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Synthesis, spectral and comparative antimicrobial study of schiff bases

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ABSTRACT

Schiff bases are very important in medicinal and pharmaceutical fields because of their wide spectrum of biological activities. Most of them show biological activities such as antibacterial, antifungal as well as antitumor activity. Transition metal complexes derived from the Schiff base ligands with biological activity have been widely studied. This research paper has been aimed at the synthesis of some novel Schiff bases of biological importance. The synthesized Schiff bases have been characterized by IR, NMR, Mass Spectra followed by Elemental analysis for carbon, hydrogen and nitrogen. All the compounds have been screened for their in vitro antibacterial activity against two Gram +ve (*Staphylococcus aureus, Bacillus subtilis*) and two Gram –ve (*Escherichia coli, Pseudomonas aeruginosa*) bacterial strains by agar-well diffusion method. The Schiff bases have been found to exhibit varied activity against different bacterial species.

Key words: Schiff bases, Spectral study and Antimicrobial Study.

1. INTRODUCTION

Compounds containing an azomethine group (-CH=N-), known as Schiff bases are formed by the condensation of a primary amine with a carbonyl compound. Schiff bases of aliphatic aldehydes are relatively unstable and are readily polymerizable while those of aromatic aldehydes, having an effective conjugation system, are more stable. Schiff bases have number of applications viz., preparative use, identification, detection and determination of aldehydes or ketones, purification of carbonyl or amino compounds, or protection of these groups during complex or sensitive reactions. They also form basic units in certain dyes. In organic synthesis, Schiff base reactions are useful in making carbon-nitrogen bonds. Schiff bases appear to be an important intermediate in a number of enzymatic reactions involving interaction of an enzyme with an amino or a carbonyl group of the substrate. One of the most important types of catalytic mechanism is the biochemical process which involves the condensation of a primary amine in an enzyme usually that of a lysine residue, with a carbonyl group of the substrate to form an imine, or Schiff base.

In this paper we have discussed the synthesis and antimicrobial activity of some novel cyclic and heterocyclic Schiff bases of biological significance. From the literature survey Synthesis[1-8] and biological activities of Schiff bases have been studied which have been reported by different authors like antibacterial[9-18], antifungal[19-25], pesticidal[26], anti-inflammatory[27] and antiviral[28].

2. MATERIAL AND METHODS

2.1. Materials

All chemicals used were of A.R. grade. Melting points were determined using open glass capillaries and are uncorrected. NMR spectra were recorded on NMR-Spectrometer (Bruker 300 M Hz). The ^{1}H-chemical shifts are expressed in ppm relative to tetramethylsilane (Me_4Si). IR spectra were recorded on FT-IR Spectrometer (Lambda Scientific). Micro analysis for C, H, and N were performed on Perkin Elmer CHN Analyzer. Mass spectra were obtained on Shimadzu GCMS-QP-2000 Mass Spectrometer.

2.2. General method for preparation of Schiff bases

0.1mole of amine (aniline, phenyl-hydrazine, 2,4-dinitrophenyl-hydrazine etc.) and 0.1 mole of carbonyl compound (antipyrine, furfuraldehyde, p-hydroxy benzaldehyde etc.) were taken and mixed in glacial acetic acid (25 ml) the resultant mixture was refluxed for (1.5-10 Hrs.), cooled the precipitates and poured on to crush ice. The precipitates were filtered and washed with distilled water (2x10 ml). Recrystalization from ethanol afforded the purified Schiff base. Compounds were also purified by silica gel column chromatography (eluent: ethylacetate/hexane :: 1-3:9-7). The

reaction was monitored by thin layer chromatography (TLC) and spots were visualized in iodine chamber.

2.2.1. Comp. No. 1: (2)-1-(2, 4-Dinitrophenyl)-2-(furan-2-ylmethylidene) hydrazine

Yield 60%, maroon powder; m.p. 179-181 ^{0}C; IR (KBr) cm^{-1}: 3275 (N-H str.), 1661 (C=N str.), 1509 [(Ar-NO_2) asym. str.], 1321 [(Ar-NO_2) sym. str.], 1138 (C-NH str.). ^{1}H NMR ($CDCl_3$): δ: 3.55 (s, 1H, Ar.-NH, exchangeable with D_2O), 6.06 (s, 1H, -CH=N-), 6.56 (dd, 1H, Furyl C_4-H), 6.63 (d, 1H, Furyl C_3-H), 6.86 (d, 1H, Furyl C_5-H), 8.31-8.36 (m, 3H, 2,4-Dinitrophenyl). MS (m/z) 276.2 ($M^{\cdot+}$), 276.2, 275.1, 246.1, 230.2, 201.2, 200.2. Anal. Calcd. for $C_{11}H_8N_4O_5$: C, 47.83; H, 2.92 ; N, 20.29 Found : C, 47.76; H, 2.90 ; N, 20.38.

2.2.2. Comp. No. 2: 4{[2(2,4-Dinitrophenyl)hydrazinylidene]methyl}-N,N-dimethyl aniline

Yield 57%, bright black powder; m.p. 103-105 ^{0}C; IR (KBr) cm^{-1}: 3278 (N-H str.), 1664 (C=N str.), 1509 [(Ar-NO_2) asym. str.], 1327 [(Ar-NO_2) sym. str.], 1131 (C-NH str.). ^{1}H NMR ($CDCl_3$) :δ: 3.09 [s, 6H, -N(CH_3)$_2$], 3.49 (s, 1H, Ar.-NH, exchangeable with D_2O), 6.09 (s, 1H, -CH=N-), 6.78 (d, 2H, J=9.0 Hz., H_b of -C_6H_4-N<), 7.79 (d, 2H, J=9.0 Hz., H_a of -C_6H_4-N<), 8.09-8.32 (m, 3H, 2,4-Dinitrophenyl). MS (m/z) 329.3 ($M^{\cdot+}$), 328.3, 314.2, 147.3, 146.3, 120.1, 103.1. Anal. Calcd. for $C_{15}H_{15}N_5O_4$: C, 54.71; H, 4.59; N, 21.27. Found C, 54.78; H, 4.52; N, 21.38.

2.2.3. Comp. No. 3: 4-{-[2-(2,4Dinitrophenyl) hydrazinylidene] methyl}phenol

Yield 65%, dark maroon powder; m.p. 250-252 ^{0}C; IR (KBr) cm^{-1}: 3300 (O-H str.), 3265 (N-H str.), 1655 (C=N str.), 1513 [(Ar NO_2) asym. str.], 1330 [(Ar-NO_2) sym. str.], 1141 (C-N str.). ^{1}H NMR (DMSO-d_6) :δ: 3.56 (s, 1H, -NH, exchangeable with D_2O), 5.55 (s, 1H, Ar.-OH, exchangeable with D_2O), 6.07 (s, 1H, -CH=N-), 6.70 (d, 2H, J=9.0 Hz., H_b of -C_6H_4-OH), 7.79 (d, 2H, J=9.0 Hz., H_a of -C_6H_4-OH), 8.28-8.52 (m, 3H, 2,4- Dinitrophenyl). MS (m/z) 302.2 ($M^{\cdot+}$), 302.2, 272.2, 271.2, 118.1, 103.1, 102.1. Anal. Calcd. for $C_{13}H_{10}N_4O_5$:C, 51.66; H, 3.34; N, 18.54; Found C, 51.51; H, 3.37; N, 18.60.

2.2.4. Comp. No. 4: N, N-dimethyl-4-[(2phenylhydrazinylidene) methyl} aniline

Yield 67%, bright brownish black powder; m.p. 142-144 ^{0}C; IR (KBr) cm^{-1}: 3273 (N-H), 1662 (C=N str.), 1140 (C-NH str.). ^{1}H NMR ($CDCl_3$) : δ: 2.99 [s, 6H, -N-(CH_3)$_2$], 3.52 (s, 1H, Ar.-NH, exchangeable with D_2O), 6.10 (s, 1H, Ar.-CH=N-), 6.72 (d, 2H, J=9.0 Hz., H_b of -C_6H_4-N<), 7.06-7.71 (m, 5H, J=9.0 Hz., H_a of -C_6H_4-N<), 7.85 (d, 2H, -Ph-N<), MS (m/z) 239.3 ($M^{\cdot+}$), 238.5, 209.2, 147.1, 162.2, 120.2. Anal. Calcd. for $C_{15}H_{17}N_3$: C, 44.29; H, 2.66; N, 18.45. Found C, 44.85; H, 2.45; N, 18.30.

2.2.5. Comp. No. 5: N, N-dimethyl-4-[(Phenylimino) methyl] aniline

Yield 69%, yellowish-green powder; m.p. 100-102 ^{0}C, IR (KBr) cm^{-1}: 1659 (C=N str.), 1132 (C-N str.). ^{1}H NMR ($CDCl_3$) :δ: 3.08 [s, 6H, -N-(CH_3)$_2$], 6.04 (s, 1H, -CH=N-), 6.75 (d, 2H, J=9.0 Hz., H_b of -C_6H_4-N<), 7.14-7.42 (m, 5H, Ph-N=) 7.82 (d, 2H, J=9.0 Hz., H_a of -C_6H_4-N<). MS (m/z) 224.3 ($M^{\cdot+}$), 224.3, 209.3, 120.1, 102.1. Anal. Calcd. for $C_{15}H_{16}N_2$: C, 80.32; H, 7.19; N, 12.49. Found C, 80.85; H, 7.25; N, 12.43.

2.2.6. Comp. No. 6: 2-{-[2-(2, 4-Dinitrophenyl) hydrazinylidene] methyl}phenol

Yield 70%, red powder; m.p. 253-255 ^{0}C; IR (KBr) cm^{-1}: 3309 (O-H str.), 3269 (N-H str.), 1657(C=N str.), 1513 [(Ar NO_2) asym. str.], 1335 [(Ar-NO_2) sym. str.], 1144 (C-NH str). ^{1}H NMR ($CDCl_3$) :δ: 3.58 (s, 1H, Ar.-NH, exchangeable with D_2O), 5.61 (s, 1H, Ar.-OH, exchangeable with D_2O), 6.05 (s, 1H, Ar.-CH=N-), 6.84-7.08 (m, 4H, Ar. ring-A), 7.99-8.48 (m, 3H, Ar. ring-B). MS (m/z) 302.2 ($M^{\cdot+}$), 302.2, 301.2, 272.2, 120.1, 118.1, 117.1. Anal. Calcd. for $C_{13}H_{10}N_4O_5$: C, 51.66; H, 3.34; N, 18.54; Found C, 51.54; H, 3.39; N, 18.58.

2.2.7. Comp. No. 7: 2-[(2-Phenylhydrazinylidene) methyl] phenol

Yield 69%, light brown powder; m.p. 140-142 ^{0}C, IR (KBr) cm^{-1}: 3308 (O-H str.), 3272 (N-H str.), 1660 (C=N str.), 1139 (C-NH str.). ^{1}H NMR ($CDCl_3$) :δ: 3.51 (s, 1H, Ar.-NH-, exchangeable with D_2O), 5.58 (s, 1H, Ar.-OH exchangeable with D_2O) 6.07 (s, 1H, Ph-CH=N-), 6.86-7.00 (m, 5H, Ar. ring-A), 7.08-7.31 (m, 4H, Ar. ring-B), MS (m/z) 212.2 ($M^{\cdot+}$), 211.1, 183.2, 119.1, 105.1. Anal. Calcd. for $C_{13}H_{12}N_2O$: C, 73.56; H, 5.70; N, 13.20. Found C, 73.48; H, 5.75; N, 13.35.

2.2.8. Comp. No. 8: 1, 5-Dimethyl-2-phenyl-3-[(phenylimino) methyl]-2,3-dihydro-1*H*-pyrazol-4amine

Yield 59%, light brown powder; m.p. 95-97 ^{0}C; IR (KBr) cm^{-1}: 3270 (N-H str.), 1669 (C=N str.), ^{1}H NMR ($CDCl_3$) :δ: 1.25 (s, 3H, C-CH_3), 2.14 (s, 2H, -NH_2,exchangeable with D_2O), 2.51 (s, 1H, N-CH-C=N-), 2.64 (s, 3H, -N-CH_3), 6.08 (s, 1H, -CH=N-), 6.82-7.06 (m, 5H, C_6H_5-N=CH-), 7.09-7.27 (m, 5H, C_6H_5-N<). MS (m/z) 292.4 ($M^{\cdot+}$), 201.3, 200.3, 158.1, 104.1, 103.1. Anal. Calcd. for $C_{18}H_{20}N_4$: C, 73.94; H, 6.90; N, 19.17. Found C, 73.82; H, 6.97; N, 19.38.

2.2.9. Comp. No. 9: 3-{[2-(2,4Dinitrophenyl) hydrazinylidene] methyl}-1, 5-dimethyl-2-phenyl-2, 3-dihydro-1H-pyrazol-4-amine

Yield 70%, brown powder; m.p. 187-189 ^{0}C; IR (KBr) cm^{-1}: 1665 (C=N str.), 1539 [(Ar-NO_2) asym. str.], 1340 [(Ar-NO_2) sym. str.]. ^{1}H NMR ($CDCl_3$) :δ: 1.29 (s, 3H, CH_3-C=), 2.45 (s, 3H, CH_3-N<), 2.52 (s, 2H, NH_2-C, pyrazole), 2.59 (s, 1H, N-CH-C=), 4.21(s, 1H, C_6H_5-NH, exchangeable with D_2O),), 6.11 (s, 1H,-CH=N-), 6.55-7.08 (m, 5H, ring-A), 7.19-8.45 (m, 3H, ring-B). MS (m/z) 397.4 (M^{+}), 367.3, 366.3, 337.4, 320.3, 304.2, 274.3. Anal. Calcd. for $C_{18}H_{19}N_7O_4$: C, 54.40; H, 4.82; N, 24.68 Found C, 54.05; H, 4.89; N, 24.35.

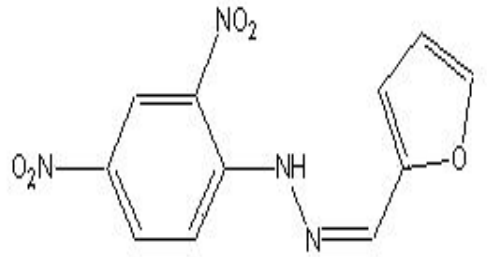
3. RESULT AND DISCUSSION

All the Schiff bases were screened for their in vitro antibacterial activity by agar-well diffusion method against Gram +ve (*Staphylococcus aureus, Bacillus subtilis*) & Gram – ve (*Escherichia coli, Pseudomonas aeruginosa*) microorganisms by preparing 200 µg/ml of test solution of each compound. Zone of inhibitions in mm were noted. The zone of inhibition for Schiff Bases varied from 0 to 20 mm. The results have been shown in Table No. 1, the activity of control (dimethyl sulphoxide) was also checked for its toxicity.

The present investigations suggest that:

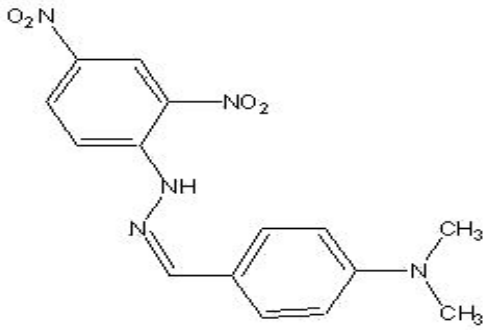
Schiff bases are affective against Gram +ve (*Staphylococcus aureus, Bacillus subtilis*) & Gram – ve (*Escherichia coli, Pseudomonas aeruginosa*) microorganisms. Schiff bases have shown weak to moderate activity. It has been found that amongst all the test compounds taken for antibacterial evaluation, Comp. No. 9 has shown maximum activity against B. subtilis. The activity of control dimethyl sulphoxide was also checked for its toxicity and it has been found that it has no effect on the growth of any microorganisms taken.

FIGURES:



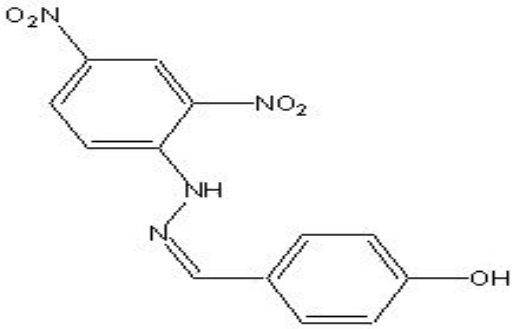
(2)-1-(2,4-Dinitrophenyl)-2-(furan-2-ylmethylidene) hydrazine

Comp. No. 1



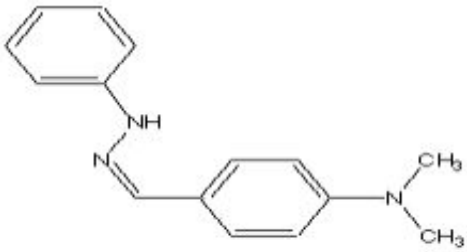
4-{-[2-(2,4-Dinitrophenyl)hydrazinylidene]methyl}-*N*,*N*-dimethylaniline

Comp. No. 2



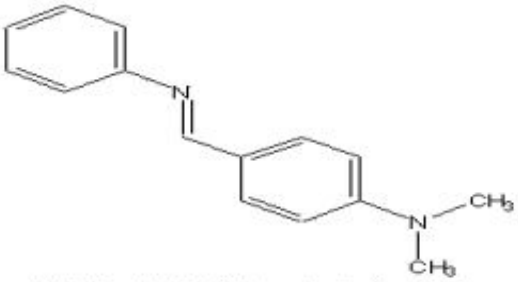
4-{-[2-(2,4-Dinitrophenyl)hydrazinylidene] methyl}phenol

Comp. No. 3



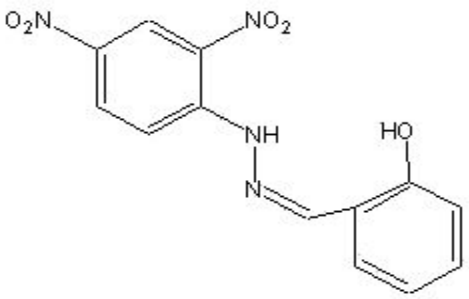
N,*N*-Dimethyl-4-[(2-phenylhydrazinylidene) methyl]aniline

Comp. No. 4



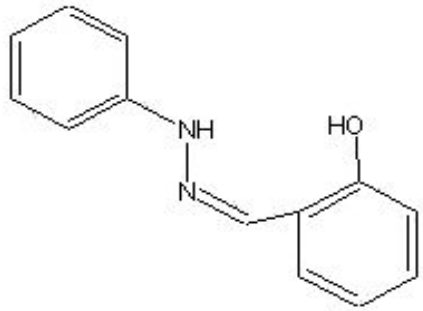
N,*N*-dimethyl-4-[(phenylimino)methyl] aniline

Comp. No. 5



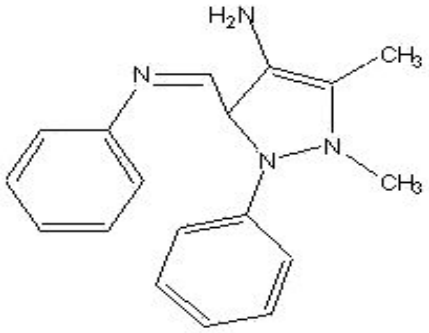
2-{[2-(2,4-Dinitrophenyl)hydrazinylidene]methyl} phenol

Comp. No. 6



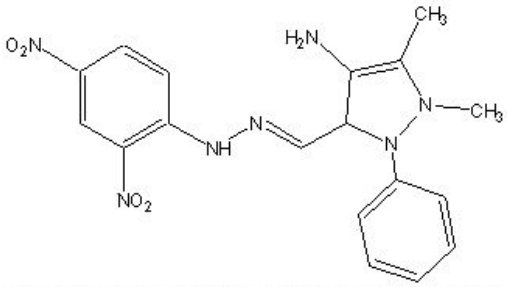
2-[(2-Phenylhydrazinylidene)methyl]phenol

Comp. No. 7



1,5-Dimethyl-2-phenyl-3-[(phenylimino)methyl]-2, 3-dihydro-1*H*-pyrazol-4-amine

Comp. No. 8



3-{-[2-(2,4-Dinitrophenyl)hydrazinylidene]methyl}-1,5-dimethyl -2-phenyl-2,3-dihydro-1*H*-pyrazol-4-amine

Comp. No. 9

Table -1: Screening of Schiff bases for anti-bacterial activity (zone of inhibition in mm.)

MICROBIAL SPECIES				
Compound No.	E. coli	B. subtilis	P. aeruginosa	S. aureus
MTCC Code	1687	441	424	737
1	14	13	00	15
2	15	12	00	14
3	00	09	13	10
4	10	11	00	12
5	13	00	00	09
6	00	10	12	10
7	10	14	16	15
8	00	09	00	13
9	00	20	00	13

00-09: Weak activity
10-16: Moderate activity

3. CONCLUSION

The Schiff bases were found to exhibit either no or low to moderate activity against one or more bacterial species. These studies may serve as a basis for the chemical modifications directed towards the development of a new class of antibacterial agents.

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