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Short Note

N-[(1*E*)-(3-Bromophenyl)methylene]-*N*-(2-piperidin-1-ylethyl)amine

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Abstract: *N*-[(1*E*)-(3-Bromophenyl)methylene]-*N*-(2-piperidin-1-ylethyl)amine Schiff base was prepared in good yield and characterized by the reflux of equivalent amounts of 2-(piperidin-1-yl)ethanamine with 3-bromobenzaldehyde. The structure of the desired Schiff base was analyzed based on: elemental analysis, EI-MS, TG/DTG, UV-visible, FT-IR, ^{1}H and ^{13}C-NMR spectral analysis. The condensation reaction was monitored by FT-IR.

Keywords: Schiff base; NMR; FT-IR; condensation; TG/DTG

1. Introduction

Schiff bases as nitrogen unsaturated organic compounds are versatile ligands which have received a great deal of attention in many area of research [1,2], as corrosion inhibitors, pigments and dyes, and small-molecule catalysts activators and in biological activities [2–6]. The Schiff base, structurally known as azomethine or imine, is a nitrogen analogue of ketone or aldehyde in which the carbonyl group has been replaced by an azomethine group after water molecule elimination [3]. Several azomethines possess remarkable antibacterial, antifungal, anticancer and diuretic activities. The lone pair of electrons belongs to the nitrogen atom of azomethine which is expected to be involved in the formation of an H-bond with the active centers of the target cell constituents and interferes in normal cell function [4].

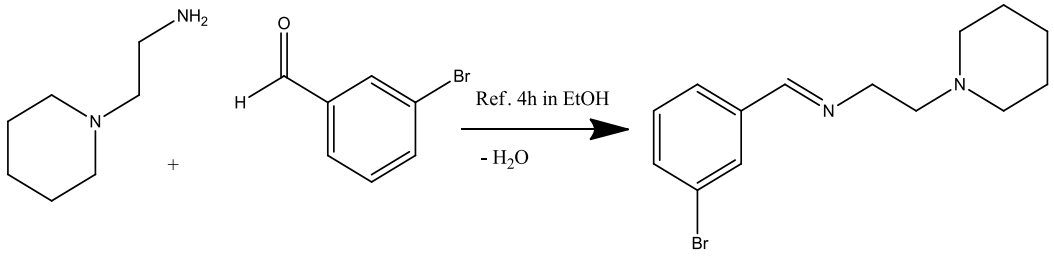
Due to the presence of >C=N-, an electron-rich functional group (π and N-free lone pair electrons), Schiff bases, as ligands, have played a very important role in the development or building of new metal ion complexes [7–9]. Despite the huge number of organic compounds, there is always an urgent need to develop new chelated ligands and enhance their properties as well as their applications.

In connection with previous research that belongs to our group on the synthesis, characterization, complexation and biological application of mono- or polydentate Schiff bases [6–11], in this work *N*-[(1*E*)-(3-bromophenyl)methylene]-*N*-(2-piperidin-1-ylethyl)amine was synthesized and characterized by various spectral techniques. The reaction was monitored by IR. The entitled compound showed promising antibacterial activities, and further future works will be carried out.

2. Results and Discussion

N-[(1*E*)-(3-bromophenyl)methylene]-*N*-(2-piperidin-1-ylethyl)amine was synthesized by condensing equimolar amounts of 2-(piperidin-1-yl)ethanamine and 3-bromobenzaldehyde in absolute ethanol under reflux conditions for 4 h, as shown in Scheme 1. The product is colorless, naturally oily,

completely soluble in dichloromethane and partially soluble in alcohols, and insoluble in water and non-polar solvents such as *n*-hexane.



Scheme 1. Synthesis of the *N*-[(1*E*)-(3-bromophenyl)methylene]-*N*-(2-piperidin-1-ylethyl)amine.

EI-MS of the compound is in good agreement with the assigned structure and showed the experimental molecular ion [M^+] *m/z* = 295.0 (295.2 theoretical).

The thermal properties of the TG/DTA of the title compound were investigated under an open atmosphere in the temperature range of 0 °C–250 °C and a heating rate of 10 °C/min. Figure 1 showed that one broad step of typical decomposition started from 98 °C and ended at 135 °C, with exothermic DTA = 128 °C signal and weight loss of ~99% (no intermediate decomposition steps).

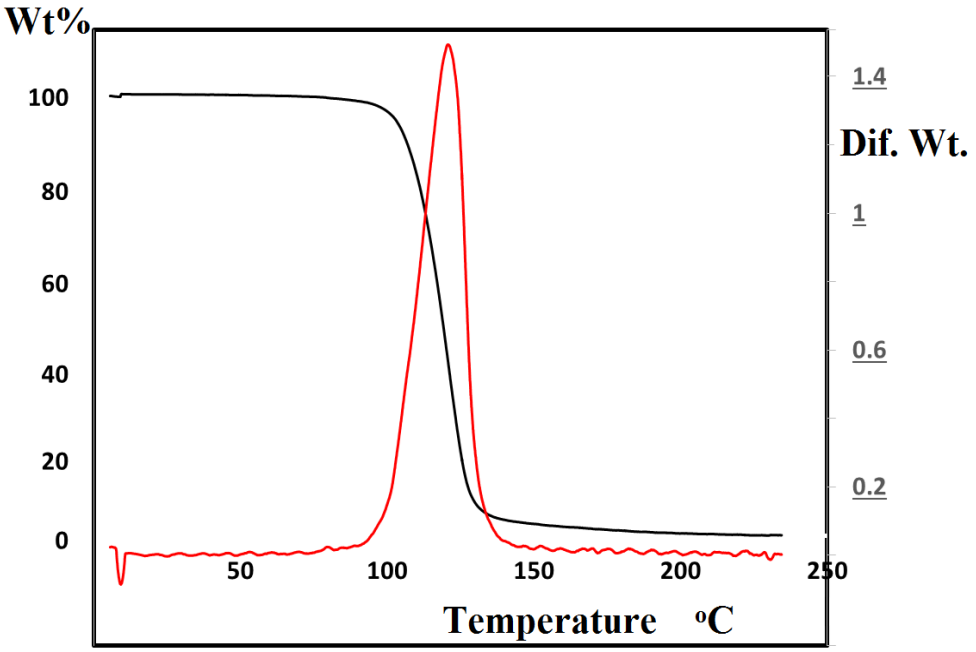


Figure 1. TG/DTA thermal curve of the desired compound.

We have monitored the reaction by using FT-IR spectroscopy. The starting materials were followed up before and after its condensation to produce the desired compound by IR measurement, as seen in Figure 2. The formation of the product was confirmed as follows: the aldehyde C=O (at 1690 cm^{-1}) shifted to C=N (at 1645 cm^{-1}) as seen in Figure 2a,c. The disappearance of the N-H amine and C-H aldehyde of stretching vibrations at 3266 and 3110 cm^{-1}, respectively, supported the success of the condensation of the starting materials to form the desired product, as seen in Figure 2.

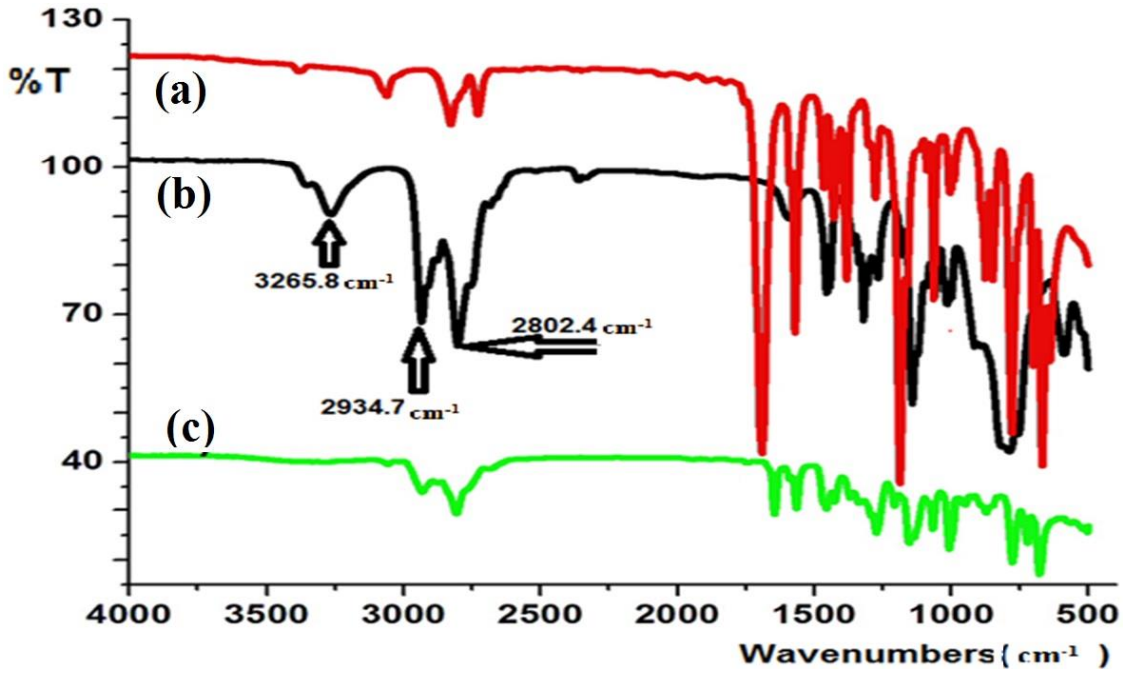


Figure 2. IR spectra of: (**a**) 3-bromobenzaldehyde (starting material), (**b**) 2-(piperidin-1-yl)ethanamine (starting material) and (**c**) *N*-[(1*E*)-(3-bromophenyl)methylene]-*N*-(2-piperidin-1-ylethyl)amine (product).

The electronic absorption spectrum of the prepared compound was acquired in CH_2Cl_2; Figure 3 shows the electronic absorption as expected (in the UV region only) at λ_{max} = 215 and 250 nm which are assigned mainly to intra-ligand π-π* transitions.

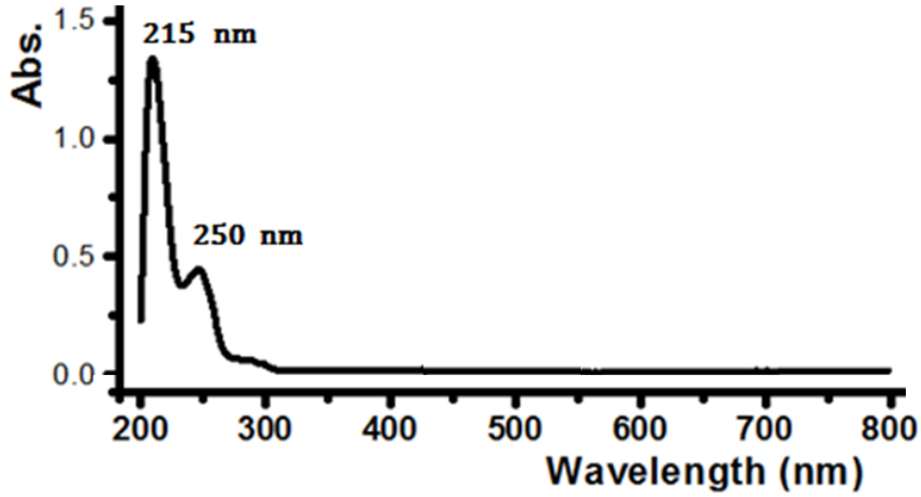


Figure 3. UV-vis spectrum of 1×10^{-5} M product dissolved in CH_2Cl_2 at RT.

The ^{1}H-NMR spectrum data is in a good agreement with its assigned structure; the signals of the aromatic, aliphatic and amide function groups are cited to their positions. The ^{1}H-NMR spectrum of the compound is shown in Figure 4. The ^{1}H-NMR spectrum showed multiple singlets at δ 1.21, 2.65, 2.76, 2.90 and δ 3.76 ppm due to aliphatic protons. The four aromatic protons resonated as a multiplet in the region of δ 7.2–7.9 ppm, and the azomethine proton resonated as a singlet at δ 8.28 ppm.

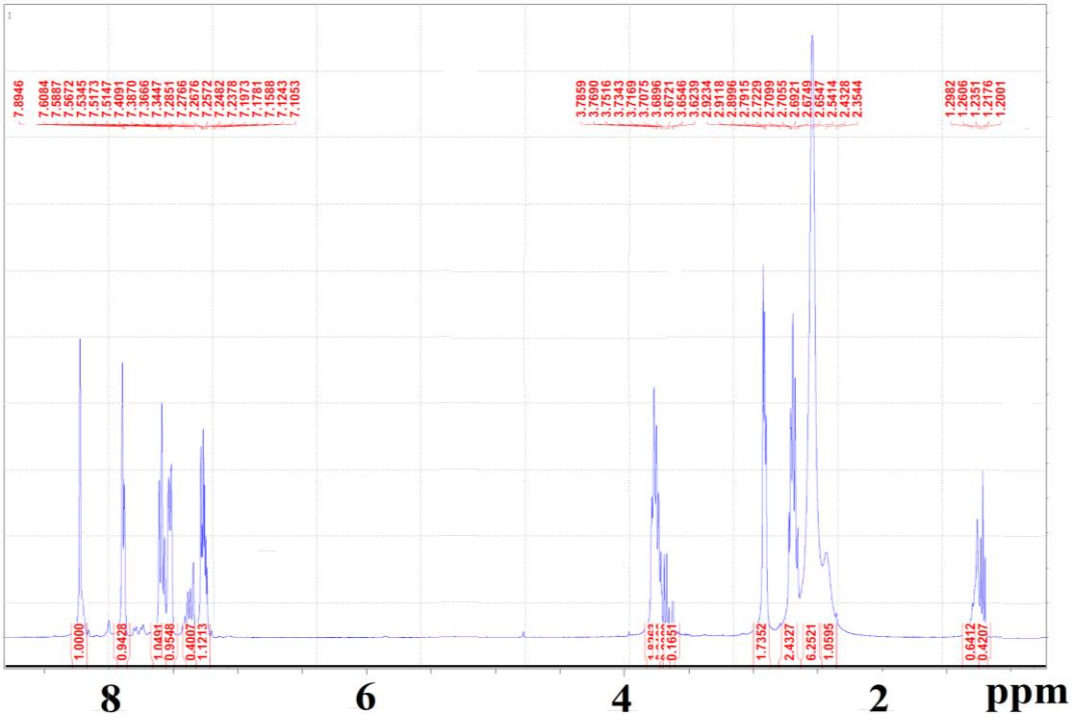


Figure 4. ^{1}H-NMR (ppm) spectrum of desired compound in $CDCl_3$ at RT.

The ^{13}C-NMR spectrum revealed several sets of signals: six signals were cited to aromatic carbons in the region of ~120–140 ppm, five types of aliphatic carbons were cited to the region of ~45–90 ppm and the azomethine carbon was cited to 161.3 ppm, as seen in Figure 5. Figure 5 showed the chemical shifts of all the carbons together with their numbers.

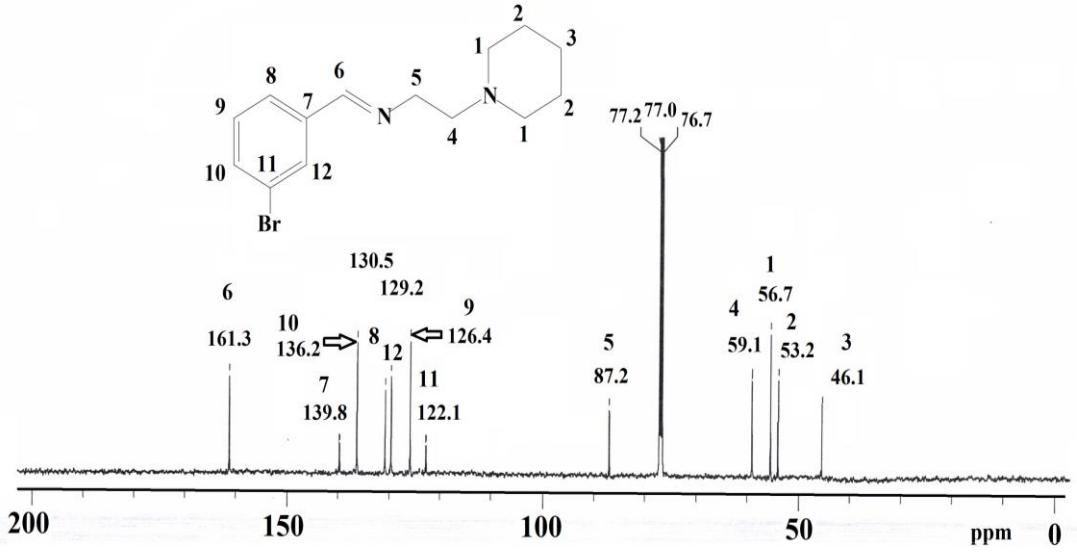


Figure 5. ^{13}C-NMR (ppm) spectrum of desired compound in $CDCl_3$ at RT.

3. Experimental Section

The UV-visible spectrum was measured on TU-1901 double-beam UV-visible spectrophotometer. The IR spectra for samples were recorded by PerkinElmer Spectrum 1000 FT-IR Spectrometer (PerkinElmer Inc., Waltham, MA, USA). High-resolution NMR was recorded on Bruker DRX 500

spectrometer (Bruker, Mainz, Germany) (^{1}H, 500 MHz frequency, ^{13}C, 125 MHz) at 298 K. EI-MS data was obtained on a Finnigan 711A (8 kV) (PerkinElmer Inc.). TG spectrum was measured by using a TGA-7 PerkinElmer thermogravimetric analyzer (PerkinElmer Inc.).

A solution of 3-bromobenzaldehyde (2 mmol) in EtOH (30 mL) was mixed with 2-(piperidin-1-yl)ethanamine (2.1 mmol) and allowed to reflux under stirring for 4 h. The resulting mixture was concentrated under reduced pressure and the title compound was precipitated as oil upon addition of 100 mL of *n*-hexane. The oily product was washed three times with (80 mL) of distilled water.

The elemental analysis of the compound is consistent with the proposed molecular formula (Calcd. for $C_{14}H_{19}BrN_2$: C, 56.96; H, 6.49; Br, 27.07; N, 9.49. Found: C, 56.85; H, 6.31; Br, 26.88; N, 9.45).

Yield 85%, at RT the product is colorless oil; Molecular formula $C_{14}H_{19}BrN_2$; IR: 3020 $cm^{-1}{}_{\text{C–H Ph}}$, 2970–2780 $cm^{-1}{}_{\text{C-H aliphatic}}$, 1645 $cm^{-1}{}_{\text{C=N}}$. ^{1}H-NMR (500 MHz, $CDCl_3$): δ (ppm) 1.21 (m, 2H, $NCH_2CH_2\underline{CH_2}$), 2.65 (m, 4H, $NCH_2\underline{CH_2}CH_2$), 2.76 (2m, 4H, $N\underline{CH_2}CH_2CH_2$), 2.90 (m, 2H, $=NCH_2\underline{CH_2}N$), 3.76 (m, 2H, $=N\underline{CH_2}CH_2N$), 7.0–8.0 (4m, 4H, Ph), 8.28 (s, 1H, HC=N). ^{13}C-NMR (125 MHz, $CDCl_3$): δ (ppm) 46.1 (CH2, 1C, C3), 53.2 (CH2, 2C, C2), 56.1 (CH2, 2C, C1), 59.1 (CH2N, 1C, C4), 87.2 (CH2N, 1C, C5), 122.1 (CHBr, 1C, C11), 126.4 (CH, 1C, C9), 129.2 (CH, 1C, C12), 130.5 (CH, 1C, C8), 46.1 (CH, 1C, C10), 46.1 (C, 1C, C7), 161.3 (CHN, 1C, C6). $[M^+]$ = 295.0 *m/z*.

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Author Contributions: Y.A.-D. performed the experiments; A.A.-A. and S.A. measured and analyzed the NMR; M.A.-N. and M.S. helped in discussion the results and I.W. wrote the paper.

Conflicts of Interest: The authors declare no conflict of interest.

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