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Biocidal screening of Bromo-Substituted 4-biphenyl acetamides derivatives.

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ABSTRACT

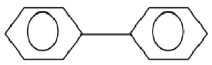
The present paper deals with the Biocidal screening of Bromo-Substituted 4-biphenyl acetic acid amides by condensation of corresponding acid chlorides with suitable amines. the structure of newly synthesized compounds were elucidated on the basis of their IR, TLC and elemental analysis data. The compounds were also screened for their anti-bacterial and anti-fungal activity.

Key words: Synthesis, biphenyl derivatives, spectral and biocidal activity.

Introduction

4-BPAA itself has been reported to possess many effective pharma cological activities, such as anti-inflammatory, analgesic, anti-bacterial and topical non steroidal anti-inflammatory activity. The ointment and various types of patches containing 4-BPAA work very effectively as anti-inflammatory and analgesic agents. 4-BPAA cyclodextria inclusion compounds are reported to show effective mono nuclear rogenic anti-inflammatory properties and its phenyl alkanamide derivatives have shown agro horticultural bacteriocidal activity.

As the literature findings have shown the encouraging results regarding the anti-inflammatory, analgesic and antipyr etic properties of BPAA and its derivatives. It was therefore decided to explore the synthesis of new derivatives derived from BPAA.



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 Bromo-Substituted 4- biphenyl actamides derivatives are biocidal screening to evaluate their possible use as antifungal and antibacterial activities.

Experimental

Agar Agar Media / Czepeck's Media :

This media was used for the growth of fungus. Thus, first of all prepared this media according to our requirement. 500ml. media was sufficient for 24 petric plates, so that 250ml media was sufficient for 12 petric plates.

Procedure of Preparation of Czapeck's media :

Dissolved all the contents of czapeck's media in calculated amount for 500 ml. distilled water. then used conical flask of 500ml. capacity, which was already 24 hourse sterilized in an oven at a maintained temperature (30^{0}C). Take 500 ml. distilled water in the sterilized conical flask, then add all the contents one by one carefully in the conical flask, in sterilized chamber. Hands were also sterilized for preserving the experiment from bacterial contaimination after that conical flask in autoclave and heated it upto 15 mm. pressure. Then, release the pressure upto the zero point. Now, put the conical flask on the table for achieving the room-temperature. But, media should not be cooled down, if it becomes freezed out then useless for us.

Procedure of Preparation of Fungus Solution :

Take distilled water (25 ml) in a conical flask and add some procelline pieces in it. Then, sterilized the conical flask in autoclave upto 100 mm. pressure, Now, the pressure of autoclave comes down upto zero Point, then release the Pressure of autoclave and wait for few minuts. Now, open the autoclave and Put the conical flask on the table for achieving room temperature. then, inject very few quantity of fungus used for the growth such as :- **Fujeerium-Udum** with the help of Anaculation needle in sterilized medium. Now, Shake very well the conical flask to spread out the spares of fungus in the water finely.

Procedure of Growing the Fungus :

Take two petric plates and paur 1 ml. solution of fungus (used for the identification of antifungal Properties of compound) in each Petric Plate, add Agar-Agar Media (15ml) in each Petric Plate. Then, wait for 4-5 days for the growth of fungus in these Petric Plates.

After the browth of particular fungus, cut the fungus of a particular size (3mm). These blocks were replaced in another petric plate alongwith Czapeck's media (15ml) and the solution of compound (1ml). Now, the identification of antifungal property of a Particular compound on specific fungus might be Possible.

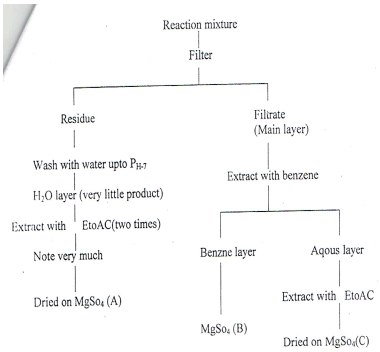
Synthesis of Compounds

This paper includes the synthesis of simple Bromo-Substituted 4-biphenyl acetamides analoges. The syhthesis of these analoges containing three step. In 1st step converted 4-biphenyl acetic acid[13] (4-BPAA) into 3, 3 Dibromo-4-Biphenyl acetic acid

Dissolved 4-BPAA (1gm) in glacial acetic acid (25 ml) and add bromine (1ml) of brmine in (1 ml) of acetic acid under stirring on magnetic stirrer for half and hour in ice-cool medium, then add ice-cool water (100ml) in the reaction mixture. Light yellow crystalline solid separated out, stirred the reaction mixture again for 15 minutes at room temperature and filter through the buchnel funnel.

2nd Step :- Preparation of 3, 3' – Dibromo-4 Biphenyl – acety1 chloride (1 $_B$) from 3, 3' – Dibromo-4 Biphenyl-acetic acid (1).

3, 3'- Dibromo-4-biphenyl acetic acide (1) (500mg) dissolved in dry benzene (100ml) then add thionyl chloride (0.5 ml) dropwise alongwith stirring in a R.B., flask of 50ml then reflux the reaction This scheme is clear from following diagramatic representation.



mixture for 2 ½ hours on water bath at 78-80^{0}C. Then recovered benzene and thionyl chloride from reaction mixture through distillation traces of solvent through vaccum cump. 3, 3' Dibromo-4-biphenyl acetyl. chloride obtained as a viscous oil.

3rd Step :- Preparation of 3, 3'-Dibromo-N-phenyl – 4 – biphenyl acetamide (12_A) from 3, 3'-Dibromo-4-Biphenyl acetyl chloride (1_B).

Dissolved Anilline (250mg) in benzene (10ml) in a R.B. flask and add 4N-NaOH in it. Take 1_B (542 mg) and dissolved it in dry benzene (10ml), then pour it slowly drop wise under stirring in the R.B. flask. Stirring continue for 3 hours and workup the reaction mixture after 20 hours.

The same procedure were synthesized Dibromo 4-Biphenyl acetamide derivative such as –

A_1 - (3, 3' Dibromo – N – Phenyl – 4 Biphenyl acetamide)
A_2 - (3, 3' Dibromo – N – Phenyl – 4 Biphenyl acetamide)
A_3 - (3, 3' Dibromo – N – P – Tolune – 4 - Biphenyl acetamide)
A_4 - (3, 3' Dibromo – N – a – Naphthyl– 4 Biphenyl acetamide)
A_5 - (3, 3' Dibromo – N – Phenyl – Thiomide – 4 Biphenyl acetamide)
A_6 - (3, 3' Dibromo – N – Benzyl – 4 Biphenyl acetamide)

Result and Discussion

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 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.

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. These compounds have tested for their antibacterial activity.

The compounds A1, A2 & A3 showed high activity, while other A4, A5& A6 compounds showed less activity against the above organism.

RESULTS CAN BE SUMMERISED AS GIVEN BELOW

S.No.	Code	Experimental yield (100%)	Yield obtained (%)
1.	A_1	570mg	270mg (43.36%)
2	A_2	480 mg	290mg(60.42%)
3	A_3	585 mg	470mg (80.34%)
4	A_4	630 mg	570mg (90.48%)
5	A_5	640 mg	500mg (78.12%)
6	A_6	880 mg	780gm (88.64%)

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