LOD (limit of detection) value was calculated based on formula  $3\sigma/S$ , where the slope *S* and standard deviation  $\sigma$  of the intercept was obtained from the calibration curve.<sup>7</sup>

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