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A Focus towards p-vinyl benzaldehyde containing quinoxaline - Photoluminescence and Antibacterial activity

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ABSTRACT: Quinoxaline display a wide range of biological performance and electron transport properties. Poly(p-phenylene vinylene) is one of the most imperative part of conjugated polymers having an ample assortment of applications in light-emitting diodes. The existence of electron withdrawing quinoxaline ring has been used in π-conjugated structures to construct the OLED materials. In the present investigation, p-vinyl benzaldehyde substituted quinoxaline derivatives were synthesized using 3-methyl-quinoxalin-2-one with terephthaldicarboxaldehyde via Wittig reaction. The structures of synthesised compounds were confirmed by FT-IR, ^{1}H, ^{13}C, ^{31}P-NMR, MASS spectral data. The result of Fluorescent investigational examination reveals that p-vinyl benzaldehyde containing quinoxaline derivatives exhibited photoluminescence nature with green emission maxima at shorter wavelengths of 470nm. The Phosphonium compound and p-vinyl benzaldehyde capped quinoxaline derivatives were subjected to four different bacteria viz., Staphylococcus aureus, Bacillus substilis, Escherichia Coli, Pseudomonas auroginosa. A result of the antibacterial studies reveals that compounds acquire substantial activity in assessment with Ampicillin.

Keywords: p-phenylene vinylene, quinoxaline derivatives, photoluminescence, Wittig reaction, spectral studies.

I. INTRODUCTION

Quinoxaline derivatives are an important class of fused heterocycles that display a wide range of biological, pharmacological, and medicinal properties involving antiviral, antibacterial, anti-inflammatory, and anti-protozoal and as kinase inhibitors[1-5]. Many quinoxaline derivatives have a wide application as dyes, electroluminescent materials, organic semiconductors, cavitands, chemically controllable switches, and DNA cleaving agents [6-11]. The conjugated polymers have attracted significant attraction due to their vast applications in numerous fields of research viz., photodiodes[12], photovoltaic cells [13], nonlinear optics[14], and laser devices [15]. The emergence of conjugated polymers as a new class of electronic materials has attracted many researchers for their tremendous potential applications. Conjugated polymers are semi-conductors viz., poly(phenylene vinylene)s (PPVs), polyfluorenes(PFs), polythiophene (PT) have been synthesized and utilized as emissive layers in light-emitting diodes (LED) [16]. p-type (electron donor, hole transport) and n-type (electron acceptor, electron transport) based polymeric semiconductors are essentially needed for creating more efficient and high performance electronic and opto-electronic devices[17]. Among the types of polymeric semi-conductors, n-type semi-conductors are feasible with nitrogen heterocyclic containing polymers jointly considered as "heterocyclic polymers".

In general, the conjugated polymers, the barrier of electron injection is much higher than that of hole injection. To improve the efficiency of these, it is necessary to balance the rate of injection of electrons and holes from opposite electrodes into the device. Hence, the high electron affinity substituents such as quinoxaline [18-20], oxadiazole[21], triazole[22], and quinoline[23] have been introduced into conjugated polymers. It has been well known that inclusion of heterocyclic compound in the back of conjugated polymers found to have good semi-conducting properties with outstanding thermal and oxidative stability, low moisture absorbing capabilities and excellent film forming capacities. Although, a variety of N-heterocyclic compounds containing PPV viz, oxadiazole, pyridine, quinoline, substituted quinoline, have been published[24-25].

To our knowledge, there is not much report apart from our group on substituted quinoxaline containing PPV. Further, it would be an interesting to investigate the effect of substitutent on the optical, electronic properties of conjugated polymer. In this paper, our interest towards the synthesis, photophysical and electrochemical aspects of a new conjugated vinyl benzaldehyde capped with quinoxaline derivative.

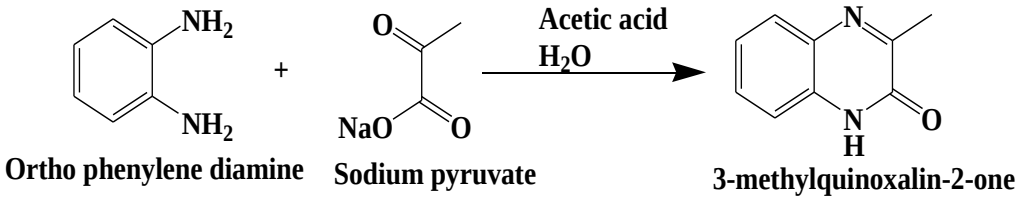
II. EXPERIMENTAL

2.1. Materials

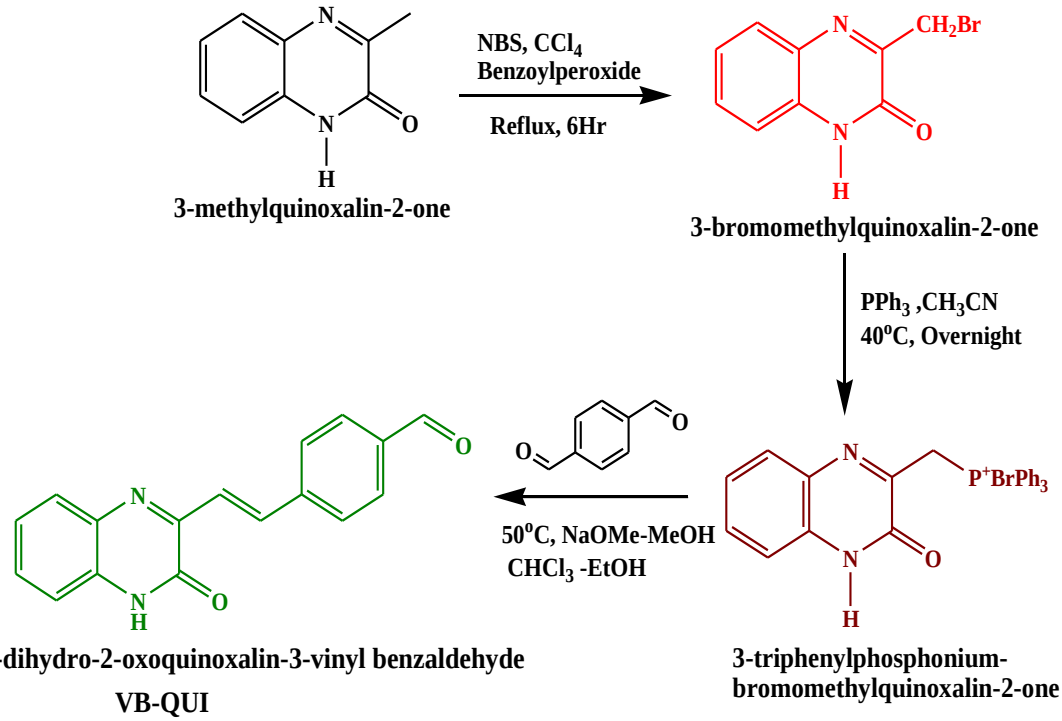
All the chemicals were obtained from Avra chemicals, Hyderabad, India and were used as supplied. Solvents used were purified and dried according to the standard procedure.

2.2. Characterisation Methods

The UV-Visible spectra were recorded on an Alpha-Bruker UV spectrophotometer equipped in the range between 200-800nm. Room temperature FTIR spectra were recorded as KBr pellet with an Alpha-Bruker FTIR spectrophotometer in the range of 4000-400cm^{-1}. Nuclear magnetic resonance spectra with different core viz., ^{1}H NMR, ^{13}C NMR and ^{31}P NMR were recorded in either DMSO-d_6 or $CDCl_3$ on Bruker ADVANCE III 500mHz spectrometer. The fluorescence spectra of the synthesised compound in ethanol were recorded on fluorescence spectrophotometer, FP-8500, JASCO. Mass spectroscopy was recorded on ES-FIGIEAN ionization mass spectrometer.



Scheme 1: Synthesis of 3-methylquinoxalin-2-one



Scheme 2. Synthesis of vinyl benzaldehyde capped quinoxaline derivative

2.3. Synthesis of 3-methylquinoxalin-2-one

To the solution of o-phenylenediamine (3.996g, 37.0mmol) in acetic acid (150ml) and water (200ml), which was heated upto 80°C, to this sodium pyruvate (4.07g, 37.0mmol) was added with intensive stirring. On the next day, the solvents was evaporated under reduced pressure and the crude product was purified by chromatography using silica gel. Hexane-Ethyl acetate (9:1) mixtures were used as eluent (yellow solid, yield 72%) m.p. 235-237°C **;** (Fig. 1) UV(λmax, nm): 200-250(π–π*), 300-350(n-π*)**;** (S. Fig. 1) FTIR (KBr, cm^{-1}): 3419.79(N-H, st), 2914.44(C-H, st), 1664.57(C=O, st), 1436.04(C=N, st) 1381.03(C-N), 754.17(C-H, b); (S. Fig. 2) ^{1}H NMR(CDCl3, 300MHz), d(ppm): 7.80(2H,dd), 7.31(2H,m), 2.63(3H,s), 7.9(1H,s); (S. Fig. 3) Mass(m/z): Calculated M.W 159.2, Observed M.W 160.1(M^{+}).

2.4. Synthesis of 3-bromomethylquinoxalin-2-one

3-methylquinoxalin-2-one (1.6g, 0.01mol) and NBS (1.80g, 0.01mol) were refluxed overnight in 30ml ccl_4 cointaining 0.08g (0.0003 mol) benzoyl peroxide. The byproduct NBS was removed by filteration. The reaction medium was washed with ccl_4 and the solvent evaporated. (Red crystals, Yield: 71%) m.p. 118-120 ° C (S. Fig. 4) FT-IR (KBr, cm^{-1}): 2924.09, 3053.32 (C-H, st), 3439.08 (N-H, st) 1674.21 (C=O, st) 1475.54 (C=N, st) 1379.10 (C-N, st) 763.81 (C-Br, st) (S. Fig. 5) ^{1}H-NMR (DMSO, ppm): 4.6 δ(2H, s) 7.8 δ (1H, d) 7.4 δ (2H, m) 7.2 δ (1H, m) 8.7 δ(1H,s); (Fig. 2)

2.5. 3-triphenylphosphonium-bromomethylquinoxalin-2-one

3-bromomethyl-quinoxalin-2-one (0.24g, 1mmol) and triphenylphosphine (0.26g, 1mmol) was dissolved together in acetonitrile(20ml). The solution was stirred overnight at 40°C. The resulting precipitate was recrystallized from toluene-methanol mixture (2:1) to yield brown sticky phosphonium ylide compound. (S. Fig. 6) FT-IR (KBr, cm-1): 2924.09, 3057.17 (C-H, st), 3446.79 (N-H, st), 1710.86 (C=O, st), 1436.97 (C=N, st), 1311.59 (C-N, st**),** 858.32 (C-Br, st), 721.38 (C-P, st), 540.07 (P-Br, st) (S. Fig. 7) ^{1}H-NMR (DMSO, ppm): 2.540δ (2H, s), 7.390δ (6H, m), 7.459δ (6H, m), 7.493δ(3H, m), 7.591 δ (2H, m), 7.619 δ (2H, m) (Fig. 2) ^{31}P-NMR (DMSO, ppm): 25.696δ (1P, s)

2.6. Synthesis of vinyl benzaldehyde capped quinoxaline derivative.

The vinyl benzaldehyde capped quinoxaline derivative were prepared from the corresponding phosphonium salt using the well-known Wittig reaction[16-17]. The phosphonium salt (0.50g, 1mmol) and terephthaldicarboxaldehyde (0.135g, 1mmol) were dissolved in a mixture of absolute ethanol and dry chloroform (12ml, 3+1 v/v) under N_2 atmosphere. Then, a predetermined amount of sodium methoxide (25wt% in methanol, 1.3ml, 5.6mmol) was added and the resulting solution was stirred at 50° C overnight. Precipitation in methanol gave precipitate, which was reprecipitated from dichloromethane -methanol. Formed compound was purified by dissolving in acetonitrile and chloroform . (yield; 80%, m.p. 110-112 ° C) (S. Fig. 8) FT-IR (KBr, cm-1): 2918(C-H, st), 3392 (N-H, st), 1689 (C=O, st), 1588 (C=N, st), 1347 (C-N, st**),** 1429 (C=C, st) **(S.** Fig. 9) ^{1}H-NMR ($CDCl_3$, ppm): 10.081δ (1H, s) 9.902δ (1H, s) 5.559δ (1H, d) 5.055 δ(1H, d) 7.951δ(1H, m) 7.665δ(1H, m) 7.555 δ(1H,m) 7.468 δ(1H, m) 7.424 δ(2H, m) 7.174δ(2H, m) (S. Fig. 10) ^{13}C-NMR ($CDCl_3$, ppm): 126.74-127.97 δ (vinyl carbons), 191.00 δ(C=O), 129.00-150.98 δ(Aromatic ring carbons) (Fig. 4) Mass(m/z): Calculated M.W 276.29, Observed M.W 277.24(M+1) (S. Fig. 11) UV (nm) : 200-252nm (π-π*) (Fig. 4) PL : 470nm Emission.

3. Results and discussion

3.1. Synthesis and Characterisation

The quinoxaline derivatives have been prepared by Condensation of o-phenylene diamine with 1,2-dicarbonyl compound in acetic acid at 80°C resulted into 3-methylquinoxalin-2-one. The structure of the synthesised compound was confirmed by FTIR, ^{1}H NMR and MASS spectral techniques. Although water is a desirable solvent for chemical reactions for many reasons like, cost, safety and environmental impacts, use of water in our reactions have generated moderate yields only. (~25% after 24 hours). However, the reaction holds goods with acidic medium. (Scheme 1)

The vinyl benzaldehyde capped quinoxaline derivatives have been prepared similar to our earlier report[17,24,25] The scheme of the entire reaction have been listed in scheme 2. The reaction was carried out in four stages.

Stage 1 – Condensation reaction between diamino and diketone compound

Stage 2 – Bromination of 3-methyl-quinoxalin-2-one with NBS in CCl_4

Stage 3 – Formation of phosphonium ylide compound

Stage 4 – VB-QUI, the target compound obtained by the reaction between phosphonium ylide with terephthaladehyde.

Formation of various stages of compounds were confirmed using spectral studies viz., UV, FTIR, ^{1}H, ^{13}C and ^{31}P NMR, MASS spectroscopy.

Fig. 1 summarised the UV spectral details of 3-methylquinoxalin-2-one. From the Figure 1 the n-π* and π-π* transitions were observed at 300-350nm and 200-250nm respectively., which implies the C=N, C=O and C=C in the 3-methylquinoxalin-2-one moiety. FTIR spectrum of 3-methylquinoxalin-2-one has shown in supplementary Fig 1. The week peak at 3008.95, 2914.44, 2848.86cm^{-1}were attributed to aromatic and aliphatic C-H stretching frequency. The peak at 3419.79 corresponds to N-H stretching frequency. The peak at 1570.06 cm^{-1} implies the C=C in the phenyl ring. The peak at 1664.57$cm^{-1}$for C=O stretching frequency. The bending frequency at 1381.03cm^{-1}, 754.17cm^{-1} reveals that C-N, C-H frequency respectively. The ^{1}H NMR spectrum of 3-methylquinoxalin-2-one have displayed in Supplementary Fig. 2 The doublet of doublet at 7.261-7.364 ppm, 7.489-7.825 ppm for aromatic protons and 2.631 ppm signal for aliphatic protons. Supplementary Fig. 3 shows

the MASS spectrum of 3-methylquinoxalin-2-one. From the spectra molecular ion peak observed at 159.2 this value agreed well with the theoretical value.

The stage 2 bromo methylated quinoxaline derivative was confirmed by FTIR, ^{1}H NMR spectroscopy. Supplementary Fig. 4 shows the FTIR spectrum of 3-bromomethyl-quinoxalin-2-one. It displays transmittance peak at 763.81 cm^{-1} for functional group C-Br stretching and 2858.51- 3157.47 cm^{-1}stretching frequency for phenyl nucleus. The peak at 1674.21cm^{-1} for C=O stretching frequency. Supplementary Fig. 5 depicts the ^{1}H NMR spectrum of 3-bromomethyl-quinoxalin-2-one. According to this spectrum, the bromomethylated proton signal observed at downfield region at 4.6 ppm may be due to electronegative bromo group attached in the methyl proton and signal at 8.7 ppm for N-H proton and signal appeared at 7.2-7.8 ppm for aromatic protons.

The stage 3 Phosphonium ylide was obtained from bromomethylated quinoxaline. Supplementary Fig. 6 summarised the FTIR spectrum of phosphonium salt. FTIR information for the formation of the phosphonium salt were confirmed by the appearance of P-Br, C-P stretching frequency[17] at 534.28cm^{-1}, 707.88 cm^{-1}. Supplementary Fig. 7 depicts the ^{1}H NMR spectrum of phosphonium salt it displays the aromatic signals around 7.260-7.639 ppm and methylene proton signal at 2.515-2.602 ppm. Fig. 2 shows the ^{31}P NMR spectrum of phosphonium compound. They displayed singlet signal at 25.698 ppm due to single phosphorous appeared in the compound.

The stage 4 vinyl benzaldehyde containing quinoxaline derivative has been obtained from phosphonium salt. Supplementary Fig. 8 summarised FTIR spectrum of QUI-PPV. It displays week transmittance peak at 2918 $cm^{-1}$for C-H stretching frequency. The peak at 3392cm^{-1} due to N-H stretching and peak at 1588cm^{-1}, 1689 cm^{-1}, 1429cm^{-1} for C=N, C=O, C=C stretching frequency. The C-N stretching frequency appeared at 1347cm^{-1} The^{1}H NMR spectrum of VB-QUI compound have shown in Supplementary Fig. 9. They displayed multiplets at 7.167-8.199 ppm attributable to aromatic protons, the signal at 10.02ppm for N-H proton and 3.946, 5.559 ppm due to olefinic protons and 1.22 ppm attributable to aliphatic protons. Supplementary Fig. 10 show the ^{13}C NMR spectrum of VB-QUI compound. They displayed signal at 191.00 ppm for carbonyl carbon and signal at 126.74 – 127.97 ppm due to vinyl carbons, further the signals appeared at 129.00-150.98 ppm for aromatic ring carbons.. Supplementary Fig. 11. summarised the UV spectrum of VB-QUI compound. From the Figure $\lambda_{max}$was observed at 200-252 nm which implies π-π* transition. The molecular weight was found from the GC-MASS spectrum shown in Fig 3. The molecular ion peak was observed at 277.24 found to be agreed well with the theoretical value.

3.2. Photoluminescent properties

Supplementary Fig. 10 displays the uv-vis absorption of the VB-QUI compound in the ethanol solution. The uv-vis absorption spectra of the solution exhibited the band around 200-250nm may be due to the π-π* electronic transition associated with the π-conjugation in the compound. Fig. 4 displays PL spectra of VB-QUI compound in the ethanol solution. In the PL spectra the compound showed a strong green emission approximately 470nm.

3.3. Anti-bacterial activity

The anti-bacterial activity of the synthesised vinyl benzaldehyde substituted quinoxaline was evaluated using two-Gram positive (Bacillus substilis and Staphylococccus aureus) and two-Gram negative (Escherichia coli and Pseudomonas auroginosa) bacteria. Ampicillin is used as positive control. The MIC values of the compound were determined by broth dilution method[26]. Among the tested micro-organism the VB-QUI compound exhibited the best antibacterial activity, with a MIC value of 0.12mg except Gram-negative Pseudomonas auroginosa bacteria.

III. CONCLUSIONS

The vinyl benzaldehyde capped quinoxaline derivative compound was synthesised through Wittig reaction using Phosphonium salt and terephthaldehyde. The resulting compound was characterised by UV, FTIR, ^{1}H, ^{13}C, ^{31}P NMR and GC-MASS spectral studies. The vinyl benzaldehyde introduced into the quinoxaline derivatives in the conjugation unit showed strong green emission at 470nm in the Photoluminescene spectra. Anti-bacterial activities of the synthesised compound were studied using Gram positive and Gram negative bacteria. In comparison with positive control ampicillin, the compound show relatively good anti-bacterial activity against tested micro-organism except Gram-negative Pseudomonas auroginosa bacteria.

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Table 1: Antibacterial study of the vinyl benzaldehyde capped quinoxaline derivative compound

Table 2. MIC (Minimum inhibitory concentration) values of VB-QUI compound

Compound	Bacillus substilis	Staphylococccus aureus	Escherichia Coli	Pseudomonas auroginosa
PVB-QUI	0.12mg	0.12mg	0.125mg	**0.25mg**

Concentrations of synthesised compound (PVB-QUI)	Bacillus substilis			Staphylococccus aureus			Escherichia Coli		Pseudomonas auroginosa	
	OD Values		% of inhibition	OD Values		% of inhibition	OD Values	% of inhibition	OD Values	% of inhibition
2mg	0.467	66.54728	0.347	73.59209	0.477	60.31614	0.621			52.73973
1mg	0.583	58.23782	0.5	61.94825	0.677	43.677205	1.111			15.44901
0.5mg	0.686	50.8596	0.591	55.02283	0.69	42.595674	1.253			4.642314
0.25mg	0.798	42.83668	0.691	47.41248	0.739	38.519135	1.204			**8.371385**
0.125mg	0.855	**38.75358**	0.727	**44.67275**	0.805	**33.028286**	1.303			0.837139
Control	1.396	0	1.238	0	1.202	0	1.314			0

Stage I : 3-methylquinoxalin-2-one

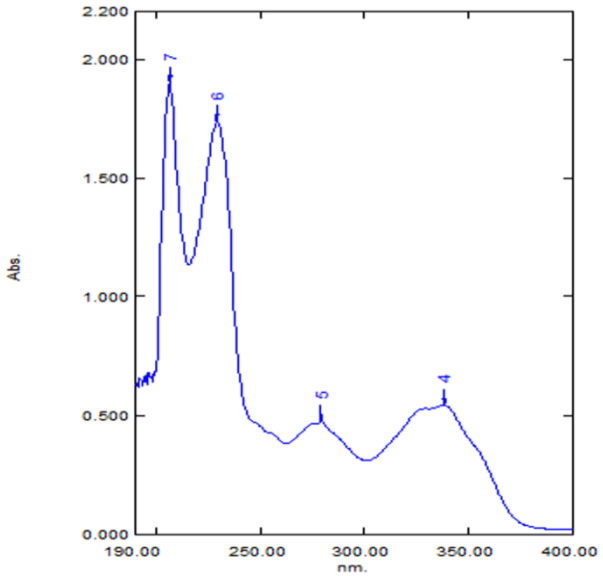
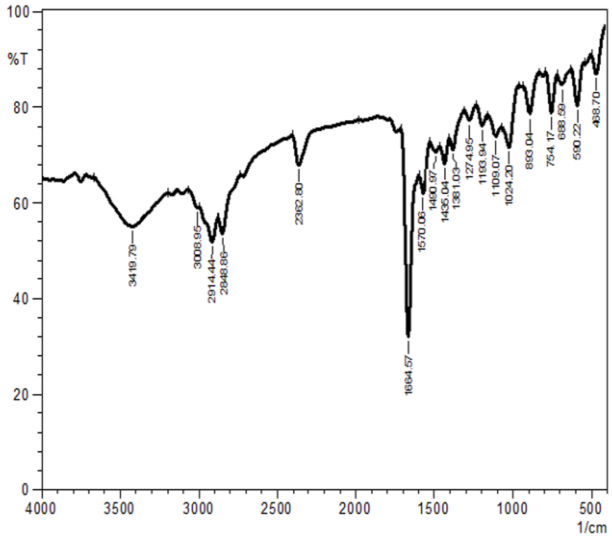
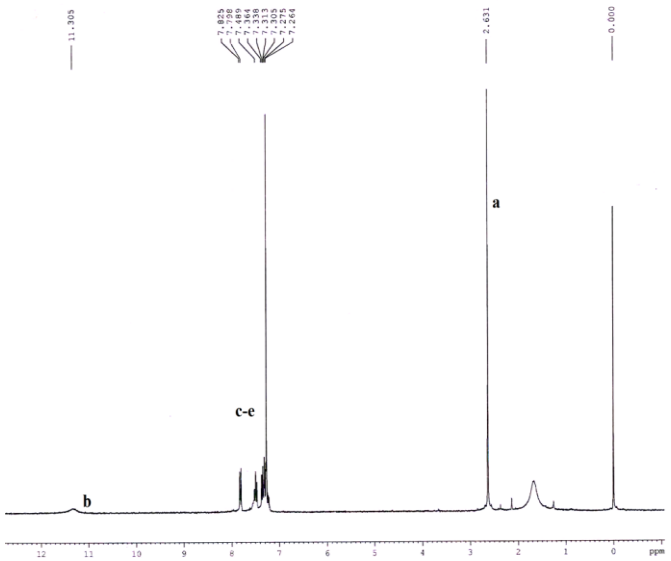


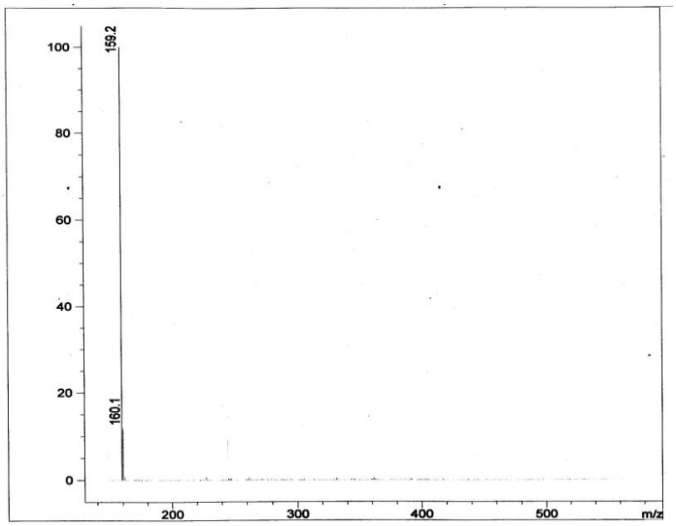
Figure 1: UV spectrum of 3-methylquinoxalin-2-one



Supplementary Figure 1: FTIR spectrum of 3-methylquinoxalin-2-one

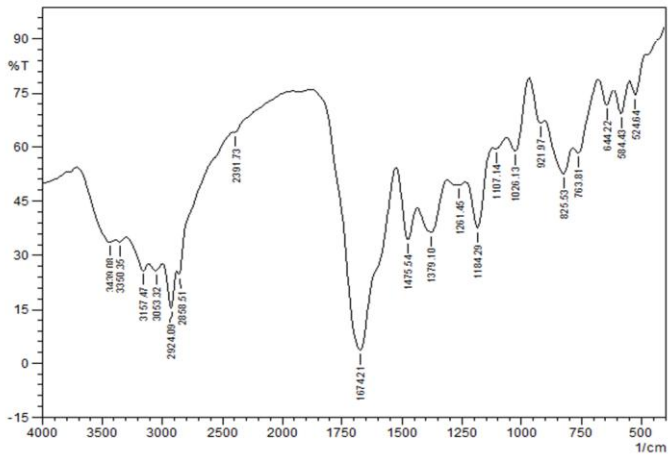


Supplementary Figure 2: ^{1}H NMR spectrum of 3-methylquinoxalin-2-one

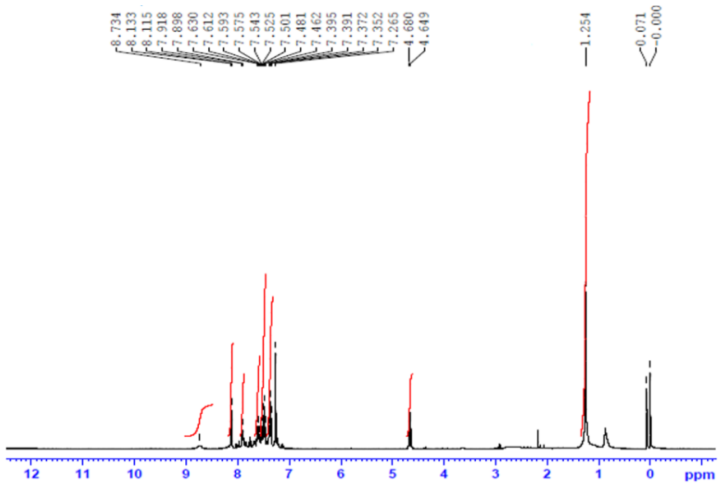


Supplementary Figure 3: MASS spectrum of 3-methylquinoxalin-2-one

Stage II: 3-bromomethyl-quinoxalin-2-one

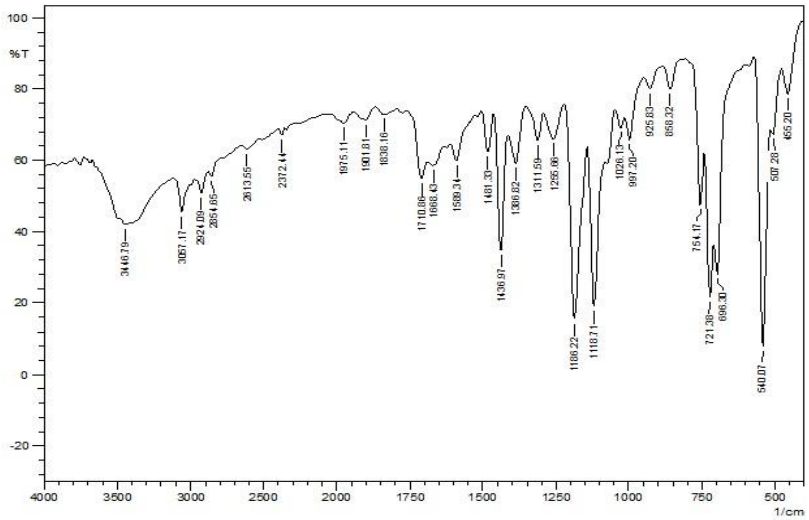


Supplementary Figure 4 : FTIR spectrum of 3-bromomethyl-quinoxalin-2-one

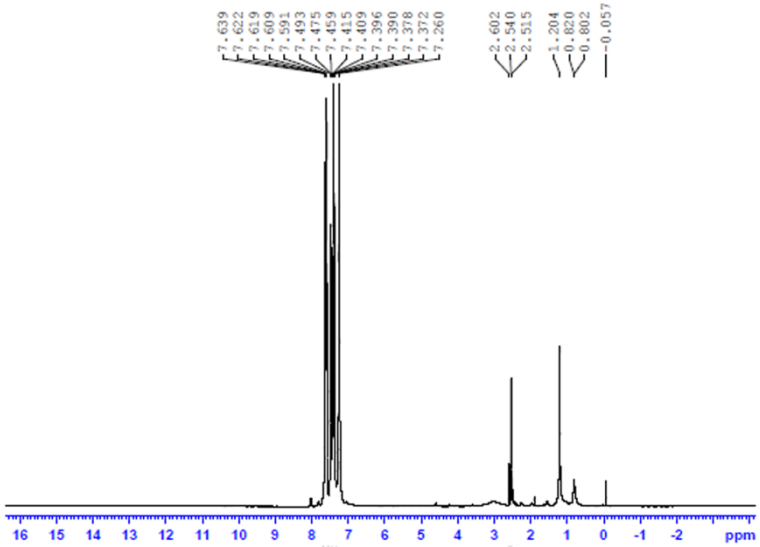


Supplementary Figure 5 : ^{1}H NMR spectrum of 3-bromomethyl-quinoxalin-2-one

Stage III: 3-triphenylphosphonium-bromomethylquinoxalin-2-one



Supplementary Figure 6**:** FTIR spectrum of 3-triphenylphosphonium-bromomethylquinoxalin-2-one



Supplementary Figure 7: ^{1}H NMR spectrum of 3-triphenylphosphonium-bromomethylquinoxalin-2-one

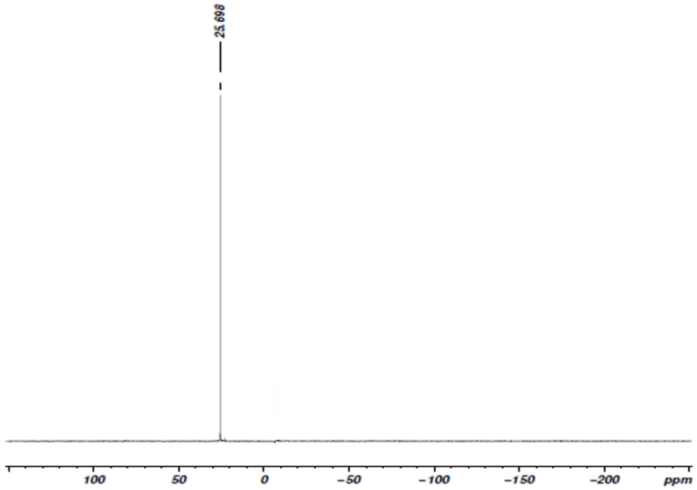
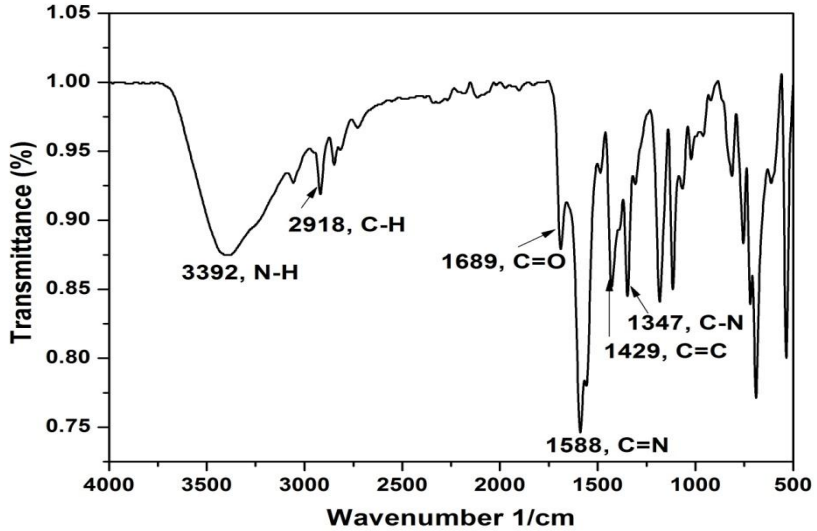
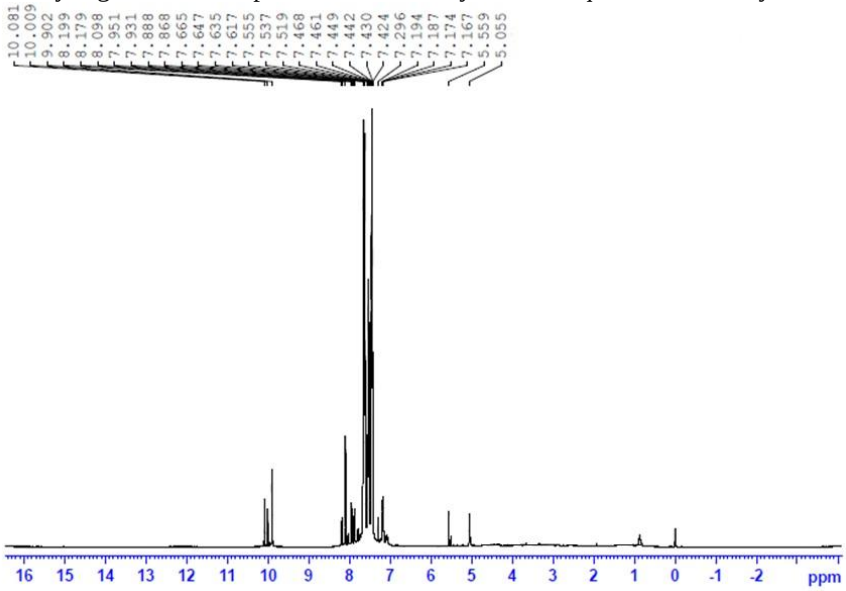


Figure 2: ^{31}P NMR spectrum of 3-triphenylphosphonium-bromomethylquinoxalin-2-one

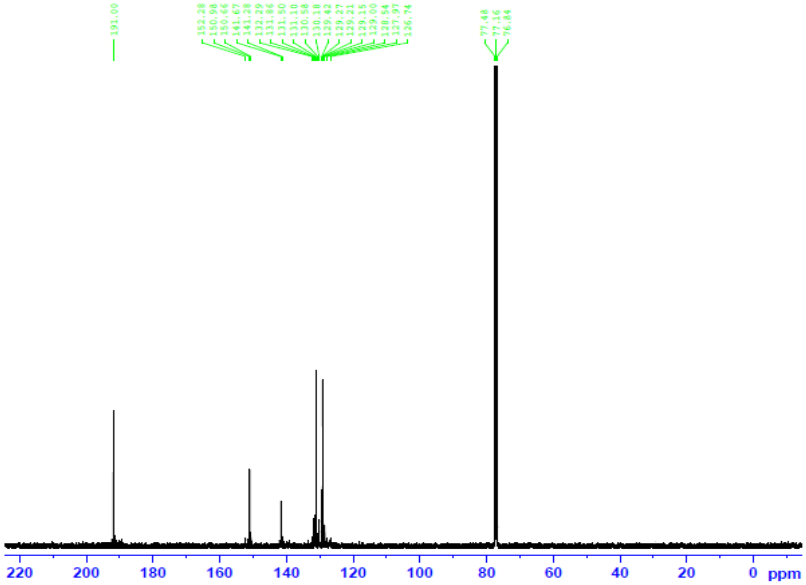
Stage IV: 1,2-dihydro-2-oxoquinoxalin-3-vinyl benzaldehyde



Supplementary Figure 8: FTIR spectrum of 1,2-dihydro-2-oxoquinoxalin-3-vinyl benzaldehyde



Supplementary Figure 9: ^{1}H NMR spectrum of 1,2-dihydro-2-oxoquinoxalin-3-vinyl benzaldehyde.



Supplementary Figure 10: ^{13}C NMR spectrum of 1,2-dihydro-2-oxoquinoxalin-3-vinyl benzaldehyde.

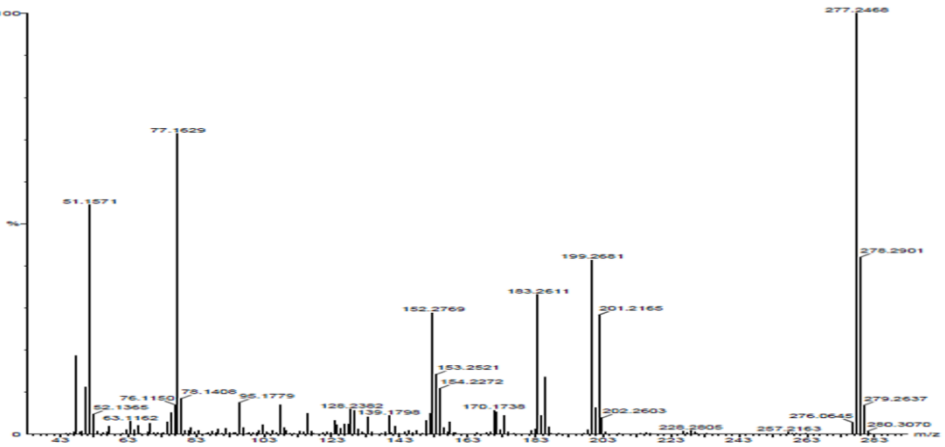
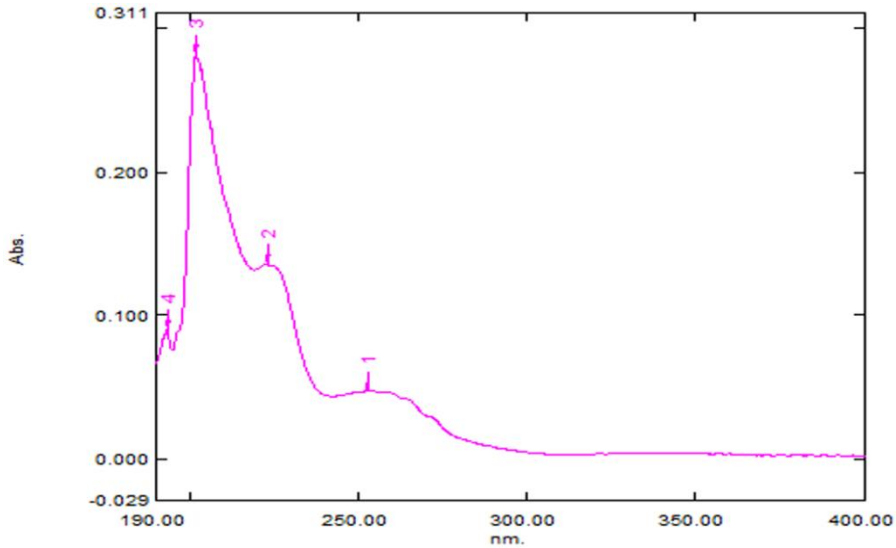


Figure 3: GC MASS spectrum of 1,2-dihydro-2-oxoquinoxalin-3-vinyl benzaldehyde



Supplementary Figure 11: UV spectrum of 1,2-dihydro-2-oxoquinoxalin-3-vinyl benzaldehyde.

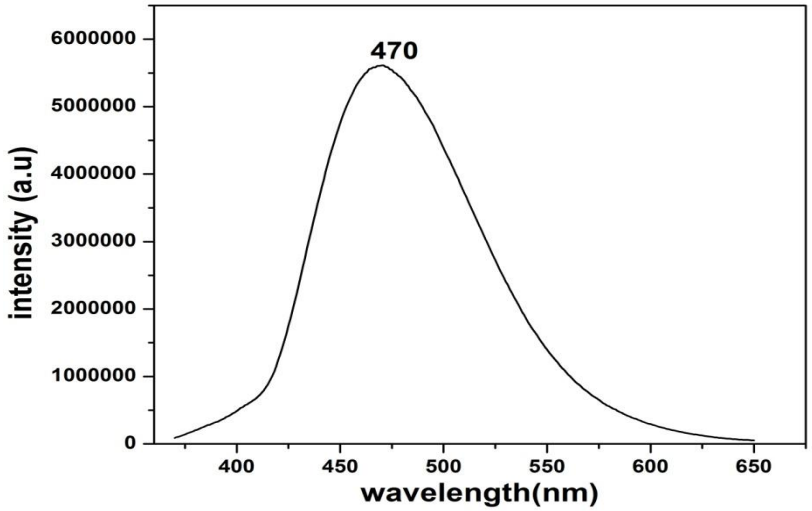


Figure 4: PL spectrum of 1,2-dihydro-2-oxoquinoxalin-3-vinyl benzaldehyde.