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Synthesis and biocidal screening of 4-biphenyl acetamides derivatives.

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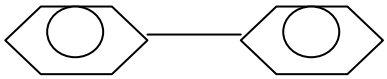
Abstract
The present paper deals with the synthesis of 4-biphenyl acetic acid amides by condensation of corresponding acid chlorides with suitable amines. the structure of newly synthesized compounds were elucidated on the basis of their IR, TLC and elemental analysis data. The compounds were also screened for their anti-bacterial and anti-fungal activity.
Key Words – Synthesis, biphenyl derivatives, spectral and biocidal activity.

Introduction

Biphenlys are the polynculear aromatic hydrocarbons having more than one aromatic nucleus. The two aromatic nuclei are attached to each other at only one point. Thus, biphenyls with independent benzene rings have been catagorizzed in the class of polyphenyl compounds or isolated polynuclear hydrocarbons.



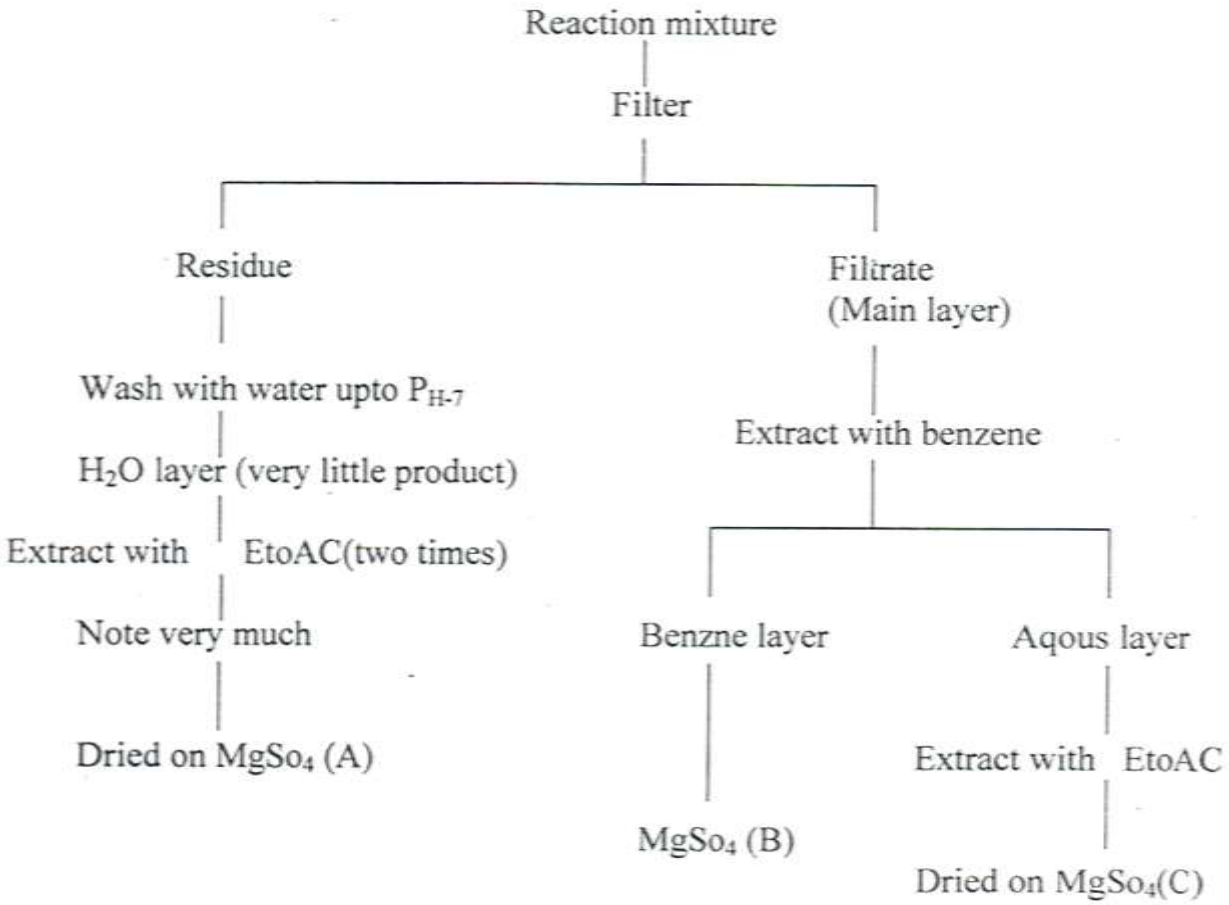
Biphenyls and polynculear aromtic hydrocarbons (PAHs) have been reported in the literature to found naturally at several places in the environment. American Chemical Society reported a novel palladium. catalyzed ullmann- type reductive coupling of aryl-halides, under an air atmosphere and in equeous acetone to obtain different type of biphenyl derivatives. [11-12] The newly synthesized 4- biphenyl actamides derivatives are biocidal screening to evaluate their possible use as antifungal and antibacterial activities.

Experimental

All the chemicals used for the synthesis were of Analar grade. Distill solvent were used throughout the experiment.

Synthesis of Compounds

This paper includes the synthesis of simple 4-biphenyl acetamides analoges. The syhthesis of these analoges containing two step. In 1st step converted 4-biphenyl acetic acid[13] (4-BPAA) into 4-biphenyl acetyl chloride[4-BPAC(as a various liquid)] by refluxing 4-BPAA with thionyl chloride in dry benzene for 2 ½ hours and in 2nd step viscous oil treated with different type of suitable amines at room temp in the presence of 4N-NaOH by stirring, in order to prepared different types of amides of 4BPAA. This scheme is clear from following diagramatic representation.



Ist Step 4-BPAC (1gm) in dry benzene (25 ml) {benzene distilled over on anhydrous Cacl21 and thionyl chloride (1ml) added in a 100 ml of R.B. flask, and refluxed the reaction mixture for 2 ½ hrs. After 1 hour the colour of mixture change from yellow to brown. After 2 ½ hrs thionyl chloride along with benzene. Traces of thionyl chloride removed with the help of vaccum pump 4-Biphenyl acetyl chloride obtained in oily form and w3ecl without further purification in next step to form different types of amides of 4-BPAA.

IInd Step 4- E3PAC (875 mg), benzene (25m1), ethyl acetate (25ml) and aqous ammonia (3.0 ml + 15 ml water) one by one slowly add in a 250 ml of R.B. flask slowly under stirrng at room temp and stirring continue for 3 hrs. Workup the reaction mixture with benzene + Ethylacetate after 20 hrs, after checked the TLC of reaction mixture. Reaction mixture takes in a saperatory funnel along with distilled water. Compound is in the benzene layer, wash out the benzene layer with water 3-4 times to remove the basic nature of the benzene layer.

When benzene layer becomes neutral, this layer was taken in a conical flask and add $MgSo_4$ (to absorb the moisture of benzene layer), wait for 5-10 minutes. Filtered the solution in a R.B. flask and recovered the benzene from reaction mixture by distillation and traces of benzene with the help of vaccum pump. Concentrate residue was treated with hexane for complete precipitation. Light pale yellow coloured crystalline solid compound obtained, filter through whatman filter paper No. 42, wash the ppt. with hexane 2-3 times, dry and weigh. The same procedure were synthesized 4-Biphenyl acetamide derivatives such as –

A_1 - (4- Biphenyl acetamide)

A_2 - (N-phenyl - 4-Biphenyl acetamide)

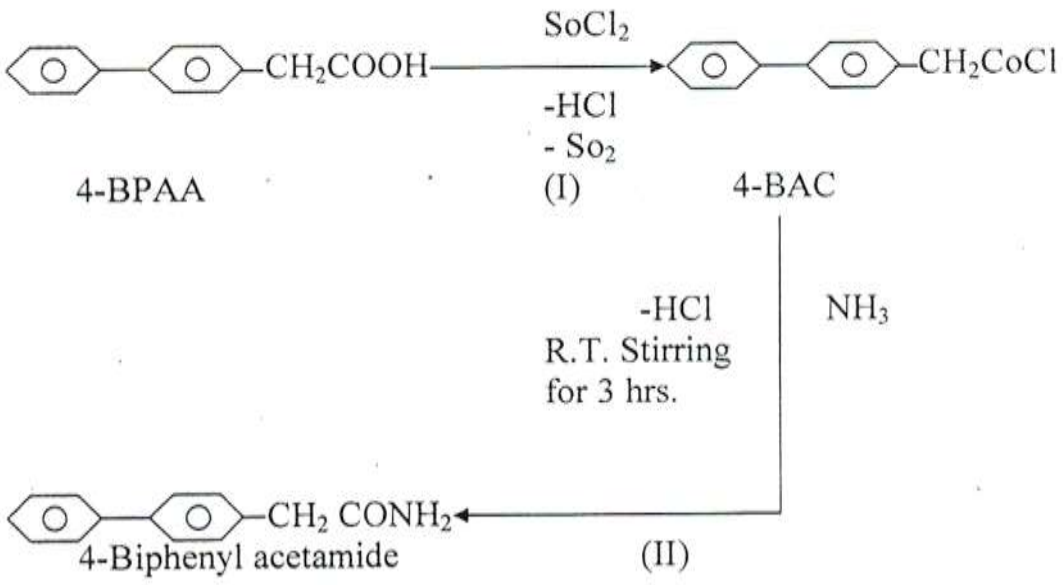
A_3 - (N-p- 'T'oluene - 4- Biphenyl acetamide)

A_4 - (N-a- Napthyl- 4- Biphenyl acetamide)

A_5 - (N-phenyl thioamide-4- Biphenyl acetamide)

A_6 - (N-Bcnzyl- 4 - Biphenyl acetamide)

Scheme – I & II



Various types of amides of 4-BPAA having two-Co groups and laving-CO-NH-CO type bonding During the synthesis of such type of the compounds first of all we do the acetylation of 4 -BPAA as discussed earlier then 4-Biphenyl acetyle chloride (4 - BPAC) react with different types of uitable aliphatic and aromatic amines having free–NH2 group to prepare a various types of amides of 4-BPAA. The characteristic JR bands (4000-200 cm^{-1}) for the 4-BPAA, 4-BPAC and

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4-BPAA derivatives compounds provide meaningful information regarding the bonding sites of the amides. The IR spectra show characteristic bands in the region 3243-3255 cm^{-1} with free $>NH_2$[14] and the region 1630-1645 cm^{-1} showed >CO group.

In this scheme commercially available and synthesized amides were used those having free-NI-12 group. But the results were not poor and an average 30-90% yield of such type of the synthesized amides obtained and with very few failures.

The analytical results, melting point, colour, yield and 1R bands of the compounds are presented in table-1.

Biocidal Activity

The compounds were also screened for their antifungal activity of disc-plate method(15 against C.lunata Seven days old culture were used as test organism which were grown on dextrose-agar medium. The fungi were grown at R.T. 10 ± 30C and the average of three replications was recorded with control plate. The percentage inhibition (16) was calculated as (C-T) x 100/C where C-diameters of fungus colony in control plate and T-diameter of Fungus colony in test plate. The compounds A1, A2 & A3 showed high activity, while other A4, A5& A6 compounds showed less activity against the above organism table-2.

The antibacterial activity results are given in table-2. The compounds. were screened for their against pathogenic bacteria E. Coli[17-19] by using disc-plate method. The medium used thought the experiments was Hi-media.[15] These compounds have tested for their antibacterial activity. The antibacterial activity of first-three compounds were found higher than last three compounds.

TABLE-1

Physical Characteristics and analytical data of 4-BPA analogs.

Compounds Code	IR Bands	Obtained yield (mg)/(%)	M.F./M.W.	Colour/M.P. (^{0}C)	T.L.C. (20-25% EtoAc.:hexane)
A_1	> Co-1639 cm^{-1} >NH-3352.9 cm^{-1}	1210/84	$C_{14}H_{13}NO$/211	Yellow/200	Rf-0.4337
A_2	> Co-1652.7 cm^{-1} >NH-3343.7 cm^{-1}	875/64	$C_{20}H_{17}NO$/287	Pale Yellow/197	Rf-0.44
A_3	> Co-1654.3 cm^{-1} >NH-3243.8 cm^{-1}	890/80	$C_{21}H_{19}NO$/301	Brown/195	Rf-0.3139
A_4	> Co-1658.4 cm^{-1} >NH-3232.4 cm^{-1}	610/45	$C_{24}H_{19}NO$/337	Light Purple/190	Rf-0.418
A_5	> Co-1652.7 cm^{-1} >NH-3349.9 cm^{-1}	390/30	$C_{21}H_{18}N_2OS$/346	Cream/173	Rf-0.45
A_6	> Co-1635.8 cm^{-1} >NH-3287.2 cm^{-1}	590/90	$C_{21}H_{19}NO$/301	Cream/198	Rf-0.310

TABLE-2

Biocidal Screening of 4-BPA analogs.

Compounds Code	Time Fungi/Bacteria	Temperature	Control (in mm) (Ethyl alcohol) C.Lunata/E.Coli	C.Lunata / E. Coli (in mm)		
				250 ppm	500 ppm	1000 ppm
A_1	144/55	10 ± 3^0C	18/20	13/10	6/5	0/1
A_2	144/48	12 ± 2^0C	20/21	17/8	8/4	2/0
A_3	200/60	10 ± 2^0C	15/18	11/12	3/6	1/2
A_4	188/55	13 ± 3^0C	22/16	19/17	10/8	2/2
A_5	140/60	10 ± 2^0C	15/18	16/10	8/5	2/1
A_6	188/60	13 ± 3^0C	11/16	//11	5/6	1/0

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