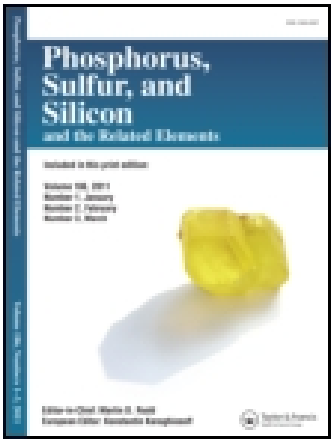

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Some Novel Sulfanilyl Amino Acids and Dipeptides Derivatives

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Some Novel Sulfanilyl Amino Acids and Dipeptides Derivatives

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*3-(N-phenylcarboxamido)-coumarin **1** reacted with chlorosulfonic acid to give the corresponding sulfaniyl chloride **2**. However, isonicotinic acid anilide **14**, and nicotinic acid anilide **33**, reacted with chlorosulfonic acid in a 1:6 molar ratio, only for conversion into sulfanilyl chlorides **15** and **34**. Treatment with nucleophilic reagents afforded amino acid derivatives **3–6**, **16–20**, and **35–37**. Some of the corresponding methyl esters **7–9**, **21**, **24**, and **38–39** were prepared. Hydrazinolysis of some methyl esters yielded hydrazides **25–28** and **40–41**. Coupling reactions of some amino acid derivatives with amino acid methyl ester hydrochloride in **THF-Et_3N** medium using the dicyclohexyl carbodiimide **DCC** furnished the desired dipeptide methyl esters **10–13**, **29–32**, and **42–43**. The spectral properties of the compounds are briefly discussed.*

Keywords Chlorosulfonation of 3-(N-phenyl carboxamidocoumarin; nicotinic acid anilide; isonicotinic acid anilide; and reactions with different essential amino acids

INTRODUCTION

Sulfanilamides are valuable antibacterial drugs.[1] Other sulfanilamides are active as diuretics and against fungal diseases in plants.[2] Earlier investigations have shown that various nicotinic acid and isonicotinic acid derivatives exhibit powerful antimicrobial and antitubercular properties.[3–5] The compounds from the anilide of isonicotinic acid are of special interest in view of the use of isonicotinic acid hydrazide as an antitubercular drug and as a selective herbicide.[6] Several coumarins have biological activities. 3-carboxylic acid derivatives possess sedative and hypotic properties,[7] while 4-hydroxy coumarins are blood anticoagulants, which have been used as rodenticides.[8] The work reported here is a continuation of our general program on the chemistry and reactivity of aryl sulfanilyl amino acid derivatives as candidate pesticides. Some have been found to possess hypoglycemic, antipyretic, analgesis

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diuretic, bacteriostatic, and other pharmacological activities. It has been reported that such compounds have biological activity.[9–21] We decided to prepare some coumarins amino acid derivatives. Coumarin was first sulfonated by Perkin,[22] and Rubtzov and Fedossova,[23] showed that treatment with hot chlorosulfonic acid caused electrophilic substitution to occur preferentially in sulfanilyl chloride.

DISCUSSION

3-(N-phenylcarboxamido)-coumarin **(1)** was prepared by heating ethyl-3-coumarin carboxylate with aniline or by the action of thionyl chloride and aniline on 3-coumarin carboxyhlic acid. When **(1)** was reacted with an excess of chlorosulfonic acid for 7 days at r.t., an excellent yield (85%) of the p-sulfanilyl chloride **(2)** was isolated. The anilino ring was more susceptible to sulfonation than the coumarin nucleus due to a combination of the electron donating effect of the nitrogen lone pair of electrons and the with drawing effect of the amido group. The mass spectra of compound **(2)** showed the molecular ions (M^+, 365, 363) with no trace of higher molecular mass ions confirming the absence of disulfonation. Isonicotinic acid anilide **(14)** required a large excess of chlorosulfonic acid (6 mol) to obtain a good yield of sulfanilyl chloride **15** due to the presence of the basic pyridyl nitrogen atom; on the other hand, nicotinic acid anilide **(33)** with chlorosulfonic acid, under the same conditions, gave a lower yield.

The lower yield of sulfanilyl chloride obtained from nicotinic acid anilide as compared with that obtained from isonicotinic acid anilide is probably due to the more basic character of the nitrogen atom in nicotinic acid anilide, which tends to form a hydrochloride salt in the presence of chlorosulfonic acid.

Compounds **2**,**15**, and **34** reacted with nucleophilic reagents under anhydrous conditions in the case of coumarin reactions. The corresponding sulfanilylamino derivatives **3–13**, **16–32**, and **35–43** were obtained.

Coumarin derivatives **3–13** showed the **IR** spectra of **NH** and SO_2 absorption bands, which appeared in the normal positions,[24] and showed the carbonyl stretching absorption band in the region 1740–1715 and the C—O—C band at 1110–1105 cm^{-1}. In the 3(N-p-sulfanilylphenyl carboxamido)- coumarin (Table I), the frequency of the carbonyl band was slightly lower (1710–1700), while that of C—O—C absorption was significantly higher (1210–1205).[25,26]

EXPERIMENTAL

Melting points were taken on a Griffin melting point apparatus and were uncorrected. Infrared analysis of 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}H NMR. Mass spectra were obtained using a Schimadzu GC MS QP 1000 Ex spectrometer using the direct inlet system. TLC analyses were carried out on Merek (Darmstadt, Germany) silica gel plates and were developed with n-butanol-acetic acid-water (4:1:1) using iodine, ninhydrin, and benzidine as spraying agents.

Compounds **2**,**15**, and **34** were prepared according to the procedures described earlier.[27,28]

Coupling Reactions: 3–6, 16–20, and 35–37: General Procedure

To an amino acid (0.1 mol) in a water (25 mL) THF (15 mL) mixture was added triethylamine (5 mL), followed by a portionwise addition of sulfonyl chlorides (0.1 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. Water (50 mL) was added, and the solution acidified with 2M HCl to pH5. The crude products were filtered and recrystallized (ethanol-water). All the products **3–6**, **16–20**, and **35–37** were chromatographically homogeneous by iodine and benzidine development in TLC (cf. Chart 1, Table I).

IR of **4**: ν3250 cm^{-1} (NH), ν1700 cm^{-1} (C=O), ν1610, 1590 cm^{-1} (Ar–C=C), ν1365, 1150 cm^{-1} (SO_2), ν1200 cm^{-1} (C–O–C).

IR of **6c**: ν3250 cm^{-1} (NH), ν1705 cm^{-1} (C=C), ν1615, 1590 cm^{-1} (ArC=C), ν1355, 1165 cm^{-1} (SO_2), ν1200 cm^{-1} (C–O–C).

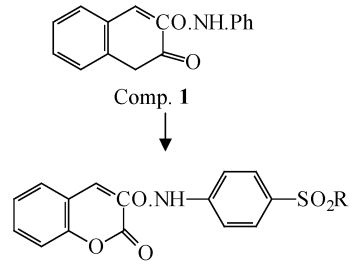
IR of **19**: ν3300 cm^{-1} (NH), ν1690 cm^{-1} (C=O), ν1590 cm^{-1} (Ar-C=C), ν1320, 1160 cm^{-1} (SO_2).

^{1}H NMR of **4**: (DMSO-d_6): δ1.2(s, CH_3-alalyl), δ4.1(s, H, CH alalyl), δ8.1–7.3(s, 9H, Ar–H), δ8.9(s, H, SO_2-NH), δ11.3, (s, H, COOH), δ11.7(s, H, CONH). **MS of 4: m/z 416 (M^+)**.

^{1}H NMR of **6b**: (DMSO-d_6): δ8.1–7.2[s, 9H, Ar–H), δ8.5 (s, 2H, NH_2), δ8.8(s, H, SO_2–NH), δ11.6(s, H, CONH). **MS of 6b: m/z 12 359 (M^+)**.

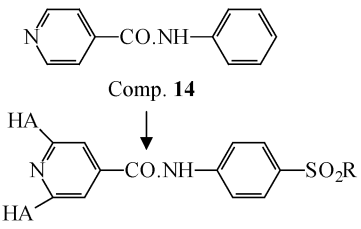
^{1}H NMR of **6c**: (DMSO-d_6): δ1.9[s, 6H, $(CH_3)_2$], δ8.5–7.3(s, 9H, Ar–H), δ10.1(s, H, SO_2–NH), δ11.9(s, H, CONH), δ11.9(s, H, CONH). **MS** of **6c**: m/z 399 (M^+).

Type (A):



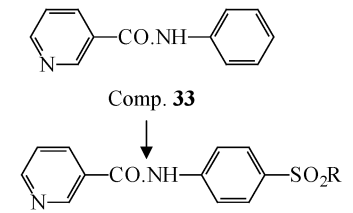
Comp. **2**, R = Cl
Coumarin-3-(N-Phenylsulfanilyl) amino acid derivatives **3–13**

Type (B):



Comp. **15**, R = Cl
N^4-(Isonicotinoyl) sulfanilylamino acid derivatives **16–32**

Type (C):



Comp. **34**, R = Cl
N^4-(Nicotinoyl) sulfanilylamino acid derivatives **(35–43)**

Chart 1

^{1}H NMR of **18**: (DMSO-d_6): δ 1.8[s, 6H, $(CH_3)_2$], δ1.97(s, H βCH valyl), δ4.33, (s, H, α CH valyl), δ7.8–7.2 (s, 6H, Ar-H), δ8.8–8.7(s, 2H, 2HA), δ9.9(s, H, SO_2-NH), δ10.9(s, H, CONH), δ11.6(s, H, COOH). **MS of 18: m/z 377 (M^+)**.

^{1}H NMR of **20b:** (DMSO-d_6): δ 1.9[s, 6H, $(CH_3)_2$], δ7.8–7.2 (s, 6H, Ar-H), δ8.8–8.7(s, 2H, 2HA), δ9.7(s, H, SO_2−NH), δ10.8(s, H, CONH). **MS of 20b**: **m/z 305 (M^+)**.

TABLE I Novel Sulfanilylamino Acids and Dipeptide Derivatives 3–13, 16—32, and 35–43

Compound. no.	R	M.P. °C	Yield %	R_f	Molecular formula	Elemental analysis % Calculated/found			
						% C	% H	% N	% S
3	Gly	174–176	65	0.87	$C_{18}H_{14}N_2O_7S$	53.73	3.48	6.97	7.96
						53.70	3.41	6.94	7.93
4	DL—Ala	146–148	68	0.69	$C_{19}H_{16}N_2O_7S$	54.81	3.85	6.73	7.69
						54.80	3.81	6.71	7.62
5	L—Val	168–170	68	0.62	$C_{20}H_{21}N_2O_7S$	56.76	4.50	6.31	7.21
						56.71	4.48	6.30	7.18
6a	L—Leu	190–192	72	0.59	$C_{22}H_{22}N_2O_7S$	57.64	4.80	6.11	6.99
						57.60	4.79	6.09	6.92
6b	NH—NH_2	280–282	70	0.75	$C_{16}H_{13}N_3O_5S$	53.48	3.62	11.70	8.91
						53.41	3.60	11.66	8.88
6c	NH—N=$(CH_3)_2$	207—209	79	0.69	$C_{19}H_{17}N_3O_5S$	57.14	4.20	10.53	8.02
						57.10	4.11	10.43	8.00
7	Gly—OMe	208–210	76	0.69	$C_{19}H_{16}N_2O_7S$	54.81	3.85	6.73	7.69
						54.80	3.81	6.71	7.63
8	DL—Ala—OMe	160–162	77	0.52	$C_{20}H_{18}N_2O_7S$	55.81	4.19	6.51	7.44
						55.77	4.08	6.48	7.34
9	L—Leu—OMe	166–168	80	0.72	$C_{23}H_{24}N_2O_7S$	58.47	5.08	5.93	6.78
						58.41	5.01	5.91	6.71
10	Gly—Gly—OMe	183–185	58	0.74	$C_{21}H_{19}N_3O_8S$	53.28	4.02	8.88	6.77
						53.21	4.00	8.81	6.73
11	DL—Ala—Gly—OMe	178–180	64	0.78	$C_{22}H_{21}N_3O_8S$	54.21	4.31	8.62	6.57
						54.20	4.30	8.60	6.51
12	L—Val—Gly—OMe	187–189	72	0.67	$C_{24}H_{25}N_3O_8S$	55.92	4.85	8.16	6.21
						55.90	4.81	8.11	6.18
13	L—Leu—Gly—OMe	173–175	77	0.70	$C_{25}H_{27}N_3O_8S$	56.71	5.10	7.94	6.05
						56.70	5.03	7.91	6.00

(Continued on next page)

TABLE I Novel Sulfanilylamino Acids and Dipeptide Derivatives 3–13, 16—32, and 35–43 *(Continued)*

Compound. no.	R	M.P. °C	Yield %	R_f	Molecular formula	Elemental analysis % Calculated/found			
						% C	% H	% N	% S
16	Gly	250–252	40	0.77	$C_{14}H_{13}N_3O_5S$	50.15	3.88	12.54	9.55
						50.00	3.83	12.51	9.51
17	DL—Ala	270–272	43	0.81	$C_{15}H_{15}N_3O_5S$	51.58	4.30	12.03	9.17
						51.51	4.11	12.00	9.00
18	L—Val	289–291	59	0.66	$C_{17}H_{19}N_3O_5S$	54.11	5.04	11.14	8.49
						54.00	5.00	11.00	8.43
19	L—Leu	294–296	60	0.55	$C_{18}H_{21}N_3O_5S$	55.24	5.37	10.74	8.18
						55.10	5.31	10.71	8.13
20a	L—Ph—ala	227–278	66	0.79	$C_{21}H_{19}N_3O_5S$	59.29	4.47	9.88	7.53
						59.00	4.41	9.80	7.51
20b	NH—N=$(CH_3)_2$	240–242	45	0.64	$C_{14}H_{15}N_3O_5S$	55.08	4.92	13.77	10.49
						55.00	4.91	13.71	10.41
21	Gly—OMe	208–210	55	0.66	$C_{15}H_{15}N_3O_5S$	51.58	4.30	12.03	9.17
						51.53	4.20	12.00	9.00
22	DL—Ala—OMe	185–187	52	0.61	$C_{16}H_{17}N_3O_5S$	52.89	4.68	11.57	8.82
						52.31	4.60	11.10	8.80
23	L—Val—OMe	190–194	47	0.68	$C_{18}H_{21}N_3O_5S$	55.24	5.37	10.74	8.18
						55.08	5.23	10.71	8.06
24	L—Leu—OMe	180–182	54	0.76	$C_{19}H_{23}N_3O_5S$	56.30	5.68	10.37	7.90
						56.21	5.61	10.31	7.81
25	Gly—N_2H_3	185–187	73	0.91	$C_{14}H_{15}N_5O_4S$	48.14	4.30	18.34	9.17
						48.00	4.11	18.31	9.03
26	DL—Ala—N_2H_3	194–196	85	0.82	$C_{15}H_{17}N_5O_4S$	49.59	4.68	19.28	8.82
						49.44	4.61	19.03	8.79
27	L—Val—N_2H_3	186–188	86	0.89	$C_{17}H_{21}N_5O_4S$	52.17	5.37	17.90	8.18
						52.01	5.27	17.81	8.00

28	L–Leu–N_2H_3	165–167	70	0.66	$C_{18}H_{23}N_5O_4S$	58.33	5.68	17.28	7.90
						53.11	5.53	17.03	7.81
29	Gly–Gly–OMe	173–175	83	0.70	$C_{17}H_{18}N_4O_6S$	50.25	4.43	13.79	7.88
						50.00	4.39	13.66	7.71
30	DL–Ala–Gly–OMe	168–170	66	0.85	$C_{18}H_{20}N_4O_6S$	51.43	5.76	13.33	7.62
						51.33	5.69	13.22	7.51
31	L–Val–Gly–OMe	205–207	74	0.80	$C_{20}H_{24}N_4O_6S$	53.57	5.36	12.50	7.14
						53.44	5.21	12.43	7.03
32	L–Leu–Gly–OMe	160–162	77	0.61	$C_{21}H_{26}N_4O_6S$	54.55	5.63	12.12	6.93
						54.44	5.51	12.03	6.81
35	Gly	176–178	50	0.81	$C_{14}H_{15}N_3O_5S$	50.15	3.88	12.54	9.55
						50.00	3.77	12.49	9.51
36	L–Val	228–230	57	0.77	$C_{17}H_{19}N_3O_5S$	54.11	5.04	11.14	8.49
						54.00	5.00	11.00	8.44
37	L–Leu	256–258	62	0.71	$C_{18}H_{21}N_3O_5S$	55.24	5.37	10.74	8.18
						55.11	5.21	10.00	8.03
38	L–Val–OMe	135–137	64	0.75	$C_{18}H_{21}N_3O_5S$	55.24	5.37	10.74	8.18
						55.00	5.11	10.61	8.04
39	L–Leu–OMe	126–128	71	0.78	$C_{19}H_{23}N_3O_5S$	56.30	5.68	10.37	7.90
						56.11	5.63	10.11	7.81
40	L–Val–N_2H_3	230–282	79	0.89	$C_{17}H_{21}N_5O_4S$	52.17	5.37	17.90	8.18
						52.11	5.21	17.88	8.11
41	L–Leu–N_2H_3	165–167	77	0.83	$C_{18}H_{23}N_5O_4S$	53.33	5.68	17.28	7.90
						55.21	5.61	17.11	7.81
42	L–Val–Gly–OMe	160–162	64	0.61	$C_{20}H_{24}N_4O_6S$	53.57	5.36	12.50	7.14
						55.44	5.51	12.41	7.00
43	L–Leu–Gly–OMe	120–122	60	0.71	$C_{21}H_{26}N_4O_6S$	54.55	5.63	12.12	6.93
						54.31	5.51	12.03	6.82

Synthesis of Sulfanilylamino Acid Methyl Esters 7–9, 21–24, and 38–39: General Procedure

A suspension of coupling reaction products **3**, **4**,**6a**, **16–19**, and **36–37** (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. after standing. Overnight, the solvent was removed by vacuum distillation. The residual solid material was recrystallized (methanol-water) (Table I).

IR of **8**: ν3460,cm^{-1} (NH), ν1370, 1170 cm^{-1} (SO_2–NH), ν1440, 1360 cm^{-1} ($COOCH_3$), ν1710 cm^{-1} (C=O), ν1310, 1610 cm^{-1} (SO_2).

^{1}H NMR of **23**: (DMSO-d_6): δ8.8–7.2(s, 8H, Ar–H), δ3.87–3.81(s, 3H, $COOCH_3$), and disappear of OH protons, and other peaks in support of their structures. **MS of 23: m/z 391 (M^+)**.

Synthesis of Sulfanilyamino Acid Hydrazides 25–28 and 40–41: General Procedure

The methyl esters **21–24** and **38–39** (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 r.t. The crystalline products **25–28** and **40–41** were filtered off, washed with water, and recrystallized (ethanol–water).

Hydrazides **25–28** and **40–41** were shown to be chromatographically to be homogeneous (Table I).

IR of **28**: ν3200 cm^{-1} (NH), ν1690 cm^{-1} (C=O), ν1580 cm^{-1} (ArC=C), ν1320, 1160 cm^{-1} (SO_2).

^{1}H NMR of **27:** δ5.61(s, 2H, NH_2), δ5.51(s, H, N<u>H</u>), δ7.8–7.1 (s, 6H, Ar–<u>H</u>), δ8.7–8.6 (s, 2H, <u>H</u>A), δ9.2 (s, H, SO_2–N<u>H</u>). **MS of 27**: **m/z 391 (M^+)**.

Synthesis of Sulfanilyl Dipeptide Methyl Esters 10–13, 29–32, and 42–43: General Procedure

To a solution of amino acid methyl ester hydrochloride (0.11 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 sulfanilyl amino acid (0.005 mol) and dicyclohexylcarbodiimide **DCC** (1.62 g) were added to the 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, which 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 **11**: ν3300, 3100 cm^{-1} (NH, CONH), ν1700 cm^{-1} (C=O), ν1360 cm^{-1} ($COOCH_3$).

^{1}H NMR of **31**: δ3300 cm^{-1} (NH), ν1370, 1170 cm^{-1} ($SO_2 \cdot NH$), ν1445, 1350 cm^{-1} ($COOCH_3$), ν1760, cm^{-1} (C=O), ν1610, cm^{-1} (ArC=C), ν1380, 1180 cm^{-1} (SO_2).

^{1}H NMR of **12**: (DMSO-d_6): δ1.8[s, 6H, $(CH_3)_2$ valyl], δ1.94(s, H βC<u>H</u> valyl), δ4.31, (s, H, αC<u>H</u> valyl), δ3.80 (s, 3H, $COOCH_3$), δ8.0–7.2(s, 9H, Ar-H), δ8.7(s, H, SO_2—N<u>H</u>), δ11.4(s, 2H, CON<u>H</u>). **MS of 12: m/z 515 (M^+).**

^{1}H NMR of **30**: (DMSO-d_6): δ1.27(s, 3H, CH_3-alalyl), δ3.80(s, H, $COOCH_3$), δ4.31(s, H, αC<u>H</u> alalyl), δ7.60(s, H, SO_2—N<u>H</u>), δ8.8–7.8 (s, 8H, Ar—H), δ8.03(s, 2H, 2CON<u>H</u>). **MS of 30: m/z 420 (M^+).**

The remaining dipeptides methyl esters **10–13**, **29–32**, and **42–43** gave analogous peaks in support of their structures.

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