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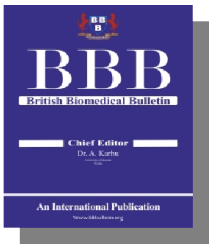

Original

Synthesis, Characterization and Biological Evaluation of 1,3,4 Oxadiazole Derivatives Containing Indole Moiety Bearing-Tetrazole

S. Muralikrishna*, P. Raveendrareddy, L. K. Ravindranath and P. Jagadeeswara Rao

Department of Chemistry, S. K. University, Anantapur, India

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Corresponding author: Department of Chemistry, S. K. University, Anantapur, India.

E-mail address: muralisphd@gmail.com

A B S T R A C T

Schiff base synthesis of 1,3,4 oxadiazole derivatives containing Indole moiety bearing thiazolidinone ring were synthesized by the condensation of 2-(3-(4-oxo-3-(p-tolyl) 1H-tetrazol-5-yl)-1H-indol-1-yl)-N-(1,1,1-trifluoropropan-2- ylidene) acetohydrazide with acetic anhydride. To this reaction was subjected in schiff base reaction. It forms 2-(1-((4-acetyl-5-methyl-5-(trifluoromthyl)-4, 5-dihyro-1, 3, 4-oxadiazol-2-yl) methyl)-1H-indol-3yl)-3-(p-tolyl) 1H-tetrazol-5-yl). The structure of these newly synthesized compounds was characterized by ^{1}H NMR, ^{13}CNMR, Mass, IR, and elemental analysis.

Introduction

Hetero cyclic compounds represent an important class of biological molecules. The hetero cyclic molecules which, posses indole, 1, 3, 4 oxadiazole and thiazolidinone moieties exhibit a wide range of biological activities. Indoles are one of the most important alkaloid molecules found extensively in biological systems, which play a vital role in many of the biochemical processes. Indole ring constitutes an important basic skeleton and development of the drug. The classical indole drugs are indomethacin and indoxole. Indole derivatives found to possess high which includes, antibacterial, analgesic, antipyretic, antifungal, inflammatory, anthelmintic, cardiovascular, anticonvulsant and selective COX-2 inhibitory activities.

Tetrazole and its derivatives have attracted much attention because of their unique structure and applications asantihypertensive, antialergic, antibiotic and anticonvulsant agents[1-7].

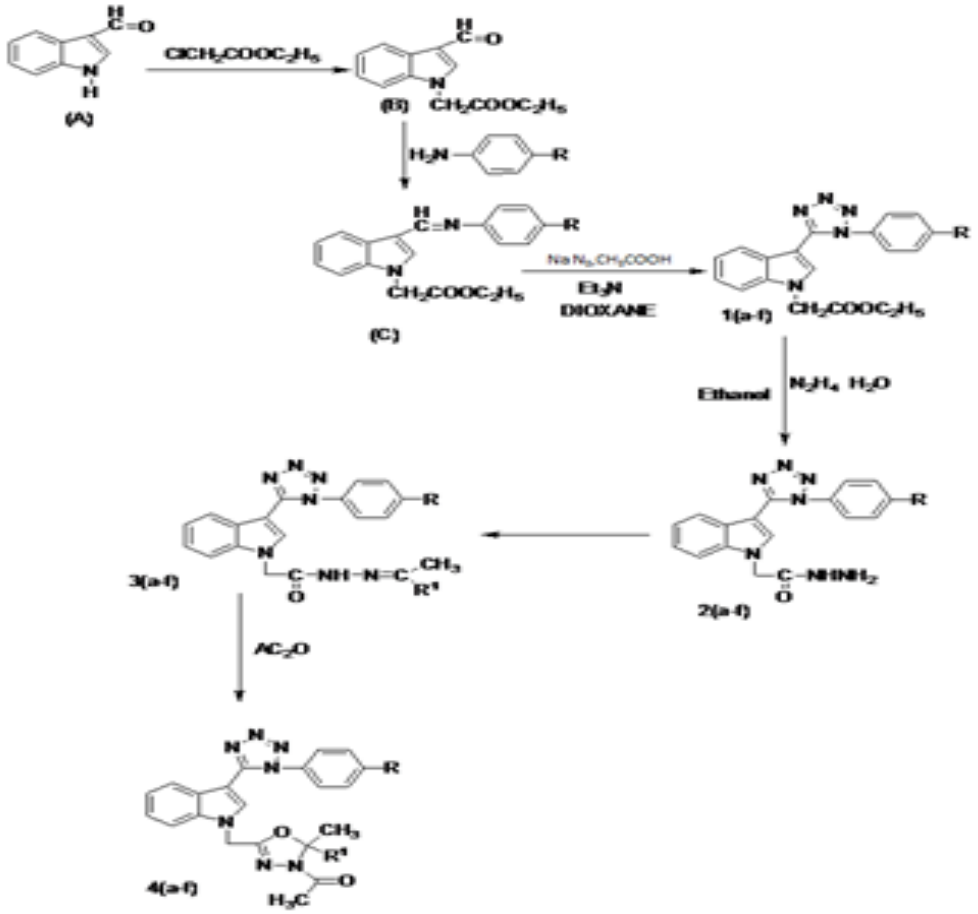
Among the five member heterocyclic compounds, 1, 3, 4-oxadizoles has become an important synthonfor the

development new therapeutic agents. Compounds with 1, 3, 4-oxadiazole core substantiates for broad spectrum of biological activities including antimicrobial, antifungal, antiinflammatory[8]., anti-convulsant[9]., antioxidant, analgesic[10]. and mutagenic activity[11]. Compounds containing quinoline moiety are most widely used as antimalarials[12]., antibacterials[13]., antifungals[14]., anticancer agents[15]. And potential HIV-1 integrase inhibitors[16-17].

Results and Discussion

Synthesis of 2-(3-formyl-1H-indol-1-yl) acetate (B)

An equimolar mixture of indole-3-carbaldehyde (A) and chloro ethyl acetate were dissolved in dimethyl formamide solvent and to this reaction mixture anhydrous K_2CO_3 was added and the reaction mixture was stirred at room temperature (35^0C) for 8 hours and the progress of the reaction was monitored by TLC using cyclohexane and ethyl acetate solvent mixture (7:3) as eluent the reaction mixture was kept overnight. After completion of the reaction the solvent was evaporated on Rota-evaporator. The gummy solid was separated and it was recrystalised from -2-propanol-petroleum ether (80^0C) solvent mixture. The crystalline solid was found to be -2-(3-formyl-1H-indol-1-yl) acetate. With a yield of 75% and mp 143-145^0C.The indole-3-carbaldehyde used in the present studies was purchased from Aldrich company and was used without any further purification. Yield 75%, m.p.: 143-145^0C.



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compound	4(a)	4(b)	4(c)	4(d)	4(e)	4(f)
R	H	CH_3	OCH_3	Cl	NO_2	CF_3
R^1	CF_3	CF_3	CF_3	CF_3	CF_3	H

The IR(KBr) spectrum of 2-(3-formyl-1H-indol-1-yl) acetate was recorded in the range 4000-667cm^{-1} and the absorption signals were found at 3032(√-Ar-H), 2980 and 2960 (√ aliphatic CH_2 andCH_3), 1760 (√ CO of ester group), and 1182(√ C-O-C of ester group).

^{1}HNMR Spectra (δ_{PPm})

The ^{1}HNMR spectra of 2-(3- formyl-1H-indol-1-yl) acetate was recorded in DMSO-d6 solvent. The NMR signal of 2-(3-formyl-1H-indol-1-yl) acetate was found at $\delta_{PPm,}$ 1.29 (t,3H, J=13.2Hz, CH_3 of ethyl group), 4.13 (q, 2H, J=13.2Hz, CH_2 of ethyl group), 4.78(s, 2H, N-CH_2 group) and6.92 , 7.58 (m, 10H, C_8H_5N indole nucleus).

Synthesis of Ethyl 2-(3-phenyl imino) methyl-1H-Indole-1-yl-acetate (C)

Equimolar quantity of ani (ine(3) and ethyl-2-(-3-formyl-1H-indol-) -yl)ace (ate(B) were dissolved in absolute alcohol, to this three dropsacetic acidacid is added then heated on a steam bath for 5-6hrs at 100^{0}C. After standing for 24hrs at room temperature, the product was dried and recrystalised from warm absolute alcohol. The separated solid was identified as ethyl 2-(-3-(((-4-nitro phenyl) imino) me thyl)-1H-indol-1-yl)acetate. Yield 75%, m.p.: 154-156^{0}C

IR Spectra (√, cm^{-1})

IR (KBr) spectrum of ethyl 2-(3-phenyl imino)metbyl-1H-Indole-1-yl-acetate 1(a)was recorded in the range 4000-667cm^{-1} and IR absorption signals were found at 3032 (√ Ar-H), 2980 and 2960 (√ aliphatic CH_2 and CH_3), 1760 (√ CO of ester group), 1610(√ C=N group) and 1182(√ C-O-C of ester group).

^{1}H NMR spectra (300MHZ, (CD) $_2$ SO, TMS): δ

^{1}H NMRSpectra ethyl 2-(3-phenyl imino)methyl-1H-Indole-1-yl-acetate 1(a)was recorded in DMSO-d6 solvent. The NMR signal of ethyl 2-(3-phenyl imino)metbyl-1H-Indole-1-yl-acetate(A) was found at $\delta_{PPm,}$ 1.29(t,3H, J=13.2Hz, CH_3 of ethyl group), 4.13 (q, 2H, J=13.2Hz, CH_2 of ethyl group), 4.78(s, 2H, N-CH_2 group) and 6.92 , 7.58 (m, 10H, C_8H_5N indole nucleus and C_6H_5 phenyl nucleus) and 8.44(s, 1H, N=CH group).

The compound (A) was converted into tetrazole on treatment with (1) PCl_3, 100^{0}C, 1hr (2) NaN_3 (ice cold), $ZnCl_2$, Sodium acetate, acetone, water, RT. The formation compound was confirmed by IR, NMR data.

NMR spectra

1.29(t,3H,CH_3 of C_2H_5), 4.78(s,2H N-CH_2-C =O), 4.13(q,2H,-O-CH_2 Of OC_2H_5), 6.92-7.58(m,10H,Ar-H,8.44(N=CH).

IR spectra

The compound (A) shows signals at, 1610(C=N), 1760 (ester –C=O), 3032(Ar-H), 1182(-C-O-C).

Ethyl2-(3-(1-phenyl-1H-tetrazol-5-yl)-1H-indol-1-yl)acetate 1(a)

A mixture of Schiffs base (0.01Mol) and (1) PCl_3, 100^{0}C,1hr (2) NaN_3 (ice cold), $ZnCl_2$, Sodium acetate, acetone, water, RT. The reaction was cooled and the resulting solid was washed with sodium bicarbonate solution and recrystalised from absolute alcohol. The formation compound was confirmed by IR, NMR spectral data.

NMR spectra

1.32(t, 3H, CH_3 of OC_2H_5), 3.70 (s,2H N-CH_2-C =O), 4.25 (q,2H,-O-CH_2 of OC_2H_5), 7.2-7.30(m,10H, due to 5H of indole, 5H of phenyl ring).

IR spectra

The compound 1 (a) shows signals at, 1616 (C=N), 1170 (-C-O-C-), 1723 (-C=O), (C-S-C), 695

Synthesis of ethyl 2-(3-(3-(4methyl phenyl)-1H-tetrazol-5-yl)-1H-indol-1-yl)acetate1(b). ^{1}H NMR spectra (300MHZ, (CD) $_2$ SO, TMS)

1.35 (t,3H,CH_3 of C_2H_5), 2.25(s,3H,CH_3 attached to phenyl ring,3.72 (s,2H N-CH_2-C =O), 4.28,(q,2H, O-CH_2 Of OC_2H_5), 7.22-7.32(m,9H,due to 5H of indole,5H of phenyl ring).

IR spectra

The compound 1(b) shows signals at, 1612 (C=N),1165 (-C-O-C-),1720 (-C=O),(C-S-C),693

Synthesis of ethyl 2-(3-(3-(4methoxy phenyl)- ^{1}H-tetrazol-5-yl)-1H-indol-1-yl)acetate1 (c). ^{1}H NMR spectra (300MHZ, (CD) $_2$ SO, TMS)

1.37 (t, 3H, CH_3 of C_2H_5), 2.27 (s, 3H, CH_3 attached to phenyl ring), 4.29 (q, 2H, O-CH_2 Of OC_2H_5), 7.25-7.35 (m, 9H, due to 5H of indole, 5H of phenyl ring).

IR spectra

The compound 1(c) shows signals at, 1610(C=N),1160 (-C-O-C-),1715 (-C=O),(C-S-C),691

Synthesis of ethyl 2-(3-(3-(4-chloro phenyl)-1H-tetrazol-5-yl)-1H-indol-1-yl)acetate1(d). ^{1}H NMR spectra (300MHZ, (CD) $_2$ SO, TMS)

1.39 (t,3H,CH_3 of C_2H_5), 3.73 (s,2H N-CH_2-C =O), 4.29 (q,2H,-O-CH_2 of OC_2H_5),7.28-7. 35 (m, 9H, due to 5H of indole, 5H of phenyl ring)

IR spectra

The compound 1 (d) shows signals at, 1605 (C=N), 1155 (-C-O-C-), 1710 (-C=O), (C-S-C), 690

Synthesis of ethyl 2-(3-(3-(4-nitro phenyl)-1H-tetrazol-5-yl)-1H-indol-1-yl)acetate1(e). ^{1}H NMR spectra (300MHZ, (CD)$_2$ SO, TMS)

1.40 (t,3H,CH_3 of C_2H_5), 3.75 (s,2H N-CH_2-C =O), 4.30 (q,2H,-O-CH_2 of OC_2H_5),7.29-7. 36 (m, 9H, due to 5H of indole, 5H of phenyl ring)

Ispectrara; The compound (1(e) shows signals at, 16 (0(C=,),1140 (-C-O-C,),1705 (-C=,),(C-S-,),698

Synthesis of ethyl 2-(3-(3-(4-trifluoro methyl phenyl)- 1H-tetrazol-5-yl)-1H-indol-1-yl)acetate1(f). ^{1}H NMR spectra(300MHZ,(CD)$_2$ SO,TMS)

1.42 (t,3H,CH_3 of C_2H_5), 3.77 (s,2H N-CH_2-C =O), 4.32 (q,2H,-O-CH_2 of OC_2H_5),7.31-7. 37 (m, 9H, due to 5H of indole, 5H of phenyl ring)

Ispectrara; The compound (1(f) shows signals at, 16 (5(C=,),1175 (-C-O-C,),1730 (-C=,),(C-S-,),700

Synthesis of 2-(3-(3-(4-substituted phenyl) - 1H-tetrazol-5-yl) -1H-indol-1-yl) acetohydrazide (2)

A solution of 1 (a) (0.01mol) and hydrazine hydrate (0.015) in ethanol (20ml) was refluxed for 5 hours. The reaction mixture was cooled and poured into ice cold water with stirring. The separated solid was filtered, washed with water and recrystalised from ethanol.

NMR spectra

4.36 (s,2H N-CH_2-C =O), 4.98 (s,1 H,-N-NH), ,4.28(s,2H,-NH_2)

6.9-8.3 (m, 10H due to 5H of indole, 5H of phenyl ring).

IR spectra

The compound 2 (a) shows signals at, 1620 (C=N), 1175 (C-O-C), 1730 (C=O), 698 (C-S-C).

Synthesis of 2-(3-(4-oxo-3-(p-tolyl) 1H-tetrazol-5-yl)-1H-indol-1-yl)-N-(1,1,1-trifluoropropan-2- ylidene) acetohydrazide (3)

To the solution of 2(a) (0.01mole) in hot methanol (25ml), acetophenone (0.01) and a drop of glacialaceticacid were added. The solid that separated on refluxing for 3hours was filtered wash with cold methanol and recrystalised from methanol to give 7(a).M.P.236^{0}C, yield 84%.

NMR spectra

2.54(s,1H, N=C-CH_3), 3.75 (s,2H N-CH_2-C =O), 4.90 (s,1 H,-N-NH), 7.1-8.3(m,10H due to 5H of indole,5H of phenyl ring).

IR spectra

The compound 3(a) shows signals at,1680(C=O,imide),1620(C=N), 3185(-NH),2950(-CH of aliphatic),3200(Ar-H), 700 (C-S-C)

Synthesis of 2-(1-((4-acetyl-5-methyl-5-(trifluoromthyl)-4,5-dihyro-1,3,4-oxadiazol-2-yl) methyl)-1H-indol-3-yl)-3-(p-tolyl) 1H-tetrazol-5-yl (4)

A mixture of 3(a) (0.01mole) and excessive acetic anhydride (10ml) was refluxed for two hours.

The excessive aceticanhydride was distilled off and the residue was poured into crushed ice. The solid thus obtained was filtered, washed with water and recrystalised from aqueous methanol to furnished obtained compound. M.P.185^{0}C, yield 56 %

NMR spectra

2.42(s,3H,CH_3), 2.46 (s,3H,-CO-CH_3),3.77(s,2H,-N-CH_2), 7.2 -8.5 (m,10H due to 5H of indole,5H of phenyl ring).

IR spectra

The compound 4(a) shows signals at,1680(C=O), ,1622 (C=N), 3130 (-NH of oxadizole),C-F(750) ,3200(N-H), 750(C-O -C) .

Synthesis of 2-(1-((4-acetyl-5-methyl-5-(trifluoromthyl)-4,5-dihyro-1,3,4-oxadiazol-2-yl) methyl)-1H-indol-3-yl)-3-(p-tolyl) 1H-tetrazol-5-yl (4a)

^{1}H NMR spectra (300MHZ, (CD) $_2$ SO, TMS)

2.42(s,3H,CH_3),2.46(s,3H,-CO-CH_3),3.77(s,2H,-N-CH_2), 7.2 -8.5 (m,10H due to 5H of indole,5H of phenyl ring).

IR spectra

The compound 4 (a) shows signals at, 1680 (C=O), 1622 (C=N), 3130 (-NH of oxadizole), C-F (750), 3200 (N-H), 750 (C-O -C).

Synthesis of 2-(1-((4-acetyl -5-(trifluoromthyl)-4,5-dihyro-1,3,4-oxadiazol-2-yl) methyl)-1H-indol-3-yl)-3-(p-tolyl) 1H-tetrazol-5-yl (4b)

^{1}H NMR spectra (300MHZ, (CD) $_2$ SO, TMS)

2.23(s,3H,attached to phenyl ring), 2.43(s,3H,-CH_3), 2.48(s,3H,-CO-CH_3),3.78(s,2H,-N-CH_2) , 7.3 -8.6 (m,9H,due to 5H of indole,5H of phenyl ring)

IR spectra

The compound 4(b) shows signals at,1680(C=O), ,1620 (C=N), 3100 (-NHof oxadizole),745(C-F) ,3195(N-H), 743(C-O -C) .

Synthesis of 2-(1-((4-acetyl -5-(trifluoromthyl)-4,5-dihyro-1,3,4-oxadiazol-2-yl)methyl)-1H-indol-3-yl)-3(4-methoxy phenyl) 1H-tetrazol-5-yl (4c)

^{1}H NMR spectra (300MHZ, (CD) $_2$ SO, TMS)

2.25(s,3H,attached to phenyl ring), ,2.44(s,3H,-CH_3), 2.50 (s,3H,-CO-CH_3),3.79(s,2H,-N-CH_2), 7.45 -8.65 (m,9H,due to 5H of indole,5H of phenyl ring)

IR spectra

The compound 4(c) shows signals at,1680(C=O),1620 (C=N), 3098 (-NH of oxadizole),740(C-F),3190(N-H), 741(C-O -C) .

Synthesis of 2-(1-((4-acetyl -5-(trifluoromthyl)-4,5-dihyro-1,3,4-oxadiazol-2-yl)methyl)-1H-indol-3-yl)-3(4-Choloro phenyl) 1H-tetrazol-5-yl (4d)

^{1}H NMR spectra (300MHZ,(CD)$_2$ SO,TMS)

2.45(s,3H,-CH_3), 2.49(s,3H,-CO-CH_3), 3.80(s,2H,-N-CH_2), 7.46 -8.68 (m,9H,due to 5H of indole,5H of phenyl ring)

IR spectra

The compound 4(d) shows signals at,1680(C=O), 1618 (C=N), 3105 (-NHof oxadizole), 750(C-F), 3188(N-H), 755(C-O -C).

Synthesis of 2-(1-((4-acetyl -5-(trifluoromthyl)-4,5-dihyro-1,3,4-oxadiazol-2-yl)methyl)-1H-indol-3-yl)-3(4-nitro phenyl) 1H-tetrazol-5-yl (4e)

^{1}H NMR spectra(300MHZ,(CD)$_2$ SO,TMS)

2.45(s,3H,-CH_3),2.48(s,3H,-CO-CH_3),3.77(s,2H,-N-CH_2), 7.44 -8.68 (m,9H,due to 5H of indole,5H of phenyl ring)

IR spectra

The compound 4(e) shows signals at,1680(C=O), ,1615 (C=N), 3110 (-NH of oxadizole),755(C-F) ,3185(N-H), 748(C-O -C)

Synthesis of 2-(1-((4-acetyl -4, 5-dihyro-5-methyl-1,3,4-oxadiazol-2-yl)methyl)-1H-indol-3-yl)-3-(4-trifluoromethyl) phenyl) 1H-tetrazol-5-yl (4f)

^{1}H NMR spectra(300MHZ,(CD)$_2$ SO,TMS)

2.45(s,3H,-CH_3), 2.49(s,3H,-CO-CH_3),3.77(s,2H,-N-CH_2), 7.47 -8.69 (m,9H,due to 5H of indole,5H of phenyl ring)

IR spectra

The compound 4(f) shows signals at,1680(C=O), 1621 (C=N), 3125 (-NH of oxadizole),765(C-F), 3198(N-H), 760(C-O -C)

Anti-Bacterial Activity

The anti bacterial activity of synthesized compounds was studied by the disc diffusion method against the following pathogenic organisms. The gram-positive bacteria screened were *staphylococcus aureus* NCCS 2079 and Bacilluscereus NCCS 2106. The gram negative bacteria screened were Escherichia coli NCCS 2065 and pseudomonas aeruginosa NCCS 2200.

The synthesized compounds were used at the concentration of250 µglml and 500µglml using DMSO as a solvent the cefaclor 10µglml disc was used as a standard. (Himedia, Laboratories Ltd, Mumbai).

The test results presented in the table -1,suggest that 4a,4d,4e exhibit high activity against the tested bacteria, the rest of the compounds were found to be moderate active against the tested microorganisms.

Antifungal activity

The antifungal activity of synthesized compounds were studied by disc diffusion method against the organisms of *aspergillus*

niger NCCS1196 and *cadida albicans* NCCS34471

Compounds were treatd at the concentrations of 500µglm and 1000µglml using DMSO as solvent. The standard used was clotrimazole 50µglml against both organisms. The test results were presented in the table-2.

Conclusion

1. Furthermore the substitution with a phenyl group having a chloro group at p-position showed better activities.
2. The tetrazoles showed better antibacterial and antifungal activities.
3. Thiazoles and its derivatives were found to play an important role in medicinal chemistry as herbicidal, fungicidal, bacterial, anti-inflammatory.

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Table 1. Antibacterial activity by disc diffusion method of indolelinked 1, 3, 4 oxadiazole having tetrazole 4(a.f)

Compound	Zone of inhibition (mm)			
	Staphylococcus aureus	Bacillus cereus	Escherichia coli	Pseudomonas aeruginosa
4a	16	18	13	12
4b	14	11	15	10
4c	13	12	10	09
4d	16	17	12	11
4e	18	16	15	17
4f	11	14	13	12
Cefaclor	19	22	19	20

Table 2. Antifungal activity by disc diffusion method for indole linked1, 3, 4 oxadiazole having tetrazole 4(a-f)

Compound	Zone of inhibition (mm)	
	Asperigillus niger	Candida albicans
4a	14	16
4b	15	13
4c	17	15
4d	18	17
4e	23	21
4f	15	13
Clotrimazole	25-30	25-30