Studies on Novel Bisaryl hydrazino-s-triazine Derivatives

¹J.A.CHAUDHARI*, ²M.V.HATHI and ³R.P.PATEL

¹Shri R.K.Parikh Arts & Science College, Petlad-388450. GUJARAT

²R.R.Mehta College of Sci.& C.L.Parikh college of Commerce
Palanpur-385001. GUJARAT

³C.U.Shah Science College, Ahmedabad-380014. GUJARAT. India

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Abstract: Various 2-phenyloxy-4,6-bisarylhydrazino-1,3,5-triazines(3a-f) were prepared by reaction of 2- phenyloxy -4,6-dichloro -1,3,5-triazine and various aryl hydrazine derivatives. All the 3a-f derivatives were characterized by elemental analysis and IR spectral studies. All the compounds were screened for microbial activity against gram-positive and gram-negative bacteria.

Keywords: Phenyloxy bisaryl hydrazino s-triazine derivatives, hydrazines, IR & spectral study, antimicrobial activity.

Introduction

Number of derivatives containing s-triazine ring have been reported as hetrocyclic compounds ¹. They are applicable mostly as reactive dyes and some are used as polymers and drugs ^{2,3}. The aryl hydrazine derivatives containing s-triazine ring are not reported so far except one instance ⁴. Recently our university scientists have studied the hydrazine triazine clubbed molecules having alkoxy group ⁴. Hence in continuous of this work ⁴, the two of the authors(RPP,MVH) have reported the aryl hydrazine derivatives of based on 2-phenyloxy-4,6-dichloro -1,3,5-triazine ⁵. In continuous of this work ⁵ and the reports about the heterocyclic s-triazine derivatives ²⁻⁴ the present paper comprises the work shown in Scheme-1.

Experimental

Materials

Cyanuric chloride and all the aryl hydrazine derivatives (substitution shown in Scheme-1) were obtained as Analar grade from local dealer. 2-phenyloxy-4,6-dichloro-1,3,5-triazine was prepared by reported method⁴. All other chemicals used were of laboratory grade.

Synthesis of bisaryl hydrazino-s-triazines

2-phenyloxy-4,6-bisaryl hydrazino-1,3,5-triazines (3a-f). The general procedure for these compounds is as follows.

To a well stirred solution of 2-phenyloxy-4,6-dichloro-1,3,5-triazine (0.01 mole) in tetrahydrofuran (THF) solvent (50ml) a solution of aryl hydrazine (or substituted aryl hydrazine) (scheme-1) (0.02mole) in THF (50ml) was added gradually at room temperature. The mixture was stirred for two hours. Then the mixture was refluxed for further two hours. The resultant solid product was filtered, washed with THF and air-dried. All the (3a-f) compounds listed in Table-1 are dark yellow amorphous powders. Their melting points shown in Table-1 and they are uncorrected.

Structure - 1

Characterization of 2-Phenyloxy -4,6- bisaryl hydrazino -1,3,5-triazine (3a-f)

Ar = Shown in scheme -1

Table 1

Compound	Molecular Formula	Mol. Weight	% C		% H		% N	
			Cald.	Found	Cald.	Found	Cald.	Found
3-a	C ₂₁ H ₁₉ N ₇ O	385	65.45	65.4	4.93	4.8	25.45	25.4
3-b	C ₂₁ H ₁₇ N ₇ Cl ₂ O	454	55.51	55.4	3.74	3.6	21.59	21.5
3-c	C ₂₁ H ₁₇ N ₇ Br ₂ O	543	46.41	46.3	3.13	3.0	18.05	18.0
3-d	C ₂₃ H ₂₃ N ₇ O	413	66.83	66.7	5.57	5.5	23.73	23.6
3-e	C ₂₃ H ₂₃ N ₇ O ₃	445	62.02	61.9	5.17	5.0	22.02	21.9
3-f	C ₂₁ H ₁₇ N ₉ O ₅	475	53.05	52.9	3.58	3.5	26.53	26.4

Where, ArNHNH_2 :

Mesurements

The elemental analysis of all (3a-f) compounds were determined by TF flash EA. The FT-IR spectra of (3a-f) compounds were scanned in KBr pallets on Perkin Elmer FT-IR spectrophotometer. The NMR spectra of soluble sample No.3e was scanned on Perkin Elmer FT-NMR spectrophotometer.

Antimicrobial Activity

For the testing antimicrobial activity various microorganism were used for the study. The pour plate agar method was used for this study. Following general procedure is adopted⁷. The antimicrobial activity of all the compounds was studies at 1000 ppm concentration in vitro. The different types of microorganism used were some gram negative bacteria [*Escherichia coli*, *Proteus vulgaris*], gram positive bacteria [*Bacillus cereus*, *Streptococcus species*], fungi [*Aspergillus oryzae*], yeast [*Pichia species*] and actinomycetes [*Streptomyces coelicolor*].

The antibacterial activity of 3a-f compounds was measured on each of these microorganism strains on a potato dextrose agar medium (PDA). Such a PDA medium contained ⁵ potato 200gram, dextrose 20gram, agar 30gram, and water 1 Litre. PDA medium autoclaved at 121°C temperature and 15 lbs pressure for 15 minute. After autoclaving the compounds to be tested were inoculated (1000 ppm) in PDA medium at 42°C temperature and mix it than these media were poured in to sterile empty glass petriplates. The testing microorganism [young culture] were inoculating after solidification of the PDA medium plates. The percentage inhibition of growth of microorganism was calculated after 5 days of incubation of PDA medium plate inoculated with microorganism at appropriate temperature [Bacteria- 37°C, Fungi- 25°C, Actinomyces- room temp.] percentage of inhibition of microorganism was calculated by using the formula given below.

$$\text{Percentage of inhibition of growth of microorganism} = \frac{100(X-Y)}{X}$$

Where, X= area of bacterial growth in control plate (mm),

Y = area of bacterial growth in test plate (mm).

The antimicrobial activity of all the 3a-f compounds are furnished in Table-2

Table 2. Antimicrobial activity of 2 -Phenyloxy -4,6- bisaryl hydrazino -1,3,5- triazines (3a to f)

Sample	Percentage of inhibition of growth at 1000 ppm (%) concentrate of sample						
	<i>Bacillus cereus</i>	<i>Streptococcus species</i>	<i>Escherichia coli</i>	<i>Proteus vulgaris</i>	<i>Aspergillus oryzae</i>	<i>Pichia species</i>	<i>Streptomyces coelicolor</i>
Control	Nil	Nil	Nil	Nil	Nil	Nil	Nil
3-a	65	95	80	80	65	69	60
3-b	100	98	97	96	98	97	95
3-c	71	91	100	78	65	71	60
3-d	73	85	72	55	62	69	60
3-e	86	85	95	68	60	68	65
3-f	69	69	99	62	65	66	59

Results and Discussion

The reaction between aryl hydrazine and 2-phenyloxy-4,6-dichloro-1,3,5-triazine is facile. The products (3a-f) are dark yellow amorphous powders. The C,H,N contents of all (3a-f) shown in Table-1 are consistent with the predicated structures shown in Scheme-1. the IR spectra of all (3a-f) are almost identical. All the IR spectra comprises following important features.

1. –NH–NH– (hydrazine group) : 3280,1610,820 cm^{-1}
2. s-triazine : 1510,1250,870 cm^{-1}
3. –NH– (secondary) : 3400 cm^{-1}
4. Aryl–O–Aryl : 1200 cm^{-1}

As the compounds (3a-f) except 3e are insoluble CDCl_3 , the NMR spectral study attempted for 3e. The NMR spectrum of 3e comprises the multiplate between 6.9 to 8.1 δ ppm mainly due to aromatic protons. While the signal at 2.6 δ ppm with integration of 6H is responsible for two CH_3 of OCH_3 groups. The signals in most downfield (9.5 δ ppm) is from NH–NH protons. The result of anti microbial screening showed (Table-2) that compounds 3b,c,f displayed a high order of antibacterial activity and remaining compounds showed weak to moderate activity against both the bacteria. Similarly compounds 3a,d, and e showed higher antifungal activity and remaining compounds displayed moderate antifungal activity against both the fungi.

Conclusion

The synthesis of arylhydrazino –s-triazine is facile. The produced compounds have good microbial toxicity. Due to NH–NH groups these compounds can be utilized for epoxy resin hardner. Such work in polymer journal will be published shortly.

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