NOTE

Synthesis of Some Phthalimido-oxy and Amino-oxy Derivatives of Benzotriazole

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Some important N′-[(benzotriazol-1-yl) alkoxy] phthalimides (6a–c) were synthesized by condensation of phthalimido-oxy alkyl bromides with benzotriazole. These compounds on hydrolysis in acidic media were converted to corresponding amino-oxy compounds (7a–c).

A survey of literature reveals that benzotriazole derivatives possess immense pharmacological properties such as anti-inflammatory and analgesic[1], C.N.S. depressant[2], antimicrobial[3] and fungicidal[4]. Many phthalimido-oxy and amino-oxy compounds have been reported to possess antimalarial[5], C.N.S. depressant[6] and hypertensive[7] activities. Keeping in view the therapeutiacal uses of both the above, some phthalimido-oxy and amino-oxy derivatives of benzotriazole (6a–c and 7a–c) have been synthesized.

The starting material used was N-hydroxy phthalimide (1)[8]. This was treated with ω-ω dibromoalkanes (2) to produce phthalimido-oxy alkylbromides (3)[9]. Here excess of dibromoalkane was used in order to avoid formation of bis-phthalimido-oxy alkanes (4). Phthalimido-oxy alkylbromides (3) when condensed with benzotriazole (5) in acetone media in presence of anhydrous K_2CO_3 produced phthalimido-oxy alkyl benzotriazole (6a–c). (6a–c) on hydrolysis with a mixture of glacial acetic acid and 48% hydrobromic acid gave corresponding amino-oxy compounds (7a–c).

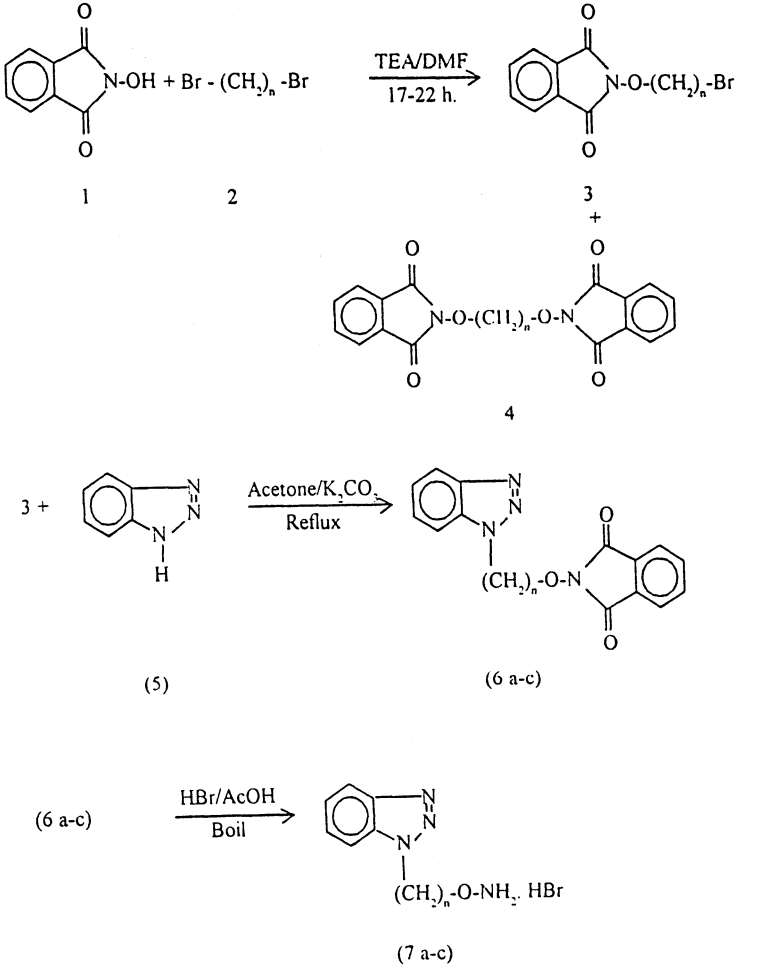
IR (KBr): *Phthalimido-oxy compound*: ν_{max} (cm^{-1}) 1791–1735 (CO—N—CO); 1614 (asymmetric stretching of N—O bond); 1278 (O—N symmetrical absorption); 2923 (asymmetric stretching —CH_2); 1371–1326 (5 membered ring attached to benzene nucleus); 1080–1032 (characteristic of triazole ring).

IR (KBr): *Amino-oxy compounds*: ν_{max} (cm^{-1}) 3064–2990 (symmetric and asymmetric stretching of NH_3^+ group); 1280 (O—N symmetric absorption); 2935 (—CH_2 stretch); 1085–1035 (characteristic of triazole ring).

TABLE-1
ELEMENTAL ANALYSIS OF SYNTHESIZED N′-[(BENZOTRIAZOL-1-YL) ALKOXY] PHTHALIMIDES

Compound	Analysis % found (calcd.)		
	C	H	N
6a	61.49 (62.33)	4.33 (3.89)	18.58 (18.18)
6b	63.75 (63.35)	4.09 (4.34)	17.24 (17.39)
6c	63.72 (64.28)	5.01 (4.76)	16.97 (16.66)

Following reaction sequence was used to synthesize above mentioned compounds:



Here n=2, 3 or 4

All the melting points are uncorrected; purity of compounds was checked by TLC and other usual methods. We have prepared N-hydroxyphthalimide (1) by the method of Orndroff and Pratt[8]; while phthalimido-oxy alkyl bromides (3) was prepared by method of Bauer and Suresh with slight modification[9].

Preparation of N-[(benzotriazol-1-yl) alkoxy] phthalimides (6a–c)

General Procedure: A clear solution of phthalimido-oxy alkylbromide (0.01 mol) and benzotriazole (0.01 mol) in acetone was refluxed on water bath for 6–10 h. Anhydrous K_2CO_3 (0.01 mol) was added to it and refluxing was continued for

the next 24–30 h. Reaction mixture was filtered. Solvent was evaporated under vacuo. Solid obtained was recrystallised from suitable solvents. Physical data of the synthesized compounds are given in Table-2.

Preparation of amino-oxy alkyl benzotriazole (7a–c)

General Procedure: Compounds (7a–c) were boiled in a mixture of glacial acetic acid and 48% hydrobromic acid (2 : 3) for 20 min. On cooling phthalic acid separated was filtered. Filtrate on evaporation under reduced pressure gave solids which were washed with ether and recrystallised. Physical data of the synthesized compounds are given in Table-2.

TABLE-2
PHYSICAL DATA OF THE SYNTHESIZED COMPOUNDS

Comp. no	m.f.	m.w.	Solvent for cryst.	m.p. (°C)	Yield (%)
6a	$C_{16}H_{12}N_4O_3$	308	ethanol	135	64
6b	$C_{17}H_{14}N_4O_3$	322	ethanol	143	58
6c	$C_{18}H_{16}N_4O_3$	336	ethanol	155	52
7a	$C_8H_{11}N_4OBr$	259	methanol	220	40
7b	$C_9H_{13}N_4OBr$	273	methanol + chloroform	245	35
7c	$C_{10}H_{15}N_4OBr$	287	methanol + chloroform	270	30

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