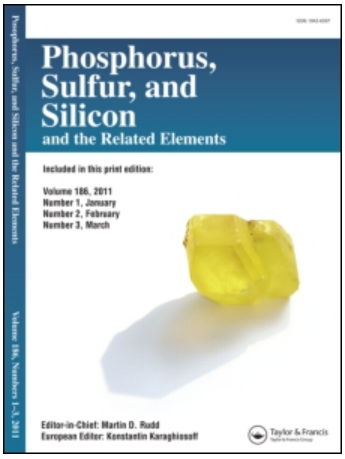

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A Comparative Study on Some Diaryl Azines Amino Acid Derivatives

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A Comparative Study on Some Diaryl Azines Amino Acid Derivatives

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*Benzaldehydeazines, p-anisaldehydeazines, and thiophene-2-carboxaldehyde azines react with excess chlorosulfonic acid to give corresponding disulfonyl chlorides **1**,**13**, and **24**. These were condensed with nucleophiles to give disulfonyl amino acid derivatives, **2–4**, **14–16**, and **25–27**. Some of the corresponding methyl esters were prepared: **5–6**, **17–18**, and **28–29**. Hydrazinolysis of these methyl esters yielded hydrazides **7–8**, **19–20**, and **30–31**. Coupling reactions of some amino acid derivatives, in **THF-Et$_3$N** medium using the dicyclohexylcarbodiimide method **DCC**, furnished dipeptide methyl esters **9–12**, **21–23**, and **32–34**. Attemped chlorosulfonation of furan-2-carboxaldehyde azine were unsuccessful. Some spectra data are briefly discussed.*

Keywords Benzaldehyde azines; chlorosulfonation of diarylazines; p-anisaldehyde azines; thiophene-2-carboxyaldehyde azines; and their reactions with essential amino acids

INTRODUCTION

The work reported here is a continuation of our general program on the chemistry and reactivity of aryl sulfonyl derivatives as candidate pesticides, which are found to possess hypoglycemic, antipyretic, analgesic diuretic, bacteriostatic, and other pharmacological activities. The compounds mentioned were found to have these activities.[1–12]

Diarylazines are known[13] to be readly formed by condensation of the appropriate aryl aldehyde and hydrazine hydrate. However, the chlorosulfonation of these compounds has been previously reported. In view of the known ability of diaryl azines to undergo 1,3-dipolar cycloaddition reactions with maleic acid derivatives.

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Sulfonyl chlorides **1**,**13**, and **24** by condensation with nucleophiles, e.g., amino acids residue, can be converted into sulfonylamino acid derivatives for biological evaluation as candidate biocides.

DISCUSSION

In diaryl azines, the C=N group is a deactivating substituent with regard to electrophilic substitution. In agreement, (Comp. **1**, R = X = H) were prepared by reaction of chlorosulfonic acid with benzaldehyde azine in a 68% yield.

The N—N bond does not undergo fission under the reaction conditions adopted, and the imino group was deactivated to electrophilic attack. The ^{1}H NMR spectrum contained an unsymmetrical aromatic region.

Other azines were treated with chlorosulfonic acid, p-anisaldehyde azines, and (Comp. **13**, R = P-OMe, X = H) accured under milder conditions than for benzaldehyde azine (Comp. **1**, R = X = H). In thiophene carboxaldehyde azines, (Comp. **24**, Y = S, X = H) the more electron rich azines react with chlorosulfonic acid, the greater reactivity of the thiophene ring as a result from electron donation from the hetero sulfur atom, which is known.[14] Generally, an electron–donner substituent gives direct electrophilic substition at 5-position of the thiophene ring. The ^{1}H NMR spectrum of disulfonylamino acids derivatives showed an AB pattern in the aromatic resonance at (δ **7.8**, **7.7**).

In the case of furan-2-carboxyladehydeazines (Comp. **35**, Y = O, X = H) powerful electron donation from the hetero oxygen atom. The **^{1}H NMR** spectrum showed a complex series of aromatic resonace at (δ **7.3–6.8**).

Furan is well known[15], to decompose with highly acidic sulfonation reagents, chlorosulfonic and sulfuric acid; the electron-withdrawing C=N group might sufficiently stabilize the furan ring to allow sulfonation, as was observed with furan-2-carboxamide,[16] and carboxanilide.[17]

EXPERIMENTAL

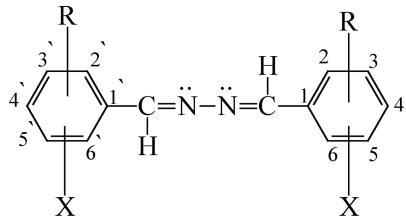
Melting points were taken on a Griffin melting point apparatus and are uncorrected. Infrared solid samples were run as a KBr disc on a Schimadzu model 440 spectrophotometer. ^{1}H NMR spectra were measured in DMSO-d_6 as a solvent unless otherwise stated using Fx 90 Q Furier Transform ^{1}HNMR. Mass spectra were obtained using a Schimadzu (Japan). GC. M.S. QP 1000 Ex spectrometer using the direct inlet system. TLC analyses were carried out on Merek silica gel plates and

developed with n-butanol-acetic acid-water (4:1:1) using iodine, ninhydrin, and benzidine as spraying agents.

Diaryl azines disulfonyl chloride **1**,**13**, and **24** were prepared according to the procedure described earlier.[18]

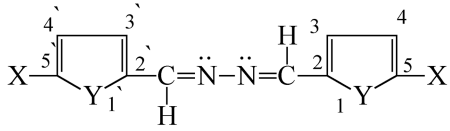
Coupling Reaction 2–4, 14–16, and (25–27): General Procedure

To an amino acid (0.2 mol) in a water (25 mL) THF (15 mL) mixture was added triethylamine (5 mL), followed by the portionwise addition of disulfonyl chlorides (0.11 mol) during 30 min. The temperature of the reaction mixture during the process of addition was kept at 10°C. Stirring continued for 4 h at 20°C. Tetrahydrofuran was removed by concentration of the reaction mixture under reduced pressure, and water (50 mL) was added and acidified with 2M HCl to pH_5. The crude products were filtered and recrystallized (ethanol-water). All products



Parent azines 1,13

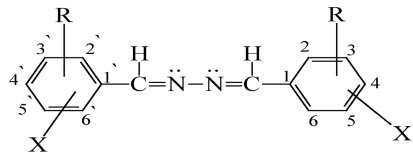
Comp. **1**, R = H, X = 3,3` SO_2Cl
Comp. **13**, R = 4-OMe, X = 3,3` SO_2Cl



Parent azines 24

Comp. **24**, Y = S, X = 5,5` SO_2Cl
Comp. **35**, Y = O, X = 5,5` SO_2Cl (Unsuccessful)

CHART 1 *(Continued)*



Parent azines 1, R = H, X = 3,3` SO_2Cl

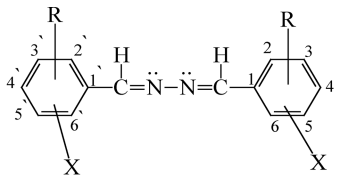
Comp.	R	X
2	H	SO_2–NH–CH(–CH_3)–COOH
3	H	SO_2–NH–CH(–CH–$(CH_3)_2$)–COOH
4	H	SO_2–NH–CH(–CH_2–CH–$(CH_3)_2$)–COOH
5	H	SO_2–NH–CH(–CH_3)–$COOCH_3$
6	H	SO_2–NH–CH(–CH_2–CH–$(CH_3)_2$)–$COOCH_3$
7	H	SO_2–NH–CH(–CH_3)–CON_2H_3
8	H	SO_2–NH–CH(–CH_2–CH–$(CH_3)_2$)–CON_2H_3
9	H	SO_2–Glycyl–Glycine
10	H	SO_2–Glycyl–L–Alanine
11	H	SO_2–Glycyl–L–Valine
12	H	SO_2–Glycyl–L–Leucine

CHART 1 (Continued)

2–4, **14–16**, and **25–27** were chromatographically homogeneous by iodine and benzidine development (cf. Charts 1, 2, and 3 and Table I).

IR of **2**: ν3350 cm^{-1} (NH), ν1660 cm^{-1} (C=O), ν 1580 cm^{-1} (C=N), ν 1600 cm^{-1} (Ar–C=C), ν1380, 1170 cm^{-1} (SO_2)

IR of **14**: ν 3270 cm^{-1} (NH), ν 1640 cm^{-1} (Co), ν 1590 cm^{-1} (SO_2), ν 1270, 1030 cm^{-1} (Ar–O–CH_3)



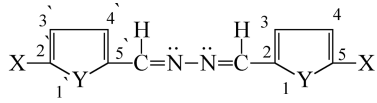
Parent azines 3 and 13 **R = P–OMe,** **X = 3,3` SO_2Cl**

Comp.	R	X
14	4–OMe	SO_2–NH–CH(–CH_3)–COOH
15	4–OMe	SO_2–NH–CH(–CH–$(CH_3)_2$)–COOH
16	4–OMe	SO_2–NH–CH(–CH_2–CH–$(CH_3)_2$)–COOH
17	4–OMe	SO_2–NH–CH(–CH_3)–$COOCH_3$
18	4–OMe	SO_2–NH–CH(–CH_2–CH–$(CH_3)_2$)–$COOCH_3$
19	4–OMe	SO_2–NH–CH(–CH_3)–CON_2H_3
20	4–OMe	SO_2–NH–CH(–CH–$(CH_3)_2$)–CON_2H_3
21	4–OMe	SO_2–Glycyl–Glycine
22	4–OMe	SO_2–Glycyl–L–Alanine
23	4–OMe	SO_2–Glycyl–L–Valine

CHART 2

IR of **25**: ν 1610 cm^{-1} (Ar.C=C), ν 1360, 1160 cm^{-1} (SO_2)

^{1}H NMR of **2**: (DMSO-d_6): δ 1.2 (s, 6H, 2$\underline{CH_3}$-alalyl), δ 4.2 (s, 2H, 2$\underline{CH}$), δ 8.8 (s, 2H, 2$\underline{CH}$=N), δ 7.8 (s, 2H, 2N$\underline{H}$), δ 8.4-7.6 (s, 8H, Ar—H), δ 9.8, (s, 2H, 2COOH), MS of 4 : m/z 594 (M^+)



Parent azines 24, Y = S, X = 5,5` SO_2Cl
Parent azines 35, Y = O, X = 5,5` SO_2Cl (Unsuccessful)

Comp.	Y	X
25	S	SO_2–NH–CH(CH$_3$)–COOH
26	S	SO_2–NH–CH(CH(CH$_3$)$_2$)–COOH
27	S	SO_2–NH–CH(CH$_2$–CH(CH$_3$)$_2$)–COOH
28	S	SO_2–NH–CH(CH$_3$)–COOCH$_3$
29	S	SO_2–NH–CH(CH(CH$_3$)$_2$)–COOCH$_3$
30	S	SO_2–NH–CH(CH$_3$)–CON$_2$H$_3$
31	S	SO_2–NH–CH(CH(CH$_3$)$_2$)–CON$_2$H$_3$
32	S	SO_2–Glycyl–Glycine
33	S	SO_2–Glycyl–L–Alanine
34	S	SO_2–Glycyl–L–Valine

CHART 3

^{1}H NMR of **15**: (DMSO-d_6): δ 0.98 [s, 12H, $(CH_3)_2$], δ 1.97 (s, 2H, 2β CH Valyl), δ 4.26 (s, 2H, α CH Valyl), δ 4.33 (s, 6H, 2O-CH_3), δ 7.88 (s, 2H, 2NH), δ 8.4–7.6 (s, 8H, ArH), δ 8.8 (s, 2H, CH═N), δ 11.3 (s, 2H, COOH)
MS of 15 : m/z 626 (M^+)

TABLE I Physical Data for Diarylazines Derivatives 2–12, 14–23, and 25–34

Compound no.	X	M.P.°C	Yield %	R_f	Molecular formula	Elemental analysis % Calculated/found			
						% C	% H	% N	% S
2	DL–Ala	268–270	71	0.81	$C_{20}H_{22}N_4O_8S_2$	47.06	4.31	10.98	12.55
						47.00	4.22	10.91	12.41
3	L–Val	310–312	74	0.80	$C_{24}H_{30}N_4O_8S_2$	50.88	5.30	9.89	11.31
						50.73	5.21	9.81	11.22
4	L–Leu	263–265	68	0.76	$C_{26}H_{34}N_4O_8S_2$	52.53	5.72	9.43	10.77
						52.50	5.63	9.40	10.60
5	DL–Ala–OMe	208–210	64	0.76	$C_{22}H_{26}N_4O_8S_2$	49.07	4.83	10.41	11.90
						49.00	4.81	10.33	11.83
6	L–Leu–OMe	110–112	79	0.79	$C_{28}H_{38}N_4O_8S_2$	54.02	6.11	9.00	10.29
						54.00	6.00	8.91	10.21
7	DL–Ala–N_2H_3	194–196	60	0.61	$C_{20}H_{26}N_8O_6S_2$	44.61	4.83	20.82	11.90
						44.53	4.75	20.78	11.88
8	L–Leu–N_2H_3	165–167	54	0.69	$C_{26}H_{38}N_8O_6S_2$	50.16	6.11	18.00	10.29
						50.01	6.01	17.90	10.18
9	Gly–Gly–OMe	183–185	80	0.84	$C_{24}H_{28}N_6O_{10}S_2$	46.15	4.49	13.46	10.26
						46.11	4.40	13.38	10.10
10	Gly–DL–Ala–OMe	187–189	76	0.86	$C_{26}H_{32}N_6O_{10}S_2$	47.85	4.91	12.88	9.82
						47.77	4.88	12.80	9.70
11	Gly–L–Val–OMe	173–175	74	0.88	$C_{30}H_{40}N_6O_{10}S_2$	50.85	5.65	11.86	9.04
						50.80	5.60	11.83	9.00
12	Gly–L–Leu–OMe	205–207	71	0.90	$C_{32}H_{44}N_6O_{10}S_2$	52.17	5.98	11.41	8.70
						52.00	5.81	11.40	8.70
14	DL–Ala	218–220	70	0.70	$C_{22}H_{26}N_4O_{10}S_2$	46.32	4.56	9.82	11.23
						46.20	4.44	9.75	11.10

(*Continued on next page*)

TABLE I Physical Data for Diarylazines Derivatives 2–12, 14–23, and 25–34 *(Continued)*

Compound no.	X	M.P.°C	Yield %	R_f	Molecular formula	Elemental analysis % Calculated /Found			
						% C	% H	% N	% S
15	L—Val	237–239	75	0.83	$C_{26}H_{34}N_4O_{10}S_2$	49.84	5.43	8.95	10.22
						49.70	5.33	8.95	10.01
16	L—Leu	208–210	73	0.77	$C_{28}H_{38}N_4O_{10}S_2$	51.38	5.81	8.56	9.79
						51.30	5.78	8.49	9.79
17	DL—Ala—OMe	178–180	83	0.64	$C_{24}H_{30}N_4O_{10}S_2$	48.16	5.02	9.36	10.70
						48.00	5.00	9.21	10.61
18	L—Val—OMe	170–172	86	0.83	$C_{28}H_{38}N_4O_{10}S_2$	51.38	5.81	8.56	9.79
						51.30	5.78	8.41	9.61
19	DL—Ala—N_2H_3	128–130	54	0.63	$C_{22}H_{30}N_8O_8S_2$	44.20	5.02	18.73	10.70
						44.10	5.00	18.71	10.60
20	L—Val—N_2H_3	77–79	71	0.69	$C_{26}H_{38}N_8O_8S_2$	47.71	5.81	17.13	9.79
						47.62	5.73	17.00	9.65
21	Gly—Gly—OMe	95–97	68	0.75	$C_{26}H_{32}N_6O_{12}S_2$	45.61	4.68	12.28	9.36
						45.52	4.61	12.01	9.30
22	Gly—L—Ala—OMe	80–82	58	0.63	$C_{28}H_{36}N_6O_{12}S_2$	47.19	5.06	11.80	8.99
						47.00	5.00	11.63	8.79
23	Gly—L—Val—OMe	110–112	54	0.66	$C_{32}H_{44}N_6O_{12}S_4$	50.00	5.73	10.94	8.33
						49.85	5.60	10.90	8.31
25	DL—Ala	183–185	65	0.86	$C_{16}H_{18}N_4O_8S_4$	36.78	3.45	10.73	24.52
						36.66	3.41	10.61	24.44
26	L—Val	205–207	67	0.84	$C_{20}H_{26}N_4O_8S_4$	41.52	4.50	9.69	22.15
						41.50	4.44	9.61	22.00
27	L—Leu	212–214	77	0.82	$C_{22}H_{30}N_4O_8S_4$	43.56	4.95	9.24	21.12
						43.51	4.81	9.10	21.00
28	DL—Ala—OMe	138–140	48	0.73	$C_{18}H_{22}N_4O_8S_4$	39.27	4.00	10.18	23.27
						39.11	3.92	10.10	23.11

29	L—Val—OMe	178–180	42	0.75	$C_{22}H_{30}N_4O_8S_4$	43.56	4.95	9.24	21.12
						43.44	4.81	9.10	21.01
30	DL—Ala—N_2H_3	122–124	60	0.77	$C_{16}H_{22}N_8O_6S_4$	34.91	4.00	20.36	23.27
						34.90	3.94	20.11	23.11
31	L—Val—N_2H_3	154–156	64	0.79	$C_{20}H_{30}N_8O_6S_4$	39.60	4.95	18.48	21.12
						39.56	4.81	18.33	21.00
32	Gly—Gly—OMe	133–135	61	0.80	$C_{20}H_{24}N_6O_{10}S_4$	37.74	3.77	13.21	20.13
						37.63	3.71	13.10	20.00
33	Gly—L—Ala—OMe	120–122	70	0.84	$C_{22}H_{28}N_6O_{10}S_4$	39.76	4.22	12.65	19.28
						39.66	4.20	12.55	19.10
34	Gly—L—Val—OMe	143–145	73	0.87	$C_{26}H_{36}N_6O_{10}S_4$	43.33	5.00	11.67	17.79
						43.10	4.91	11.61	17.66

^{1}H NMR of **26**: (DMSO-d_6): δ 4.1 [s, 2H, 2C<u>H</u>], δ 4.5 (s, 4H, 2<u>CH_2</u>), δ 4.0 (s, 6H, 2O—CH_3), δ 7.8–7.7 (s, 4H, 2 thiphene), δ 8.3–7.17 (s, 6H, 2Ar—H), δ 8.68 (s, 2H, 2CH=N), δ 11.3 (s, 2H, 2COOH), MS of 27 : m/z 606 (M^+)

Synthesis of Disulfonylamino Acid Methyl Esters 5, 6, 17, 18, 28, and 29: General Procedure

A suspension of coupling reaction products **2–4**, **17, 18**, and **28** and **29** (0.2 mole) in absolute methanol (100 mL) was cooled to –10°C, and pure thionyl chloride (2.2 mL) was added dropwise during 1 h. The reaction mixture was stirred for an additional 3–4 h at r.t. It was kept overnight, and the solvent was removed by vacuum distillation. The residual solid material was recrystallized (methanol-water) (cf. Table I, and Charts 1, 2, and 3).

IR of **17**: ν 3460cm^{-1} (NH), ν3250, 1370, 1170 cm^{-1} (SO_2-NH), ν 1445, 1360 cm^{-1} ($COOCH_3$), ν 2960 cm^{-1} (O–CH_3), ν 1760 cm^{-1} (<u>Co</u>), ν 1310, 1160 cm^{-1} (SO_2)

^{1}H NMR of **18**: (DMSO-d_6): δ 8.34–7.17 (s, 8H, Ar-H), δ 3.81–3.87 (s, 6H, 2$COOCH_3$), and disappear of OH protons, and other peaks in support of their structures.

Synthesis of Disulfonylamino Acid Hydrazides 7, 8, 19, 20 and 30, 31: General Procedure

The methyl esters **5, 6**, **17, 18** and **28, 29**, (0.2 mol) were dissolved in ethanol (100 ml) and hydrazine hydrate 85% (0.2 mol) was added. The reaction mixture was stirred for 3 h at 20°C and left 24 h at room temperature. The crystalline products **7, 8**, **19, 20** and **30, 31** were filtered off, washed with water and recrystallized (ethanol–water).

The hydrazides **7, 8, 19, 20, 30** and **31** were shown to be chromoatographically to be homogeneous. cf. Table 1, Chart 1, 2 and 3.

IR of **7**: ν 3340, 3125 cm^{-1} (NH), ν 1640, cm^{-1} (Co), ν 1600, 1550 cm^{-1} (Ar—C=C), ν 1340, 1180 cm^{-1} (SO_2)

^{1}H NMR of **20**: (DMSO-d_6): δ 9(s, <u>H</u>, SO_2N<u>H</u>), δ 8.2–7.5 (Ar-H), δ 5.52 (s, 2N<u>H</u>), δ 5.61 (s, 4H, $2NH_2$)

Synthesis of Disulfonyl Dipeptide Methyl Esters 9–12, 21–23, and 32–34: General Procedure

To a solution of amino acid methyl ester hydrochloride (0.01 mol) in THF (100 mL) was added triethylamine (5 mL). The solution was stirred at

20°C for 30 min and cooled to 0°C, where the disulfonyl amino acid (0.005 mol) and dicyclohexylcarbodiimide **DCC** (1.62 g) were added to the above mixture. The reaction mixture was stirred for 2 h at 0°C and for another 2 h at r.t. The precipitated dicyclohexylurea was filtered off, and acetic acid (2 mL) was added to the solution and was left standing overnight. The precipitate was filtered off, and the remaining solution was distilled under vacuum. The remaining solid was recrystallized from ethanol-water. The products were to be chromatographically homogeneous.

IR of **10**: ν 3300, 3100 cm^{-1} (NH, CONH), ν 1750 cm^{-1} (Co), ν 1320 cm^{-1} ($COOCH_3$)

IR of **12**: ν 3390 cm^{-1} (NH), ν 1370, 1170 cm^{-1} (SO_2-NH), ν 1665, 1530, 1280 cm^{-1} (CONH), ν 1445, 1350 cm^{-1} ($COOCH_3$), ν 1760 cm^{-1} (C=O), ν 1610, cm^{-1} (Arc=C), 1380, 1180 cm^{-1} (SO_2)

^{1}H NMR of **10**: (DMSO-d_6): δ 7.86 (s, 2H, $2SO_2\underline{NH}$), δ 8.04 (s, 2H, $2CO\underline{NH}$), δ 3.87 (s, 6H, $2COOCH_3$), 2H, ν 4.34 (s, 2H, 2α-$\underline{CH}$-alalyl), ν 1.22 (s, 6H, CH_3-alalyl) and other bands supporting the structure of dipeptide

^{1}H NMR of **23**: (DMSO-d_6): δ 8.01 (s, 2H, $2CO\underline{NH}$), δ 3.85 (s, 6H, $2COOCH_3$), ν4.26 (s, 2H, 2α-$C\underline{H}$ Valyl), δ 1.79 (s, 2H, 2β CH-Valyl), ν 0.98 [s, 12H, $2(CH_3)_2$] and other bands supporting the structure of dipeptide

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