Intramolecular Sulfamylation Reaction of *N*-(1*H*-indol-3-yl)ethylsulfamides: Synthesis of 2,3,4,9-Tetrahydro-1,2-thiazino[5,6-b]indole 1,1-Dioxides

Ji Sun Lee, Dong Geol Lee, Dong Seub Park, Sun Hee Kim, Han Sik Yoon,† and Chai-Ho Lee*

Department of Chemistry, Wonkwang University, Iksan, Jeonbuk 570-749, Korea
†School of Medical Radiation, Wonkwang Helth Science College, Jeonbuk 570-750, Korea
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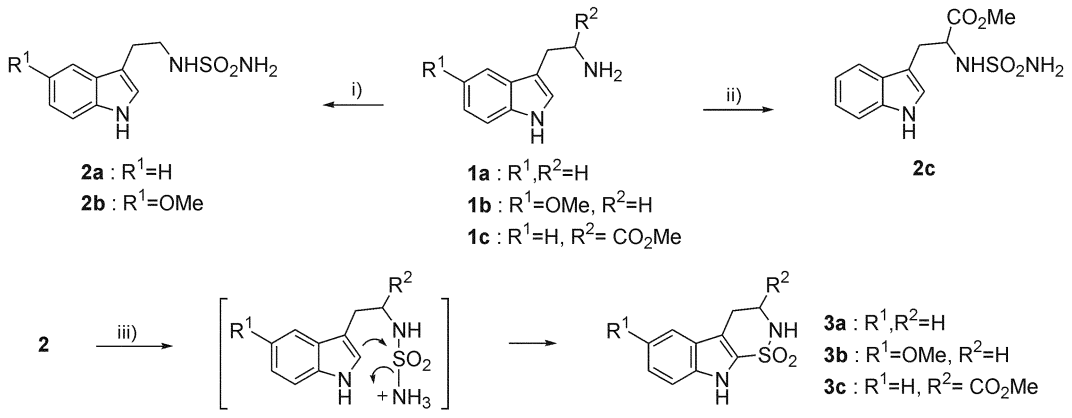
The pharmacological properties of sulfamides have commanded the interest of organic and medicinal chemists. The need for additional information is further magnified by the many useful biological properties (*i.e*., anticonvulsant, hyperglycemic, antihypertensive, histamine H_2-receptor antagonist, herbicidal, HCMV inhibitor) that have been observed for sulfamide-containing compounds.[1] The synthesis and reaction of sulfamides have been considered several times in reviews which were partially or completely devoted to sufamides.[2] One of the earliest known reactions of sulfamide is its ability to produce substituted sulfamides with alkylamines.[3] The reaction of sulfamide with aromatic amines yields not only diarylsulfamides but also gives rise to rearranged sulfanilanilides.[4] The reaction of *N,N'*-dialkylsulfamides with hypochloridate and base leads to the formation of azoalkanes.[5] Sulfamides are not as strong nucleophiles as amines; nevertheless, they can react with electrophilic reagents (*i.e*., carbonyl reagents, nitriles, and alkyl halides).[1f,2c,6] Previousely, we have demonstrated general route for the synthesis of the 1,2,5-thiadiazolidine 1,1-dioxides[7] and α-sulfamidoalkylation transformations from arylalkylsulfamides for the preparation of sulfamide derivatives.[6m,8]

In the present study, we report on the intramolecular sulfamylation reaction of *N*-(1*H*-indol-3-yl)ethylsulfamides **2** for the generation of 2,3,4,9-tetrahydrothiazino[5,6-b]-indoles **3** (Scheme 1).

The starting sulfamides **2a** and **b** were prepared from the treatment of sulfamide with the corresponding 2-arylethylamines **1a** and **b** at reflux for 12 h in H_2O, according to established synthetic protocols.[9] When *t*-butanol was reacted with an equimolar quantity of chlorosulfonylisocyanate ($OCNSO_2Cl$) in chloroform, followed by reaction with amine **1c**, the resultant was hydrolyzed with trifluoroacetic acid to give sulfamide **2c**.[10] Treatment of sulfamides **2** at reflux in acetic acid produced thiazinoindoles **3** as the major product (51-55%). A key process is the intramolecular sulfamylation reaction (**2** $\rightarrow$ **3**), which is considered to involve intramolecular aromatic attack of indole ring on protonated sulfamide group of **2** (Scheme 1).[4] Compounds **3** have been assigned as 2,3,4,9-tetrahydro-1,2-thiazino[5,6-b]indole 1,1-dioxide on the basis of the ^{1}H- and ^{13}C-NMR (500 MHz) spectral data, and mass spectroscopy. Distinctive signals of **3a** and **b** were noted in ^{1}H NMR spectra for the methylene resonances at C-4 (δ 2.87-2.95) and C-3 (δ 3.63-3.82) and in the ^{13}C NMR spectra for the C-4 (δ 22.2-22.3) and C-3 (δ 43.8-43.9 ppm). Key signals of **3c** detected in ^{1}H NMR spectra for methylene resonances at C-4 (δ 3.14 and 3.38) and C-3 (δ 4.74 ppm) and in the ^{13}C NMR spectra for the C-4 (δ 25.6) and C-3 (δ 52.1 ppm). Additional evidence



Scheme 1[a]. [a]Reagents and coditions: i) $SO_2(NH_2)_2$, H_2O, 12 h, reflux; ii) 1) $OCNSO_2Cl$, *t*-BuOH, CH_2Cl_2, 0-5 °C, 2) **1c**, Et_3N, rt, 3) CF_3CO_2H; iii) AcOH, 12 h, reflux.

*Corresponding author. E-mail: chaiho@wonkwang.ac.kr

Table 1. Crystal data and structure refinement for **3a**

Empirical formula	$C_{10}H_{10}N_2O_2S$
Formula weight	222.26
Temperature	293(2) K
Wavelength	0.71070 Å
Crystal system, space group	Monoclinic, $P2_1/c$
Unit cell dimensions	a = 10.2820(8) Å
	b = 10.3452(6) Å, β = 116.027(8)°
	c = 10.4122(14) Å
Volume	995.22(17) Å^3
Z, D_{cald}	4, 1.483 g/cm^3
μ	0.304 mm^{-1}
F(000)	464
Crystal size	0.5 × 0.5 × 0.5 mm
θ range for data collection	2.20 to 25.97°
hkl collected	+12, +12, ±12
Reflections collected/unique	2062/1954 [R(int) = 0.0587]
Completeness to 2θ = 51.94	94.5%
Refinement method	Full-matrix least-squares on F^2
Data/restraints/parameters	1954/0/137
Goodness-of-fit on F^2	1.048
Final R indices [$I > 2\sigma(I)$]	$^aR_1$ = 0.0516, $^bwR_2$ = 0.1399
R indices (all data)	$^aR_1$ = 0.0703, $^bwR_2$ = 0.1525
Extinction coefficient	0.014(4)
Largest diff. peak and hole	0.602 and −0.621 e. Å^{-3}

$^aR_1 = \Sigma \| F_o | - | F_c \| $ (based on reflections with $F_o^2 > 2\sigma F^2$. $^bwR_2 = [\Sigma [w(F_o^2 - F_c^2)^2] / \Sigma [w(F_o^2)^2]]^{1/2}$; $w = 1/[\sigma^2(F_o^2) + (0.095P)^2]$; $P = [\max (F_o^2, 0) + 2F_c^2]/3$ (also with $F_o^2 > 2\sigma F^2$)

for the structure of target compound **3a** was provided by a determination of the crystal structure by X-ray diffraction methods. Suitable crystal for X-ray analysis of **3a** has been obtained in a chloroform solution, and the crystal structures of the compound was determined by X-ray diffraction. Crystal data for complex **3a** are summarized in Table 1, refinement details are discussed in the experimental section, and selected bond distances and angles are collected in Table 2. The molecular geometries and atom-labeling schemes are shown in Figure 1.

Table 2. Elected Bond lengths [Å] and Bond Angles (deg) for Compound **3a**

Bond lengths			
S(1)-O(1)	1.431(2)	S(1)-O(2)	1.434(2)
S(1)-N(2)	1.613(2)	S(1)-C(1)	1.733(3)
N(1)-C(8)	1.371(3)	N(1)-C(1)	1.377(3)
N(2)-C(10)	1.478(4)	C(1)-C(2)	1.363(4)
C(2)-C(3)	1.433(4)	C(2)-C(9)	1.497(4)
C(3)-C(8)	1.406(4)	C(3)-C(4)	1.408(4)
C(4)-C(5)	1.377(5)	C(5)-C(6)	1.392(6)
C(6)-C(7)	1.371(5)	C(7)-C(8)	1.403(4)
C(9)-C(10)	1.515(4)		
Bond Angles			
O(1)-S(1)-O(2)	117.25(14)	O(1)-S(1)-N(2)	109.90(13)
O(2)-S(1)-N(2)	107.15(13)	O(1)-S(1)-C(1)	109.40(13)
O(2)-S(1)-C(1)	110.31(13)	N(2)-S(1)-C(1)	101.67(12)
C(8)-N(1)-C(1)	107.3(2)	C(10)-N(2)-S(1)	115.61(19)
C(2)-C(1)-N(1)	111.5(2)	C(2)-C(1)-S(1)	123.5(2)
N(1)-C(1)-S(1)	124.91(19)	C(1)-C(2)-C(3)	105.5(2)
C(1)-C(2)-C(9)	124.8(3)	C(3)-C(2)-C(9)	129.7(3)
C(8)-C(3)-C(4)	119.0(3)	C(8)-C(3)-C(2)	107.2(2)
C(4)-C(3)-C(2)	133.8(3)	C(5)-C(4)-C(3)	118.5(3)
C(4)-C(5)-C(6)	121.2(3)	C(7)-C(6)-C(5)	122.3(3)
C(6)-C(7)-C(8)	116.6(3)	N(1)-C(8)-C(7)	129.2(3)
N(1)-C(8)-C(3)	108.5(2)	C(7)-C(8)-C(3)	122.3(3)
C(2)-C(9)-C(10)	111.2(2)	N(2)-C(10)-C(9)	112.2(2)

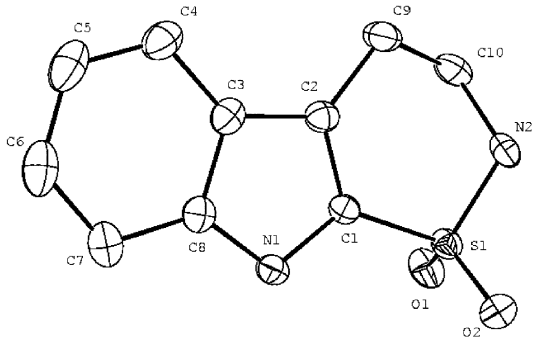


Figure 1. An ORTEP drawing of compound **3a** with atomic numbering scheme.

In conclusion, we have elucidated an intramolecular sulfamylation reaction of *N*-(1*H*-indol-3-yl)ethylsulfamides for the generation of 2,3,4,9-tetrahydro-1,2-thiazino[5,6-b]indole 1,1-dioxides.

Experimental Section

***N*-[2-(1*H*-Indol-3-yl)]ethylsulfamide (2a).** A water solution containing of tryptamine **1a** (1.6 g, 10 mmol) and sulfamide (1.0 g, 10 mmol) was heated at reflux for 12 h and then cooled to room temperature. The solid that precipitated was filtered and then washed with aqueous 1 *N* HCl solution (20 mL) and water (3 × 20 mL) to give the pale yellow powder 1.1 g (49.6 %) of **2a**; mp 137-138 °C; IR (KBr) 3422, 3420, 3400, 3264, 1321, 1140 cm^{-1}; ^{1}H NMR (DMSO-d$_6$) δ 2.89 (t, J = 7.3 Hz, 2H), 3.14 (q, J = 7.3 Hz, 2H), 6.54 (s, 2H), 6.56 (t, J = 5.6 Hz, 1H), 6.98 (td, J = 7.8 and 0.9 Hz, 1H), 7.70 (td, J = 7.8 and 0.9 Hz, 1H), 7.16 (d, J = 1.9 Hz, 1H), 7.33 (d, J = 7.8 Hz, 1H), 7.51 (d, J = 7.8 Hz, 1H), 10.8 (s, NH) ppm; ^{13}C NMR (DMSO-d$_6$) δ 25.9, 44.1, 111.9, 112.2, 118.8, 121.5, 123.3, 127.7, 136.8 ppm; LR FAB MS: calcd for [M-1]$^-$ 238.3, found 239.07.

***N*-[2-(5-Methoxy-1*H*-indol-3-yl)]ethylsulfamide (2b).** The procedure described for the preparation of **2a** was employed using **1b** (1.9 g, 10 mmol) and sulfamide (1.0 g, 10 mmol). After workup, **2b** was obtained in 43.0% yield (1.2 g); mp 130-132 °C; IR (KBr) 3404, 3322, 3246, 3129, 1335, 1148 cm^{-1}; ^{1}H NMR (Acetone-d$_6$) δ 3.01 (t, J = 7.4 Hz, 2H), 3.56 (q, J = 7.4 Hz, 2H), 3.76 (s, 3H), 5.61 (s, 1H), 5.88 (s, 2H), 6.75 (dd, J = 8.7 and 2.3 Hz, 1H), 7.09 (d, J = 2.3 Hz, 1H), 7.16 (d, J = 2.3 Hz, 1H), 7.26 (d, J = 8.7 Hz, 1H), 9.88 (s, 1H) ppm; ^{13}C NMR (Acetone-d$_6$) δ 25.6, 43.9, 55.1, 100.4, 111.6, 111.9, 123.2, 123.3, 128.0, 131.9, 153.9 ppm; LR FAB MS: calcd for [M-1]$^-$ 268.4, found 269.08.

***N*-[1-Methoxycarbonyl-2-(1*H*-indol-3-yl)]ethylsulfamide (2c).** Chlorosulfonylisocyanate (1.4 g, 10 mmol) of was

added dropwise to a cold solution of *t*-butyl alcohol (0.7 g, 10 mmol) in anhydrous dichloromethane (10 mL). Then **1c** (2.2 g, 10 mmol) and triethylamine (1.2 g, 12 mmol) was added. The mixture was stirred for 3 h at room temperature and then washed with 1 *N* HCl and with water several times. The organic layer was concentrated to dryness *in vacuo*. The residue was added to a dichloromethane (12 mL) solution containing trifluoroacetic acid (8 mL), and then the solution was stirred at room temperature for 6 h. The solution was washed with water, dried (anhydrous $MgSO_4$) and concentrated *in vacuo* to give **2c** (2.4 g, 80.1%); IR (KBr) 3400, 3261, 3153, 3096, 1341, 1163 cm^{-1}; ^1H NMR (Acetone-d_6) δ 3.26 (d, J = 6.4 Hz, 2H), 3.60 (s, 3H), 4.35 (td, J = 6.4 and 7.6 Hz, 1H), 5.98 (d, J = 7.6 Hz, 1H), 5.99 (s, 2H), 7.01 (td, J = 6.9 and 0.9 Hz, 1H), 7.09 (td, J = 6.9 and 0.9 Hz, 1H), 7.22 (d, J = 2.3 Hz, 1H), 7.37 (d, J = 6.9 Hz, 1H), 7.57 (d, J = 6.9 Hz, 1H), 10.08 (s, 1H) ppm; ^{13}C NMR (Acetone-d_6) δ 28.7, 51.6, 57.0, 109.4, 111.4, 118.4, 118.8, 121.4, 123.9, 127.7, 136.7, 172.6 ppm; LR FAB MS: calcd for $[M-1]^-$ 296.4, found 297.08.

General procedure for intramolecular sulfamylation reaction of 3. A acetic acid (20 mL) solution of sulfamides **2** (5.0 mmol) was stirred at reflux for 12 h and then cooled to rt. The solution was quenched with excess water (50 mL) and extracted with ethyl acetate (3×10 mL). The solution was washed with aqueous 5% $NaHCO_3$ (20 mL) solution and with water (3×20 mL), and then dried (anhydrous $MgSO_4$) and evaporated *in vacuo*. The solid was recrystallized from ethyl acetate to give the desired products **3**.

2,3,4,9-Tetrahydro-1,2-thiazino[5,6-b]indole 1,1-dioxide (3a): Compound **3a** was obtained from **2a** (1.2 g) in 53.0% yield (0.6 g): mp 200-245 °C dec.; IR (KBr) 3324, 3239, 1320, 1157 cm^{-1}; ^1H NMR (DMSO-d_6) δ 2.87 (t, J = 5.5 Hz, 2H), 3.63 (q, J = 5.5 Hz, 2H), 7.12 (t, J = 7.8 Hz, 1H), 7.30 (t, J = 7.8 Hz, 1H), 7.41 (d, J = 7.8 Hz, 1H), 7.60 (d, J = 7.8 Hz, 1H), 12.16 (s, 1H) ppm; ^{13}C NMR (DMSO-d_6) δ 22.2, 43.9, 112.9, 116.8, 120.6, 120.9, 125.1, 125.5, 130.5, 136.0 ppm; LR FAB MS: calcd for $[M-1]^-$ 221.2, found 222.05.

6-Methoxy-2,3,4,9-tetrahydro-1,2-thiazino[5,6-b]indole 1,1-dioxide (3b): Compound **3b** was obtained from **2b** (1.2 g) in 55.4% yield (0.7 g): mp 134-146 °C dec.; IR (KBr) 3291, 3275, 1318, 1154 cm^{-1}; ^1H NMR (Acetone-d_6) δ 2.95 (t, J = 6.0 Hz, 2H), 3.77-3.82 (m, 2H), 3.81 (s, 3H), 6.44 (t, J = 7.3 Hz, 1H), 6.96 (dd, J = 2.3 and 8.7 Hz, 1H), 7.07 (d, J = 2.3 Hz, 1H), 7.40 (d, J = 8.7 Hz, 1H), 10.93 (s, 1H) ppm; ^{13}C NMR (Acetone-d_6) δ 22.3, 43.8, 55.1, 101.1, 113.2, 116.1, 116.3, 125.6, 131.0, 131.1, 154.8 ppm; LR FAB MS: calcd for $[M-1]^-$ 251.3, found 252.06.

3-Methoxycarbonyl-2,3,4,9-tetrahydro-1,2-thiazino[5,6-b]indole 1,1-dioxide (3c): Beginning with sulfamide **2c** (1.5 g), compound **3c** was obtained in 51.2% yield (0.7 g): mp 170-180 °C dec.; IR (KBr) 3314, 1744, 1341, 1165 cm^{-1}; ^1H NMR (Acetone-d_6) δ 3.14 (dd, J = 16.8 and 11.9 Hz, 1H), 3.38 (dd, J = 16.8 and 4.3 Hz, 1H) , 3.83 (s, 3H), 4.74 (ddd, J = 12.3, 11.9, and 4.3 Hz, 1H), 6.91 (d, J = 12.3 Hz, 1H), 7.17 (td, J = 0.9 and 8.2 Hz, 1H), 7.34 (td, J = 0.9 and 8.2 Hz, 1H), 7.52 (d, J = 8.2 Hz, 1H), 7.67 (d, J = 8.2 Hz, 1H), 11.23 (s, 1H) ppm; ^{13}C NMR (Acetone-d_6) δ 25.6, 52.1, 56.8, 112.5, 115.4, 120.3, 120.6, 125.0, 125.4, 130.4, 136.4, 169.5 ppm; LR FAB MS: calcd for $[M-1]^-$ 279.4, found 280.05.

X-ray analysis of 3a. Details of the crystal data and summary of intensity data collection parameters for **3a** are given in Table 1. Crystals were grown from chloroform solution stored at room temperature. Crystal was mounted on glass fibers in random orientations, and the data were collected on a Enraf-Nonius CAD4 diffractometer equipped with graphite-monochromated Mo-Kα radiation (α = 0.71070 Å) at room temperature. Unit cell parameters were determined by using search, center, index and lest-square routine. Structure was solved by the application of direct methods using the SHELX-86 program[11] and least-squares refinement using SHELEX-97.[12] Anisotropic thermal parameters were used for all atoms except hydrogen. All the remaining hydrogen atoms were included in calculated positions.

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Supplementary material. Crystallographic Data for the strructure reported here have been deposited with the Cambridge Crystallographic Data Centre (Deposition No. CCDC-216058). That data can be obtained free of charge *via http://www.ccdc.cam.ac.uk/perl/catreq.cgi* (or from the CCDC, 12 Union Road, Cambridge CB2 IEZ, UK; fax: +44 1223 336033; *e-mail: deposit@ccdc.cam.ac.uk*).

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