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Studies on 1,3,4-Oxadiazoles. Preparation and Antimicrobial Activity of 2-Aryl-5-(5',7'-diiodo-8'-quinolinoxy)-1,3,4-oxadiazoles

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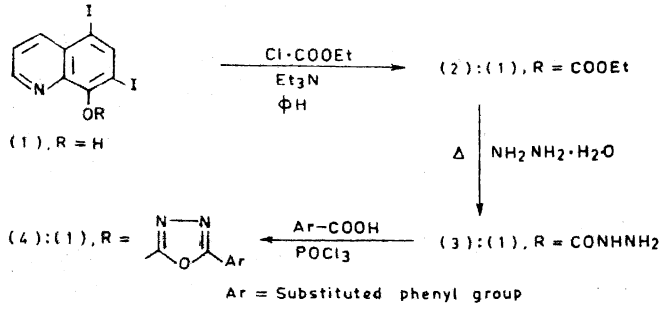
1,3,4-Oxadiazole derivatives possess diverse biological activities and there are numerous reports that highlight their chemistry and use[1]. With a view to getting newer oxadiazoles with better therapeutic activity we have synthesised compounds **4** bearing 5,7-diiodo-8-hydroxyquinolinyl moiety.

5,7-Diiodo-8-hydroxyquinoline (**1**) was condensed with ethyl chloroformate to get ethyl (5,7-diiodoquinolin-8-yl-oxy) formate (**2**), which was further treated with hydrazine hydrate to obtain the corresponding hydrazide (**3**). It was then condensed with different aromatic acids in presence of phosphorous oxychloride to yield various 1,3,4-oxadiazoles (**4**). The structures of the products have been characterised by elemental analyses and ir spectral study. The compounds were screened for their antimicrobial activity.



Antimicrobial activity : The compounds were screened for antibacterial and antifungal activity using cup-plate method[2]. The testing was carried out at a concentration of 100 μg using gram-positive bacteria *Staphylococcus aureus* and *S. citrus*, gram-negative bacteria *Escherichia coli* and *Mercsane serratia* and fungi *Aspergillus niger* and *Saccharomyces cerrevisiae*. Most of the compounds were found moderately active (13–24 mm zone of inhibition) against the aforesaid strains of bacteria and fungi.

Experimental

All melting points were determined in open capillaries and are uncorrected. The ir spectra (KBr) were taken on a Shimadzu DR-1, 435 spectrophotometer.

5,7-Diiodoquinolin-8-oxynoylhydrazide (**3**) : A mixture of **2** (0.01 mol, 4.69 g) in dioxane (35 ml) and hydrazine hydrate (0.02 mol, 1.04 g) was refluxed at 100–02° for 3 h. The resulting solid was crystallised from ethanol, (80.0%), m.p. 201–03° (Found : C, 26.32 ; H, 1.50 ; N, 9.20. $C_{10}H_7I_2N_3O_2$ calcd. for : C, 26.37 ; H, 1.53 ; N, 9.23%) ; ν_{max} 3 400 (NH), 1 680 (C=O), 770 (NH wag) and 630 cm^{-1} (C–I).

Preparation of **4** : Benzoic acid (0.05 mol, 0.61 g) was refluxed with the hydrazide (**3** ; 0.05 mol, 2.28 g) in presence of phosphorus oxychloride (0.05 mol) for 5 h. The contents were then poured into water and basified with sodium bicarbonate solution. The resulting solid was crystallised from ethanol, (Ar=Ph ; 60.0%), m.p. 173° (Found : C, 37.62 ; H, 1.62 ; N, 7.72. $C_{17}H_9I_2N_3O_2$ calcd. for : C, 37.70 ; H, 1.66 ; N, 7.76%) ; ν_{max} 1 590 (C=N), 1 130 (C–O–C), 1 040 (N–N) and 650 cm^{-1} (C–I).

Similarly, other substituted oxadiazoles were prepared (Table 1).

TABLE 1—PHYSICAL DATA OF THE OXADIAZOLES (**4**)

Sl. no.	Ar	Mol. formula	M.p. °C
1.	Phenyl	$C_{17}H_9I_2N_3O_2$	173
2.	*o*-Chlorophenyl	$C_{17}H_8ClI_2N_3O_2$	224
3.	*m*-Chlorophenyl	$C_{17}H_8ClI_2N_3O_2$	218
4.	*p*-Chlorophenyl	$C_{17}H_8ClI_2N_3O_2$	202
5.	*o*-Hydroxyphenyl	$C_{17}H_9I_2N_3O_3$	280
6.	*m*-Hydroxyphenyl	$C_{17}H_9I_2N_3O_3$	208
7.	*p*-Hydroxyphenyl	$C_{17}H_9I_2N_3O_3$	255
8.	*m*-Nitrophenyl	$C_{17}H_8I_2N_4O_4$	258
9.	*p*-Nitrophenyl	$C_{17}H_8I_2N_4O_4$	195
10.	*o*-Aminophenyl	$C_{17}H_{10}I_2N_4O_2$	189
11.	*m*-Aminophenyl	$C_{17}H_{10}I_2N_4O_2$	231
12.	*p*-Aminophenyl	$C_{17}H_{10}I_2N_4O_2$	256
13.	2,4-Dihydroxyphenyl	$C_{17}H_9I_2N_3O_4$	170
14.	2,6-Dihydroxyphenyl	$C_{17}H_9I_2N_3O_4$	184
15.	Cinnamyl	$C_{19}H_{11}I_2N_3O_2$	220
16.	*p*-Pyridyl	$C_{17}H_9I_2N_4O_4$	235
17.	*o*-Acetoxyphenyl	$C_{19}H_{11}I_2N_3O_4$	247
18.	3,4,5-Trihydroxyphenyl	$C_{17}H_9I_2N_3O_5$	186

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Studies on Thiadiazole Derivatives. Part-III. Preparation and Antimicrobial Activity of *p,p'*-Bis(2-substituted-benzalamino/benzoylamino/sulphonamido-1,3,4-thiadiazol-5-ylmethylamino)diphenyl Sulphones

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DAPSONE is a useful chemotherapeutic agent[1]. In view of the wide spectrum of activity associated with thiadiazole derivatives[2], it was planned to synthesise thiadiazole derivatives bearing dapsone moiety. *p,p'*-Bis (2-substituted-benzal / benzoyl/sulphonamido - 1, 3, 4 - thiadiazol - 5 - ylmethylamino) - diphenyl sulphones were synthesised by the condensation of different aromatic aldehydes, acid chlorides and sulphonyl chlorides with *p,p'*-bis(2-amino-1,3,4-thiadiazol-5-yl-methylamino)diphenyl sulphone. The latter was prepared by the condensation of thiosemicarbazide with *p,p'*-bis(carboxymethylamino)diphenyl sulphone in presence of phosphorous oxychloride. Chloroacetic acid was condensed with *p,p'*-diamino diphenyl sulphone in presence of alkaline medium to get *p,p'*-bis(carboxymethylamino)diphenyl sulphone. The structural assignments of the products were based on their elemental analyses, ir, nmr and mass spectral data. The products were screened for their antimicrobial activity.

Antimicrobial activity : The antimicrobial screening of thiadiazole derivatives was carried out using cup-plate method[3] at a concentration of 50 μg using gram-positive bacteria *B. mega* and *B. saphilis*, gram-negative bacteria *Escherichia coli* and *Pseudomonas floures* and fungus *Aspergillus niger*.

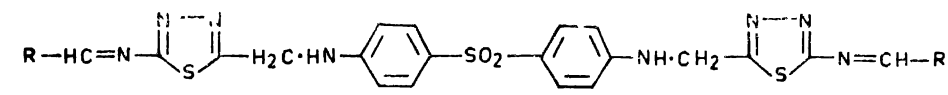
Most of the compounds were found moderately active against different strain of bacteria and fungi (10 – 20 mm zone of inhibition). The maximum activity was observed in compounds bearing 3-aminophenyl, 3-hydroxy, 4-carboxyphenyl and 3-chlorophenyl groups against *B. saphilis*, and 4-acetamidophenyl and 3-nitrophenyl groups against *B. mega*.

Experimental

Melting points of the compounds were determined in open capillary tubes and are uncorrected. Ir spectra (KBr) were recorded on a Shimadzu DR-1 435-IR spectrophotometer.

p,p'-*Bis(carboxymethylamino)diphenyl sulphone :* *p,p'*-Bis(diaminodiphenyl sulphone (0.01 mol, 2.48 g) and monochloroacetic acid (0.02 mol, 1.89 g) was condensed at 110° in presence of 15% NaOH solution for 12 h. The contents were poured into ice-water and the resulting solid was crystallised from ethanol, (2.44 g, 67%), m.p. 102° (Found : C, 57.89 ; H, 4.79 ; N, 11.20. $C_{16}H_{16}N_2O_6S$ requires : C, 58.04 ; H, 4.87 ; N, 11.28%) ; ν_{max} (KBr) 1 145, 1 290 cm^{-1} (S=O asym, sym), 1 680 cm^{-1} (C=O), 3 300 (NH), and 3 450 cm^{-1} (OH).

p,p'-*Bis (2-amino-1, 3, 4-thiadiazol-5-yl-methylamino)diphenyl sulphone*[4] : A mixture of *p,p'*-bis(carboxymethylamino)diphenyl sulphone (3.64 g, 0.01 mol), thiosemicarbazide (1.82 g, 0.02 mol) and phosphorous oxychloride (1.87 g, 0.02 mol) was heated at 60° for 1 h and then at 90 – 95° for 1.5 h.



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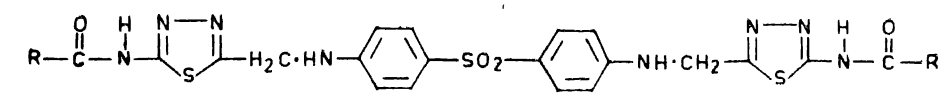

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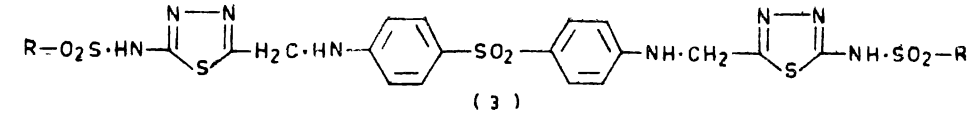

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