Synthesis, Characterization and Biological Evaluation of some new Indolylglyoxamides and Tryptamines

Dinesh verma, Meenakshi jain

Abstract— Synthesis of 1-(N-allyl)-2-(substituted-aryl)-indol-3-yl morpholinothanes (7a−i) has been successfully accomplished in high yield. 1-N-(allyl-2-(4-bromophenly)indol-3-yl)morpholino glyoxamides on Treatment with a suspension of lithium aluminium hydride in dry ether gave desired Title products. All the synthesized compounds have been characterized by elemental analysis and spectral data. They have also been evaluated for their antimicrobial activities.

Index Terms— Morpholinoethanamines, Indolylglyoxamides.

I. INTRODUCTION
Indole are one of the most important nitrogen containing heterocyclic molecules, found extensively in biological systems which play vital role in biochemical processes. Indole ring system is found in many natural products, pharmaceutical agents and polymer materials. The indole and its derivatives have great importance in clinical chemistry. The 2-aryl indole moiety is present in diverse biologically active molecules displaying antiestrogenic1, anti-inflammatory2,3 and cytotoxic properties4 Indole derivatives have also been found to form in human large intestines by anaerobic bacteria, where indole pyruvate is either converted into indoleacetate or metabolized into indole5. Most of the drugs, used in the treatment of central nervous system disorders. Indole derivatives are of particular importance because of their diverse bioactivities such as antidepressive, anti-inflammatory, hallucinogenic, antihistaminic and hypcholesterolemic activities.5-chloro-2-methylindole and 6-chloro-2-methylindole depress the brain stem and telencephalon-5-hydroxytryptamine, 5-methoxytryptamine is an important brain amine and shows central potentiating action. Tryptophan is an essential amino acid and as such is a constituent in most proteins. In animals, it also serves as a precursor for two chemically closely related hormones. Serotonin is a powerful vasoconstrictor and also a neurotransmitter substance and melatonin is believed to play an important role in controlling day and night rhythms.

Indole-3-acetic acid is a plant growth regulating hormone also derived from tryptophan.

Indole-3-yl-glyoxamides derivatives have been listed for various pharmacological activities such as antitumor8,14, antiinflammatory, anti-HIV10, antiviral11 antimicrobial8, anti hypertensive12, anti-diabetic15, cardiovascular13.

So, Keeping these observation and in connection With Our Other Work On bioactive indole derivatives, we have synthesized a number of new 1-(N-allyl-2-arylindol-3-yl)-ethanamines (7a−i).

II. EXPERIMENTAL
Characterization of Synthesized compounds has been done on the basis of elemental analyses, IR, 1H NMR and mass spectral studies. C and H analyses of compounds has been done using Coleman C and H analyzer. Nitrogen analyses has been done using Coleman N-analyzer 29,. Melting points were determined in open glass capillaries and are uncorrected. IR spectra were recorded on a Perkin-Elmer Model-557 and Nicolet-Magna Model-750 spectrophotometer in KBr pallets.1H NMR spectra were recorded on Bruker DRX 300 NMR spectrophotometer (300 MHz) using DMSO d6 as a solvent. TMS was used as internal standard. The chemical shifts are in δ ppm. Mass spectra were recorded on Jeol SX-102 (FAB) spectrometer.

In all reactions and preparations of starting materials, reagents were purified by either distillation or recrystallization. The purity of compounds were checked by TLC using silica gel-G as adsorbent in various solvent systems. Visualization was accomplished by UV light or iodine adsorption. The middle fractions boiling within ± 0.5°C of the standard boiling points were collected in each case.
III. RESULT AND DISCUSSION
Substituted 2-phenyl indoles (3a-e) were prepared by Fischer indole synthesis\textsuperscript{16} and by the method of Joshi \textit{et al}\textsuperscript{17}. 2-arylindoles undergo phase transfer catalysis using tetrabutyl ammonium hydrogen sulphate (TBAHSO\textsubscript{4}) as PT catalyst\textsuperscript{18} to give N-allyl-2-arylindoles (4a-e). N-allyl-2 arylindoles (4a-e) on treatment with oxalylchloride\textsuperscript{19}, in dry ether at 0°C, gave 3-indolylglyoxalyl chlorides (5a-e). The synthetic steps are illustrated in following (Scheme 1).

![Scheme 1](image)

IV. 1-(N-ALLYL-2-(SUBSTITUTEDARYL)-INDOL-3-YL)MORPHOLINOGLYXAMIDES (6 A-I)
1-(N-allyl-2- (substitutedaryl)-indol-3 yl) glyoxalylchloride 5mmol, 2.0 g) was dissolved in dry ether (50 ml) and was added drop wise to the ethereal solution of morpholine (10 mmol, 0.87g in 25 ml ether) with constant shaking.\textsuperscript{20} The stirring was continued for 30 minutes. The insoluble solid mass was separated, washed with ether followed by water. Crystallization from ethanol yielded the pure crystals colored crystals of product which gave a single spot on TLC. The Analytical Data Obtained are presented in (Table -1)

V. 1-(N-ALLYL)-2-(SUBSTITUTEDARYL)-INDOL-3-YL MORPHOLINOETHANMINES (7 A-I)
To a suspension of lithium aluminum hydride (LiAlH\textsubscript{4}) (2.0 gm) in dry ether (100 ml), was added. Portion wise in a slurry of the 1-N (-allyl-2-( substitutedaryl) indol-3-yl) morpholinoglyxamides (1.5gm.) in a mix of dry ether (50ml) and dry benzene (20ml) the reaction was initially vigorous. It was heated under reflux for 6 hours, on a water bath decomposed by adding wet ether (50 ml) is the first instance and then poured into 2N NaOH(200 ml) . The ethereal layer was separated and water contents were washed with ether to extract any traces of the product. All the ether portions were combined and filtered. All The solvent was removed by evaporation over water bath. oily layer thus obtained become solid on keeping cooling from ethanol . The resultant residue was purified by recrystallization from ethanol. The Analytical Data Obtained are presented in (Table- 2)
Table 1: Physical and analytical data of 1-(N-allyl-2-aryl-indol-3-yl)-glyoxamides (6a-i)

| Compd No. | R    | X   | -N-R₂   | % Yield | M.P. (°C) | M.F           | C (%)  | H (%)  | N (%)  |  |  |  |
|-----------|------|-----|---------|---------|-----------|---------------|--------|--------|--------|  |  |  |
| 6a        | allyl| H   | Piperidino | 86      | 117       | C₂₉H₂₄N₂O₂    | 79.55  | 79.49  | 3.86   | 3.82 | 7.73 | 7.69 |
| 6b        | allyl| 4-Br| Piperidino | 82      | 150       | C₂₃H₂₂BrN₂O₂  | 63.71  | 63.61  | 5.3    | 5.23 | 6.19 | 6.10 |
| 6c        | allyl| 4-F | Piperidino | 80      | 168       | C₂₄H₂₃FN₂O₂   | 73.84  | 73.79  | 5.89   | 5.84 | 7.17 | 7.11 |
| 6d        | allyl| 4-Br| Morpholino| 81      | 126       | C₂₃H₂₁BrN₂O₃ | 60.92  | 60.89  | 4.63   | 4.61 | 6.18 | 6.13 |
| 6e        | allyl| 4-Cl| Morpholino| 80      | 140       | C₂₃H₂₁ClN₂O₃ | 67.56  | 67.51  | 5.14   | 5.12 | 6.85 | 6.80 |
| 6f        | allyl| 3-NO₂| Piperidino| 78      | 94        | C₂₄H₂₃N₃O₃   | 66.51  | 66.49  | 5.31   | 5.29 | 9.69 | 9.59 |
| 6g        | allyl| 3-NO₂| Morpholino| 75      | 82        | C₂₃H₂₁N₃O₃   | 65.87  | 65.81  | 5.01   | 5.00 | 10.2 | 10.1 |
| 6h        | allyl| 4-F | Morpholino| 79      | 142       | C₂₃H₂₁FN₂O₃  | 70.40  | 70.32  | 5.35   | 5.29 | 7.14 | 7.09 |
| 6i        | allyl| H   | Morpholino| 85      | 105       | C₂₃H₂₂N₂O₃   | 73.79  | 73.71  | 5.88   | 5.84 | 7.48 | 7.31 |
### Table-2: Physical and analytical data of 1-(N-allyl-2-aryl-indol-3-yl)-ethanamines (7a-i)

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<td>H</td>
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<td>Piperidino</td>
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<td>Morpholino</td>
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<td>142</td>
<td>C₂₃H₂₅F₂N₂O</td>
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<td>Morpholino</td>
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<td>105</td>
<td>C₂₃H₂₆N₂O</td>
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IR spectra of 2-arylindoles (3a-e) showed absorption bands from 3480-3420 cm\(^{-1}\), which is attributed to >N-H stretching vibrations. In the IR spectra of N-allyl-2-arylindoles (4a-e), >N-H absorption band disappeared which supports the N-allylation of indole moiety. IR spectra of 3-indolylglyoxalyl chlorides (5a-e) show C=O stretching bands between 1750-1710 cm\(^{-1}\) and (C-Cl) absorption band in the region of 760-740 cm\(^{-1}\). In the IR spectra of N-alkylated-3-indolylglyoxamides (6a-i) C=O bands occur in the region of 1650-1600 cm\(^{-1}\), the one at higher wave number may be attributed to the amide C=O and another at lower wave number to the keto group.

In \(^1\)H NMR spectra of 2-arylindoles (3a-e), methine proton at C-3 position of indole moiety shows a resonance signal at \(\delta\) 6.9 ppm and >N-H resonance signal is observed in the region of \(\delta\) 8.0-8.6 ppm as a broad singlet. Aromatic protons are observed as multiplet from \(\delta\) 7.0-7.9 ppm. In \(^1\)H NMR spectra of N-allylated-2-arylindoles (4a-e), >N-H resonance signal disappeared and two new signals appear for allyl group\(^{23}\), one doublet appears between \(\delta\) 3.41-3.47 ppm (\(-\text{CH}_2\text{-CH=}\) and another multiplet appears between \(\delta\) 4.50-5.98 ppm (\(-\text{CH=CH}_2\)). N-alkyl-3-indolylglyoxamides show the characteristic resonance signals of -morpholino and -piperidino groups. In -morpholino one triplet appears between \(\delta\) 3.05-3.10 ppm for CH\(_2\)-N-CH\(_2\) and another triplet appears between \(\delta\) 3.21-3.25 ppm for CH\(_2\)O-CH\(_2\). In -piperidino one triplet appears between \(\delta\) 2.25-3.15 ppm for CH\(_2\)-N-CH\(_2\) and another multiplet appears from \(\delta\)1.09-1.96 ppm for CH\(_2\)CH\(_2\)CH\(_2\) protons. \(^1\)H NMR and IR spectra of (7a-i) are summarized in Table(3). The IR (Fig. 2) and \(^1\)HNMR (Fig. 1) spectral data of 7d are Presented.
### Table-3 : Spectral data of 1-(N-allyl-2-aryl-indol-3-yl)-ethanamines (7a-i)

<table>
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<tr>
<th>Compd. No.</th>
<th>IR(KBr) $V_{\text{max}}$ cm$^{-1}$</th>
<th>$^{1}$H NMR(CDCl$_3$)/DMSO$_d$_6 $\delta$ ppm</th>
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<tr>
<td>7a</td>
<td>3079 (aromatic C-H str.), 2914 (aliphatic C-H str.), 1514(C=C str.),1379(C-N str),</td>
<td>3.05(t, CH$_2$-N-CH$_2$, 4H, J=3 Hz), 3.25(t, -CH$_2$O-CH$_2$, 4H, J=3 Hz), 3.45 (d, C-H$_2$-CH=CH$_2$, 2H, J=3 Hz), 4.50-5.84(m, CH$_2$-CH=CH$_2$, 3H), 7.12-7.69 (m,Ar-h, 9H)</td>
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<tr>
<td>7B</td>
<td>3050 (aromatic C-H str.), 2864 (aliphatic C-H str.) 1543 (C=C str.), 1350 (C-N str.), 550(C-Br)</td>
<td>3.08(t,-CH$_2$-N-CH$_2$, 4H,J=3Hz)3.23(t,-CH$_2$O-CH$_2$, 4H,J=3Hz)3.41(d,-CH$_2$-CH=CH$_2$,2H,J=3Hz) 4.515.85(m,-CH$_2$-CH=CH$_2$,3H),7.14-7.66(m,Ar-H,8H)</td>
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<td>7C</td>
<td>3064(aromatic C-H str.),2936(aliphatic C-H str.),1521(C=C str.),1371(C-N str.), 764 (C-Cl)</td>
<td>3.07(t,-CH$_2$-N-CH$_2$, 4H,J=3Hz) 3.27(t,-CH$_2$O-CH$_2$, 4H, J=3 Hz), 3.48 (d, -CH$_2$-CH=CH$_2$, 2H, J=3Hz),4.52-5.89(m,CH$_2$-CH=CH$_2$, 3H),7.10-7.64(m,Ar-h,8h)</td>
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<tr>
<td>7d</td>
<td>3079 (aromatic C-H str.), 2900 (aliphatic C-H str.), 1521 (C=C str.), 1371 (C-N str),</td>
<td>3.08 (t,-CH$_2$-N-CH$_2$, 4H,J=3Hz) 3.21 (t,-CH$_2$O-CH$_2$, 4H, J=3Hz), 3.43(d,-CH$_2$-CH=CH$_2$, 2H, J=3Hz) 4.54-5.58(m,-CH$_2$-CH=CH$_2$,3H), 7.13-7.66 (M,Ar-H,8H)</td>
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<td>7e</td>
<td>3057 (aromatic C-H str.), 2936 (aliphatic C-H str.), 1521(C=C str.), 1371 (C-N str.), 1329 (-NO$_2$ str),</td>
<td>3.07 (t,-CH$_2$-N-CH$_2$, 4H, J=3Hz), 3.26 (t,-CH$_2$O-CH$_2$,4H, J=3Hz), 3.41(d,-CH$_2$-CH=CH$_2$, 2H,J=3Hz)4.48-5.81(m,-CH$_2$-CH=CH$_2$, 3H), 7.16-7.69 (m, Ar-9H)</td>
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<td>7f</td>
<td>3057 (aromatic C-H str.),2914 (aliphatic C-H str.) 1514 (C=C str), 1364 (C-N str)</td>
<td>2.25 (t,-CH$_2$-N-CH$_2$, 4H, J=3Hz), 1.51-1.89 (m, CH$_2$O-CH$_2$, CH$_2$ 6H), 4.01(D,-CH$_2$-CH=CH$_2$, 2H,J=3Hz), 4.39-5.29 (m,-CH$_2$-CH=CH$_2$, 3H), 7.18-7.67 (m,Ar-H,9H)</td>
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<td>7g</td>
<td>3071 (aromatic C-H str.), 2957 (aliphatic C-H str), 1536 (C=C str.), 557 (C-Br)</td>
<td>3.13(t,-CH$_2$-N-CH$_3$, J=3Hz), 1.41-1.56 (m, CH$_2$-CH$_2$,6H),3.41(d,-CH$_2$-CH=CH$_2$, 2H,J=3Hz)4.50-5.88(m, CH$_2$-CH=CH$_2$,3H), 7.23-7.63 (m, Ar-H,8H)</td>
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<td>3057(aromatic C-H str.), 2879 (aliphatic C-H str), 1536 (C=C str), 1386 (C-N str),743(C-Cl)</td>
<td>2.24(t,-CH$_2$-N-CH$_2$, 4H,J=3Hz), 1.47-1.87 (m, CH$_2$-CH$_2$, 6H), 3.96 (d,-CH$_2$-CH=CH$_2$, 2H, J=3Hz), 4.37-5.25(m,-CH$_2$-CH=CH$_2$, 2H),7.15-7.71(mAr-H,8H)</td>
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<td>7i</td>
<td>3071 (aromatic C-H str.), 2929 (aliphatic C-H str.) 1536 (C=C str.), 1379 (C-N str.),</td>
<td>2.28 (t,-CH$_2$-N-CH$_2$, 4H, J=3Hz), 1.43-1.84 (m,CH$_2$-CH$_2$, 6H), 3.85(d,-CH$_2$-CH=CH$_2$, 2H, J=3Hz), 4.31-5.38(m,-CH$_2$-CH=CH$_2$, 3H), 7.18-7.67(m, Ar-H, 8H)</td>
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Figure : 1

Figure : 2
ANTIBACTERIAL AND ANTIFUNGAL ACTIVITIES OF 1-[(N-ALLYL-2-ARYL-INDOL-3-YL)GLYOXAMID]ES (6B & 6H) AND 1-[(N-ALLYL-2-ARYL-3-INDOL-3-YL)-ETHANAMINES (7A-i)

Representative title compounds were screened for their antibacterial activity against gram-positive bacteria Staphylococcus aureus and gram-negative bacteria Escherichia coli at 200, 400 and 800 ppm concentration. Antifungal activity was done against Aspergillus niger and Aspergillus flavus at 200, 400 and 800 ppm concentration. Streptomycin and mycostatin were used as standard drugs for antibacterial and antifungal evaluations, respectively. Compounds were screened for their antibacterial and antifungal efficacy following the inhibition zone technique. The results obtained for antibacterial and antifungal activities are tabulated in Table-4 and Table-5, respectively.

Table-4: Antibacterial activities of 1-(N-ally1-2-aryl-indol-3-y1)-glyoxamide (6b & 6h) and 1-(N-ally1-2-aryl-indol-3-y1)-ethanamines(7a-i)

<table>
<thead>
<tr>
<th>compound no.</th>
<th>Mean value of area of inhibition in mm (1000ppm)</th>
<th>Mean value of area of inhibition in mm (800ppm)</th>
<th>Mean value of area of inhibition in mm (400ppm)</th>
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<td>IZ(AI)</td>
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<td>S.aureus 11.4</td>
<td>E. Coi 10</td>
<td>S.aureus 9.5</td>
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<td></td>
<td>6b 9(.78)</td>
<td>8(0.8)</td>
<td>6.1(.64)</td>
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<td>6h 7.2(0.63)</td>
<td>9.5(.95)</td>
<td>7.2(075)</td>
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<td>7a 9(0.78)</td>
<td>7(0.7)</td>
<td>9(0.94)</td>
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<td>7b 10.8(0.94)</td>
<td>8.9(0.89)</td>
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<td>7c 8.4(0.73)</td>
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<td>7d 12.4(1.08)</td>
<td>10.5(1.05)</td>
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<td>7e 6.2(0.54)</td>
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<td>7f 8.5(0.74)</td>
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<td>7g 10(0.87)</td>
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<td>7h 7(0.61)</td>
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IZ = Inhibition area (zone) excluding diameter of disk
AI = (activity index) = inhibition area of sample / inhibition area of standard.

Table - 5 Antifungal activities of 1-(N-allyl-2-aryl-indol-3-yl)-glyoxamides (6b&6h) and 1-(N-allyl-2aryl-indol-3-yl)-ethanamines (7a-7i)

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<td>8.6(0.82)</td>
<td>6(0.75)</td>
</tr>
<tr>
<td>6h</td>
<td>10.7(0.97)</td>
<td>9.9(0.95)</td>
<td>7.3(0.91)</td>
</tr>
<tr>
<td>7a</td>
<td>9(0.81)</td>
<td>7.3(0.70)</td>
<td>7(0.87)</td>
</tr>
<tr>
<td>7b</td>
<td>10(0.90)</td>
<td>11(1.05)</td>
<td>7.7(0.96)</td>
</tr>
<tr>
<td>7c</td>
<td>12.2(1.10)</td>
<td>9(0.86)</td>
<td>9(1.22)</td>
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<tr>
<td>7d</td>
<td>11(1)</td>
<td>10(0.96)</td>
<td>7.5(0.93)</td>
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<tr>
<td>7e</td>
<td>6(0.59)</td>
<td>4.7(0.45)</td>
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<tr>
<td>7f</td>
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<td>8(0.77)</td>
<td>8(1)</td>
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<td>7i</td>
<td>10.5(0.95)</td>
<td>10.2(0.98)</td>
<td>7(0.87)</td>
</tr>
</tbody>
</table>

IZ = Inhibition area (zone) excluding diameter of disk
AI = (activity index) = inhibition area of sample / inhibition area of standard
REFERENCES