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SYNTHESIS OF 1,3,4-OXADIAZOLE DERIVATIVE CARRYING BENZIMIDAZOLE

*Syed Nazim, Maulana Azad College
**Maqdoom Farooqui, Dr.Rafiq Zakaria Campus-I
***Ayesha Durrani, Dr.Rafiq Zakaria College For Women Aurangabad.

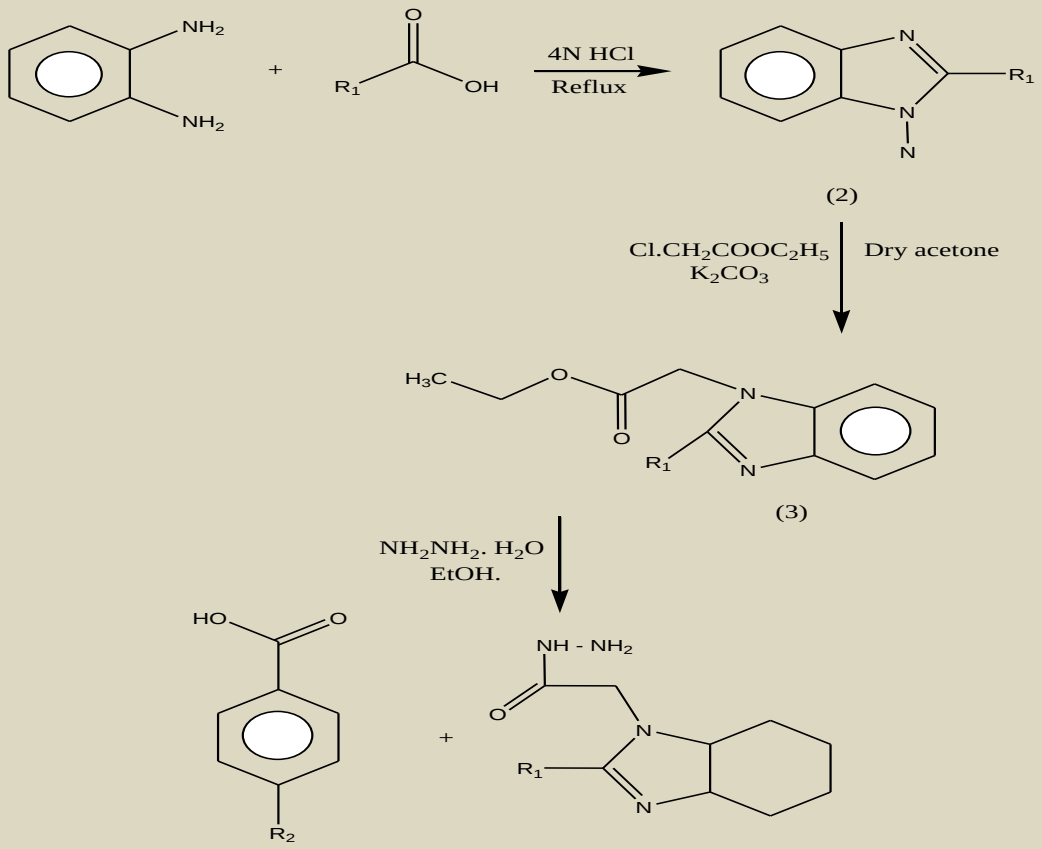
Introduction:

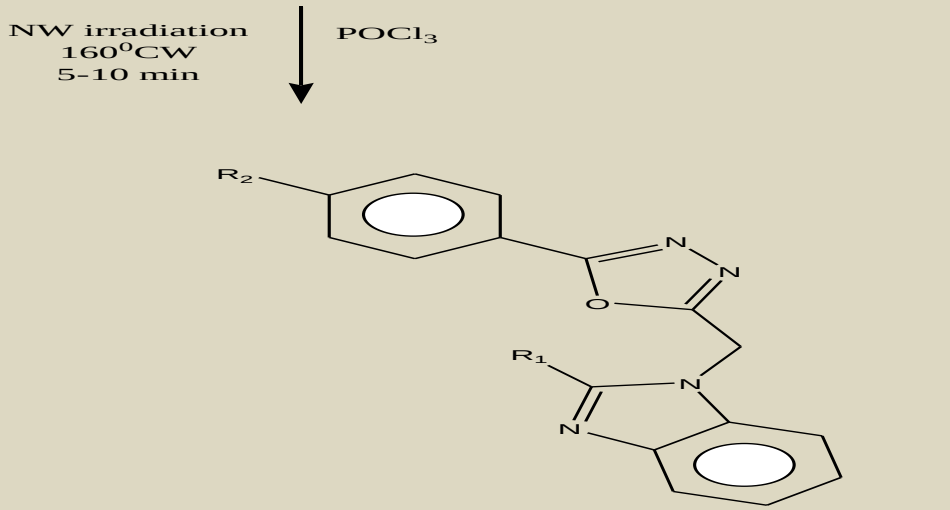
Benzimidazole and its derivatives are of great importance in medicinal chemistry because of wide variety of pharmaceutical and biological activity [1,2].

It was observed that from the literature survey that five membered heterocyclic compounds possess biological activity. Compounds having 1,3,4-oxadiazole and pyrazole nucleus have many applications in medicinal chemistry. These compounds also have been reported to have significant antitubercular activity [3,4]. Oxadiazole derivatives were reported to have number of biological activities such as fungicidal, analgesic, antibacterial, tranquilising properties etc. [5,8].The various biological activity of benzimidazole and oxadiazole derivatives, it was contemplated to synthesize a new series of 1,3,4-oxadiazoles carrying benzimidazole moiety.

Experimental:

Scheme of the reaction





(5a - e)

Materils & Methods:

Melting points were determined using open capillary method and are uncorrected. The compounds were checked for homogeneity by TLC on silica gel G. The IR Spectra were recorded by using JASCO FTIR Spectro photometer using kBr – disc method.

Step 1: Synthesis of 2-substituted benzimidazole (2)

A mixture of O-phenylenediamine (0.09 mole) and aliphatic acid (0.11 mole) was dissolved in 4N HCl and refluxed at 1000C for 12 hours. After completion of reaction TLC was carried out. The contents were cooled at room temperature and neutralized with saturated solution of NaHCO3. The solid product was filtered, washed and dried, recrystallised from suitable solvent.

Step 2: Synthesis of Ethyl – (2- substituted – 1 – H – benzimidazole – 1- yl) acetate (3):

The solution of (2) was taken 0.06 mole in 20 ml of acetone and 0.07 mole ethyl chloroacetate and potassium carbonate, about 0.12 mole was added and refluxed for 6 hour. After 6 hours product obtained was filtered, dried, excess of acetone was distilled off and solid product was separated and dried, recrystallised, melting point was recorded and TLC was checked.

Step 3: Synthesis of 2 – (- 2 – substituted – 1- H – benzimidazol – 1 – yl) – acethydrazide (4):

The (3) compound obtained was dissolved 0.04 moles in 15 ml ethanol and then mixed with hydrazine hydrate 0.044 mole refluxed this mixture about 4 hour. After 4 hours of the reaction TLC was checked. The solvent is removed by distillation and then the solid was filtered, washed and recrystallised from alcohol. The percentage yield was recorded.

Step 4: Synthesis of Oxadiazoles:

The product obtained from step 3 was mixed with 0.03 moles of aromatic acid and 1 ml of phosphorus oxychloride was mixed. The mixture was stirred to get homogenous mixture then it was heated in a beaker under Microwave at 160W for 5 min. After completion of heating TLC

was checked. The product was cooled at room temperature then excess of ice cold water was added. The product formed was filtered and recrystallised from ethanol. The percentage yield and Melting point were recorded.

Result & Discussion:

The preparation of 1,3,4-oxadiazole derivative followed the 4 step. The structure of the final compound was confirmed on the basis of spectra and analytical data. The IR spectra of 1,3,4-oxadiazole derivatives carrying benzimidazole showed absorption bands in the region of 3066 – 2965 Cm–1 which is characteristic of C-H stretching. The C=N absorption band was observed around 1580-1600 Cm-1 .

Table No 1: Characterization of 2-substituted-1(-5-substituted phenyl-1,3,4-oxadiazole-2-yl) methyl] 1H – benzimidazole:

Comp.	R1	R2	M. Pt.	Mol. Form. (wt)	Found %				
					C%	H	N	O	Cl
5a	- CH3	- H	1520	C17H14N4O = 302	67.54	4.635	22.516	5.298	--
5b	- CH3	- NH2	2000C	C17H15N4O = 303	67.32	4.950	22.44	5.280	--
5c	- CH3	CH3	1350	C18H16N4O = 316	68.354	5.063	21.518	5.06	--
5d	- CH3	- Cl	2050C	C17H13N4OCl = 336	60.714	3.869	20.238	4.76	10.416
5e	- C6H5	- H	1700C	C22H16N4O = 364	72.527	4.895	18.68	4.395	--
5f	- C6H5	- NH2	1830C	C22H17N5O = 367	71.93	4.632	19.07	4.359	--
5g	- C6H5	- CH3	1580C	C23H18N4O = 378	73.015	4.76	17.989	4.23	--
5h	- C6H5	- Cl	1690C	C23H16N4OCl = 411	67.153	3.892	16.54	3.89	8.51

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