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Synthesis of Polyhydric Phenolic Phenacyl Ethers

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ABSTRACT

Polyhdric phenolic phenacyl ehters are synthesised by micellar mediated reaction with phenacyl and its substituted phenacyl bromide. Biological importance of poly phenacyl ethers are reviewed. Physical data, IR, ^{1}HNMR and ^{13}CNMR are used to characterize the new compounds.

Key words: Phenolic ether, Antimicrobial, Phenancyl bromide.

INTRODUCTION

Phenolic ethers play huge role in medicinal chemistry and some of them are used as adhesives and holt melt. Alkyl phenolic ether prodrugs have been reported where coupling with a hydroxymethylimide using mitsunobu chemistry yielded the product[1]. Diphenyl ether like chlornitrofen was widely used in large quantities as a herbicide to control various weeds in rice fields[2]. Polybrominated diphenyl ethers are used as brominated fire retardants. These diphenyl ether had an adverse impact on the structure and function of the soil microbial community and microbial processes[3]. Polybrominated diphenyl ethers (PBDEs) were applied as polymers for many plastic and electronic products. Due to their unbiquitous distribution in the environment, potential toxicity to human and tendency for bioaccumulation, PBDEs have raised public safety concern[4]. Phenolic ethers are synthesised by various methods with different reactant and also in different envrionment. This type of reactions are involved in biological and human mechanism. Such kind of mechanism deals with mono hydric phenols and poly ring phenols. These concepts promoted us to prepare such type of phenolic compounds in different environment.

MATERIAL AND METHODS

Trihydric phenols like Pyrogallol (1,2,3 tri hydroxyl benzene), Phlorogluction (1,3,5 tri hydroxyl benzene) and Hydroxyquino (1,2,4 tri hydroxyl benzene) react with phenacyl bromide and methyl, chloro, methoxyl, bromo substituted phenacyl bromide. Here one mole of trihydric phenol is taken for the reaction and 3 moles of phenacyl and appropriately substituted phenacyl bromide. The total reaction was carried out in micellar solution[5-6]. The micellar medium have both hydrophobic and

hydrophilic region so as to facilitate the reaction to the right path. In this reaction triethylamine was used as cosurfactant[7]. Overall reaction is carried out in room temperature with continuous stirring of 5-10 hrs. Overall reaction solid product was filtered off from the reaction mixture and it was washed several time with water and petroleum ether. Dry product was tested in TLC with ethyl acetate and petroleum ether as the eluent. Synthesis of these 1,2,4 trihydric phenolic phenacyl ethers are given in scheme 1. Similar procedure was adopted for the synthesis of 1,2,3 and 1,3,5 trihydric phenolic phenacyl ethers.

RESULTS AND DISCUSSION

The reaction of trihydric phenols with variously substituted phenacyl bromides in the micellar medium yielded the desired triphenolic

Table 1: Physical data of phenolic phenacyl ethers

Cmp. No	Yield	m.p. °C	MF	FW	Found C, H, N Cal. (C,H,N)
1	62.25	68-70	$C_6H_3(OCH_2COC_6H_5)_3$	480	74.6,5.0-(47.8,4.8,-)
2.	53.98	117-119	$C_6H_3(OCH_2COC_6H_5CH_3)_3$	525	75.0,5.8,-(75.2,6.0,-)
3.	59.09	110-112	$C_6H_3(OCH_2COC_6H_5Cl)_3$	586	61.1,4.2-(61.3,4.0,-)
4.	69.75	92-94	$C_6H_3(OCH_2COC_6H_5OCH_3)_3$	574	67.8,5.0,-(68.0,4.8,-)
5.	61.14	126-128	$C_6H_3(OCH_2COC_6H_5Br)_3$	720	50.3,3.4-(50.1,3.2,-)
6.	83.32	84-85	$C_6H_3(OCH_2COC_6H_5)_3$	480	74.7,5.5-(74.9,5.3,-)
7.	83.98	120-121	$C_6H_3(OCH_2COC_6H_5CH_3)_3$	525	75.3-6.5-(75.5,6.3-)
8.	84.88	145-147	$C_6H_3(OCH_2COC_6H_5Cl)_3$	586	61.0,4.3-(61.8,4.1,-)
9.	88.23	131-133	$C_6H_3(OCH_2COC_6H_5OCH_3)_3$	574	68.0,5.0,-(68.1,5.2,-)
10.	66.78	99-101	$C_6H_3(OCH_2COC_6H_5Br)_3$	720	50.5,3.0,-(50.3,3.2,-)
11.	84.11	118-120	$C_6H_3(OCH_2COC_6H_5)_3$	480	74.0,5.5,-(74.2,5.3,-)
12.	59.98	101-103	$C_6H_3(OCH_2COC_6H_5CH_3)_3$	525	75.2,6.0,-(75.0,5.8,-)
13.	64.47	95-97	$C_6H_3(OCH_2COC_6H_5Cl)_3$	586	61.0,4.6,-(61.2,4.8,-)
14.	60.05	73-75	$C_6H_3(OCH_2COC_6H_5OCH_3)_3$	574	68.5,5.0,-(68.7,5.2,-)
15.	70.57	89-91	$C_6H_3(OCH_2COC_6H_5Br)_3$	720	50.7,3.5,-(50.9,3.3,-)

Table 2: IR spectra data of ether compounds

Compound No	C-H Aromatic	C-H Aliphatic	C=O	C=C	C-O-C
1	3082	2956	1696	1585	1226
2	3032	2920	1695	1606	1232
3	3091	2923	1688	1487	1230
4	3057	2918	1683	1502	1216
5	3032	2920	1695	1606	1232
6	3021	2956	1996	1585	1284
7	3090	2920	1676	1570	1232
8	3055	2922	1678	1596	1227
9	3056	2910	1686	1489	1228
10	3101	2920	1708	1590	1283
11	3055	2922	1678	1596	1227
12	3059	2906	1710	1581	1282
13	3092	2901	1711	1584	1283
14	3091	2923	1688	1487	1230
15	3112	2927	1595	1433	1240

(substituted) phenacyl ethers (1-15). The formation of these compounds are revealed by the preliminary laboratory analysis and then the CHN analysis (Table 1). The important group frequencies like aromatic C-H str., aliphatic C-H str., carbonyl str. aromatic C=C str., C-O-C str. (Table 2) showed the formation of the compounds(1-15). The formation and structure of these compounds are further confirmed by the data of ^{1}H NMR and ^{13}NMR spectra (Table 3 and 4).

Table 3: ^{1}H NMR data of ether compounds

Compound No	OAr, COAr	-O-CH_2-CO	CH_3
1	7.54-7.91m,7.21-7.45m	5.39s	2.44
2	7.81-7.92m,6.36-7.65m	5.42s	
3	7.85-8.04m,7.20-7.70m	5.54s	
4	7.26-7.98m,6.38-6.92m	5.51s	3.85*
5	7.29-7.99m,6.49-6.92m	5.15s	
6	7.29-7.99m,6.93-6.45m	5.58s	
7	7.45-7.87m,6.12-7.26m	5.53s	2.94*
8	7.84-8.12m,7.20-7.67m	5.12s	
9	7.26-7.95m,6.12-6.96m	5.50s	
10	7.50-8.02m,7.12-7.48m	4.58s	
11	7.85-8.04m,7.03-7.42m	5.53s	
12	7.56-7.96m,7.03-7.42m	5.11s	2.50
13	7.83-8.07m,7.21-7.50m	5.52s	
14	7.48-80.7m,6.18-7.36m	5.16s	3.37*
15	7.60-7.87m,7.26-7.27m	4.40s	

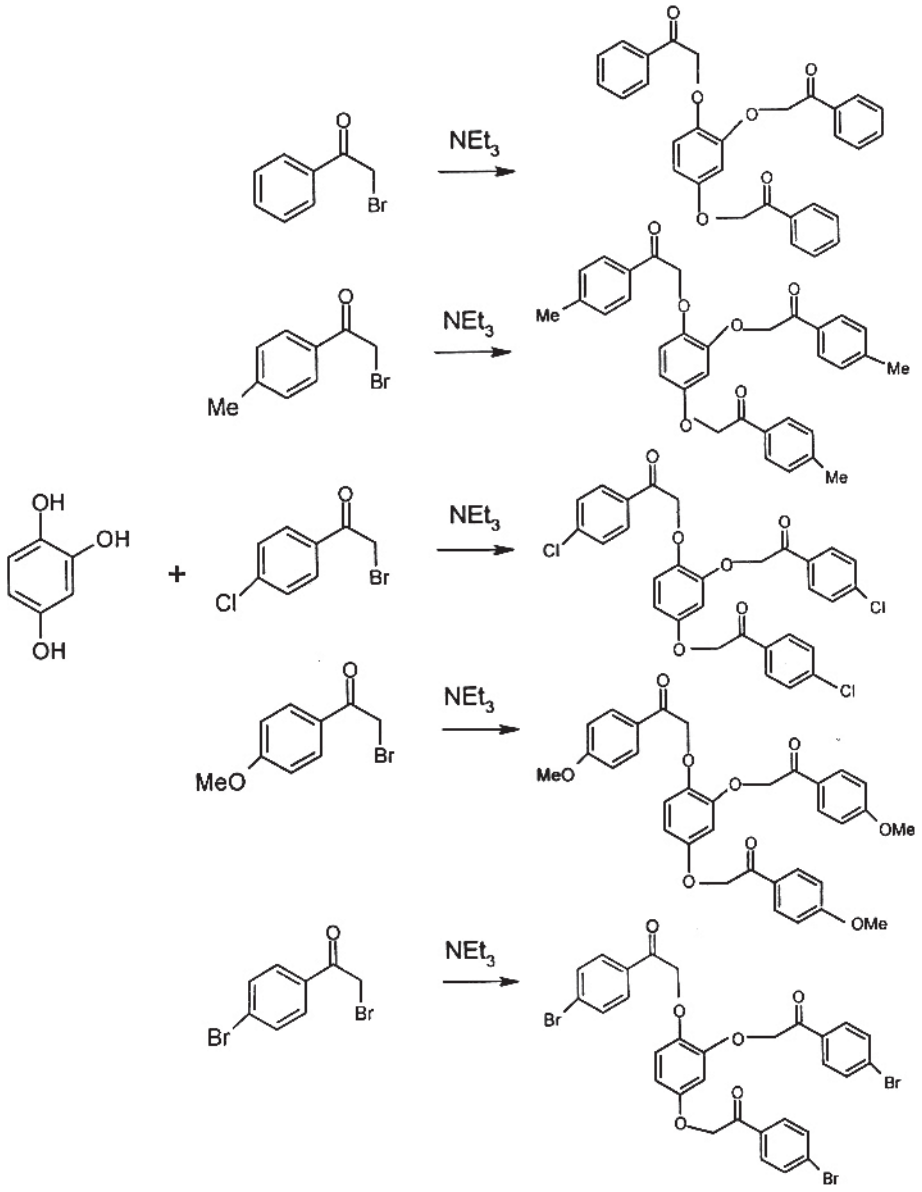
*Data correspond to methoxy methyl group

Table 4: ^{13} C NMR data of phenolic phenacyl ethers

Compound No	Ar-C=O	Aromatic carbon	*Ipso*-carbon	CH_2-O-	CH_3
1	191	135	128	65	
2	194	144	129	70	21
3	192	132	127	66	
4	197	130	114	55	55*
5	191	132	129	56	
6	191	135	128	65	
7	193	132	127	80	21
8	190	140	129	66	
9	192	130	114	76	55*
10	199	132	129	55	
11	190	140	130	30	
12	191	144	127	66	30
13	190	140	129	54	
14	192	130	127	70	55*
15	190	132	129	30	

*Data correspond to methoxy methyl group

In the above experiments three types of trihydric phenols have been chosen as the substrate. They are 1,2,3 trihydroxy-, 1,3,5 trihydroxy and 1,2,4 trihydroxy phenols. The significance of these reaction is that in order to increase the yield the reactions were mediated in micellar medium. In this medium the formation of the phenolate ion is stabilized more easily. The movelty of the trihydric phenolic phenacyl ethers are based on its biological activity. The antimicrobial activities of these compounds are presented (Table 5). Microorganisms like Staphylococcus aureus. Escherichia coli are used to determine the antimicrobial studies for the compounds.



Scheme 1: Preparation of 1,2,4 tri phenolic (substituted) phenacyl ethers.

Table 5: Biological activity data of ether compounds

Compound no	Zone of inhibition	Staphylococcus	E.coli
1	50µg	10	10
	150 µg	16	15
2	50µg	11	12
	150 µg	14	14
3	50µg	13	18
	150 µg	18	20
4	50µg	14	10
	150 µg	16	16
5	50µg	16	10
	150 µg	21	12
6	50µg	10	12
	150 µg	13	14
7	50µg	10	10
	150 µg	10	13
8	50µg	22	11
	150 µg	14	14
9	50µg	12	12
	150 µg	14	16
10	50µg	12	12
	150 µg	18	18
	Std	30	38

Standard (Std)-ciprofloxacin 5µg/disc for bacteria solvent control (Sc) - DMSO

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