Synthesis of Cyclic Sulfamides from *N,N'*-Bis(2-arylethyl)sulfamides

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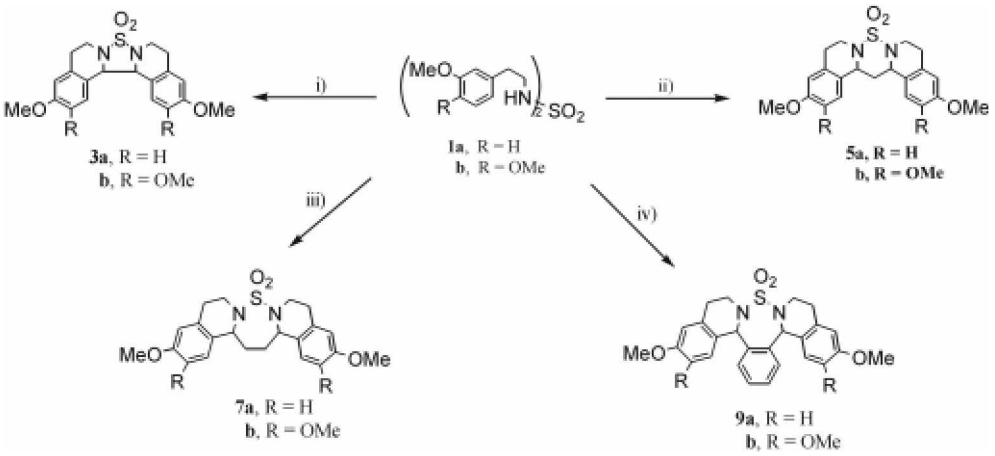
The sulfamide unit is a ubiquitous structural entity in many naturally occurring compounds and medicinal agents (*i.e.*, anticonvulsant, antihypertensive, hyperglycemia, histamine H_2-receptor antagonist, herbicide, HCMV inhibitors).[1] Previously, we have demonstrated that intermolecular α-sulfamidoalkylation reaction of *N*-(2-arylethyl)sulfamide with acetal (or aldehyde) proceeding through the intermediacy of an iminium ion provides an expeditious route for the preparation of cyclic sulfamides.[2] The α-sulfamidoalkylation reaction consists in the condensation of *N*-(2-arylethyl)sulfamide with an acetal in the acidic media to yield a tetrahydroisoquinoline containing sulfamide moiety, and is a special example of the Mannich reaction and the Pictet-Spengler reaction.[3]

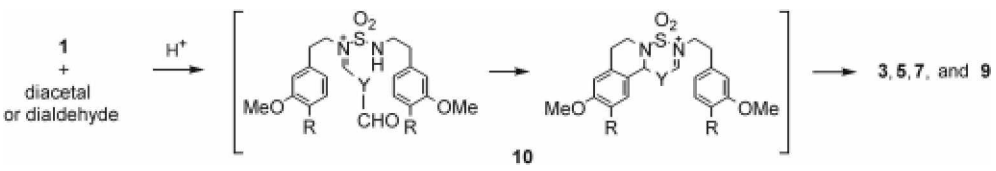
In the present study, we report on the α-sulfamidoalkylation reaction of *N,N'*-bis(2-arylethyl)sulfamides **1** with diacetals **2**, **4**, and **6** and dialdehyde **8** in formic acid for the generation of cyclic sulfamides of novel structures (Scheme 1). The starting sulfamides **1** were prepared from the treatment of sulfamide with the corresponding 2-arylethylamine at reflux for 6 hr in anhydrous pyridine, according to established synthetic protocols.[3e,4]

Treatment of sulfamides **1** with diacetals **2**, **4**, and **6** and dialdehyde **8** in formic acid (96% in water) gave as the major product (43-98% yield) the fused ring compounds **3**, **5**, **7**, and **9**. Formation of **3**, **5**, **7**, and **9** is believed to proceed by the stepwise pathway depicted in Scheme 2.

Compounds **3**, **5**, **7**, and **9** exhibited characteristic absorption bands in the infrared spectrum at 1152-1167 and 1310-1329 cm^{-1} of sulfonyl group.[5] Diagnostic signals were observed for the methine unit furnished by acetals or aldehyde at δ 4.45-4.97 ppm for **3**, **5**, **7** and 6.27-6.30 ppm for **9** in the ^1H NMR spectra, and at δ 55.0-65.2 ppm for **3**, **5**, **7** and 57.3-57.4 ppm for **9** in the ^{13}C NMR spectra. Distinctive signals for two methylene protons of tetrahydroisoquinoline ring of **3**, **5**, **7**, and **9** were noted at δ 2.66-2.83, 2.72-3.66, 2.91-3.21 and 3.74-4.02 ppm as multiplet in the ^1H NMR spectra, and at δ 28.5-30.3 and 39.0-42.9 ppm in



Scheme 1. Reagents; i) dimethoxyacetaldehyde (**2**), ii) 1,1,3,3-tetraethoxypropane (**4**), iii) 2,5-diethoxytetrahydrofuran (**6**), iv) phthalic dicaroxaldehyde (**8**).



Scheme 2

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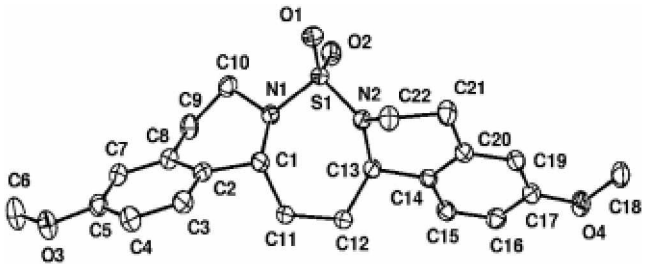

Figure 1. An ORTEP drawing of compound **7a** with atomic numbering scheme.

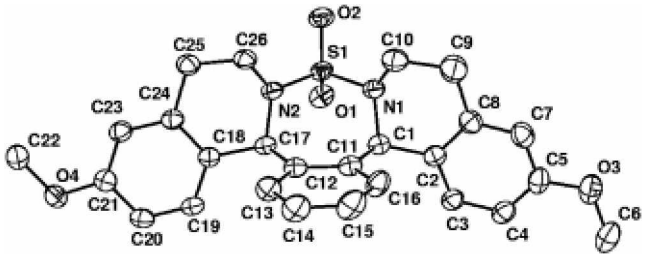

Figure 2. An ORTEP drawing of compound **9a** with atomic numbering scheme.

the ^{13}C NMR spectra. Analysis of **7a** and **9a** by X-ray crystallography confirmed the proposed ditetrahydroisoquinolino-annelated 1,2,7-thiadiazepane ring structure. ORTEP view of **7a** and **9a** is presented in Figure 1 and Figure 2, respectively.

In conclusion, we have elucidated an α-sulfamidoalkylation reaction of *N,N'*-bis(2-arylethyl)sulfamides **1** with diacetals **2**, **4**, and **6** and dialdehyde **8** in formic acid for the generation of novel cyclic sulfamides. The success achieved with arylsulfamides suggests that the corresponding alkenyl and acetylenic sulfamides should serve as suitable starting materials the construction of alicyclic based sulfamides.

General methods. Infrared spectra were obtained on a JASCO FT/IR-5300 spectrophotometer and NMR spectra were recorded on JEOL (500 MHz) FT-NMR spectrometer. Chemical shifts (δ) were given in ppm relative to TMS. Low-resolution mass spectra were obtained on a Quattro AC spectrometer at the Wonkwang University. The solvents and reactants were purchased from Aldrich Chemical Co., and were used without purification.

General procedure for α-sulfamidoalkylation reaction of *N,N'*-bis(2-arylethyl)sulfamide 1 with acetals 2, 4, and 6 (or dialdehyde 8): A formic acid (96% in H_2O, 20 mL) solution of sulfamides **1** (2.0 mmol) and acetals (2.0 mmol) was stirred for 24 hr at room temperature, and then the solution was quenched with excess water (50 mL). The solid that precipitated was filtered and then recrystallized from acetone-hexane to give the desired products.

3,11-Dimethoxy-5,6,8,9,13b,13c-hexahydro-7-thia-6a,7a-diazadibenzo[c,g]fluorine 7,7-dioxide (3a). Beginning with sulfamide **1a** (0.73 g) and acetal **2** (0.21 g), compound **3a** was obtained in 75% yield (0.58 g): mp 178-180 °C; IR (KBr) 1323, 1167 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.81 (ddd, J = 15.2, 4.1 and 3.2 Hz, 2H), 3.11 (ddd, J = 15.2, 10.5 and 4.6 Hz, 2H), 3.20 (ddd, J = 12.9, 10.5 and 3.2 Hz, 2H), 3.81 (s, 6H), 3.95 (ddd, J = 12.9, 4.6 and 4.1 Hz, 2H), 4.46 (s, 2H), 6.49 (d, J = 8.7 Hz, 2H), 6.68 (dd, J = 8.7 and 2.8 Hz, 2H), 6.77 (d, J = 2.8 Hz, 2H) ppm; ^{13}C NMR ($CDCl_3$) δ 29.6, 42.9, 55.4, 65.1, 112.0, 113.9, 112.4, 130.4, 137.2, 159.3 ppm; LR FAB MS: calcd for $[M+1]^+$ 387.14, found 387.23.

2,3,11,12-Tetramethoxy-5,6,8,9,13b,13c-hexahydro-7-thia-6a,7a-diazadibenzo[c,g]fluorene 7,7-dioxide (3b). Beginning with sulfamide **1b** (0.85 g) and acetal **2** (0.21 g), compound **3b** was obtained in 78% yield (0.70 g): mp 174-177 °C; IR (KBr) 1329, 1167 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.80 (ddd, J = 15.2, 4.1 and 3.7 Hz, 2H), 3.05 (ddd, J = 15.2, 9.4 and 4.6 Hz, 2H), 3.23 (ddd, J = 13.1, 9.4 and 3.7 Hz, 2H), 3.62 (s, 6H), 3.89 (s, 6H), 3.92 (ddd, J = 13.1, 4.6 and 4.1 Hz, 2H), 4.45 (s, 2H), 6.08 (s, 2H), 6.74 (s, 2H) ppm; ^{13}C NMR ($CDCl_3$) δ 28.9, 42.9, 55.9, 56.0, 65.2, 111.8, 112.5, 121.9, 128.1, 146.8, 148.8 ppm; LR FAB MS: calcd for $[M+1]^+$ 447.16, found 447.31.

3,11-Dimethoxy-5,8,9,13b,14,14a-hexahydro-6H-7-thia-6a,7a-diazadibenzo[a,j]anthracene 7,7-dioxide (5a). Beginning with sulfamide **1a** (0.73 g) and acetal **4** (0.33 g), compound **5a** was obtained in 63% yield (0.50 g): mp 226-228 °C; IR (KBr) 1319, 1154 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.44 (t, J = 6.9 Hz, 2H), 2.29 (ddd, J = 17.0, 7.6 and 6.9 Hz, 2H), 3.08 (ddd, J = 17.0, 6.4 and 5.5 Hz, 2H), 3.66 (ddd, J = 12.9, 6.9 and 6.4 Hz, 2H), 3.74 (ddd, J = 12.9, 7.6 and 5.5 Hz, 2H), 3.79 (s, 6H), 4.79 (t, J = 6.9 Hz, 2H), 6.69 (d, J = 2.3 Hz, 2H), 6.78 (dd, J = 8.7 and 2.3 Hz, 2H), 7.04 (d, J = 8.7 Hz, 2H) ppm; ^{13}C NMR ($CDCl_3$) δ 28.8, 32.9, 42.0, 54.6, 55.3, 112.9, 113.9, 126.2, 127.1, 135.1, 158.6 ppm; LR FAB MS: calcd for $[M+1]^+$ 401.15, found 401.24.

2,3,11,12-Tetramethoxy-5,8,9,13b,14,14a-hexahydro-6*H*-7-thia-6a,7a-diazadibenzo[a,j]anthracene 7,7-dioxide (5b). Beginning with sulfamide **1b** (0.85 g) and acetal **4** (0.33 g), compound **5b** was obtained in 71% yield (0.65 g): mp 294-295 °C; IR (KBr) 1319, 1165 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.07 (dd, J = 14.2 and 12.4 Hz, 1H), 2.02 (dd, J = 14.2 and 2.8 Hz, 1H), 2.83 (ddd, J = 16.0, 5.7 and 4.8 Hz, 2H), 2.91 (ddd, J = 16.0, 7.3 and 5.0 Hz, 2H), 3.48 (ddd, J = 11.9, 5.7 and 5.0 Hz, 2H), 3.57 (ddd, J = 11.9, 7.3 and 4.8 Hz, 2H), 3.85 (s, 6H), 3.87 (s, 6H), 4.97 (dd, J = 12.4 and 2.8 Hz, 2H), 6.59 (s, 2H), 6.63 (s, 2H) ppm; ^{13}C NMR ($CDCl_3$) δ 28.5, 35.0, 40.8, 55.9, 56.5, 57.3, 109.4, 111.6, 125.8, 127.0, 147.9, 148.4 ppm; LR FAB MS: calcd for $[M+1]^+$ 461.17, found 461.32.

3,11-Dimethoxy-5,6,8,9,13b,14,15,15a-octahydro-7-thia-6a,7a-diazadinaphthaleno[1,2-a;1',2'-e]cycloheptene 7,7-dioxide (7a). Beginning with sulfamide **1a** (0.73 g) and acetal **6** (0.32 g), compound **7a** was obtained in 88% yield (0.73 g): mp 198-204 °C; IR (KBr) 1315, 1153 cm^{-1}; ^1H NMR ($CDCl_3$) δ 2.01-2.03 (m, 2H), 2.17-2.19 (m, 2H), 2.82 (ddd, J = 16.2, 4.3 and 3.7 Hz, 2H), 3.01 (ddd, J = 16.2, 10.2 and 5.2 Hz, 2H), 3.51 (ddd, J = 13.2, 10.2 and 3.7 Hz, 2H), 3.78 (s, 6H), 3.99 (ddd, J = 13.2, 5.2 and 4.3 Hz, 2H), 4.72-4.74 (m, 2H), 6.65 (d, J = 2.5 Hz, 2H), 6.75 (dd, J = 8.7 and 2.5 Hz, 2H), 7.00 (d, J = 8.7 Hz, 2H) ppm; ^{13}C NMR ($CDCl_3$) δ 30.3, 35.0, 40.3, 55.0, 55.4, 112.9, 113.4, 128.1,

Table 1. Crystal data and structure refinement for **7a** and **9a**

	7a	**9a**
Temperature	293(2) K	293(2) K
Wavelength	0.71070 Å	0.71070 Å
Crystal system, space group	Monoclinic, $P2_1/c$	Monoclinic, $P2_1/n$
Unit cell dimensions	a = 16.1253(13) Å α = 90°.	a = 11.0637(7) Å
	b = 7.1637(8) Å β = 92.308(6)°.	b = 16.2745(16) Å β = 91.915(6)°
	c = 17.2203(12) Å γ = 90°.	c = 12.2462(10) Å
Volume	1987.6(3) $Å^3$	2203.8(3) $Å^3$
Z, D_{calc}	4, 1.385 mg/m^3	4, 1.394 mg/m^3
μ	0.195 mm^{-1}	0.184 mm^{-1}
F(000)	880	976
Crystal size	0.4 × 0.4 × 0.45 mm	0.35 × 0.40 × 0.45 mm
θ range for data collection	1.26 to 25.97 deg.	2.08 to 25.97 deg.
Index ranges	0<=h<=19, 0<=k<=8, -21<=l<=21	0<=h<=13, 0<=k<=20, -15<=l<=15
Reflections collected / unique	3898 / 3776 [R(int) = 0.0441]	4544/4315 [R(int) = 0.0324]
Refinement method	Full-matrix least-squares on F^2	Full-matrix least-squares on F^2
Data / restraints / parameters	3776/0/270	4315/0/310
Goodness-of-fit on F^2	0.960	1.034
Final R indices [I>2σ(I)]	[a]$R1$ = 0.0516, [b]$wR2$ = 0.1375	[a]$R1$ = 0.0531, [b]$wR2$ = 0.1451
R indices (all data)	[a]$R1$ = 0.1178, [b]$wR2$ = 0.1704	[a]$R1$ = 0.1079, [b]$wR2$ = 0.1713
Largest diff. peak and hole	0.190 and -0.303 e. $Å^{-3}$	0.259 and -0.367 e. $Å^{-3}$

[a]$R1 = \Sigma||F_o|-|F_c|| / \Sigma|F_o|$ (based on reflections with $F_o^2 > 2\sigma F^2$). [b]$wR2 = [\Sigma[w(F_o^2-F_c^2)^2]/\Sigma[w(F_o^2)^2]]^{1/2}$; $w = 1/[\sigma^2(F_o^2)+(0.095P)^2]$; $P = [\max(F_o^2, 0)+2F_c^2]/3$(also with $F_o^2 > 2\sigma F^2$).

129.2, 135.6, 158.3 ppm; LR FAB MS: calcd for $[M+1]^+$ 414.2, found 414.4.

2,3,11,12-Tetramethoxy-5,6,8,9,13b,14,15,15a-octahydro-7-thia-6a,7a-diazadinaphthaleno[1,2-a; 1',2'-e]cycloheptene 7,7-dioxide (7b). Beginning with sulfamide **1b** (0.85 g) and acetal **6** (0.32 g), compound **7b** was obtained in 98% yield (0.93 g): mp 226-228 °C; IR (KBr) 1310, 1152 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 2.04-2.07 (m, 2H), 2.18-2.20 (m, 2H), 2.76 (ddd, *J* = 16.0, 4.2 and 3.7 Hz, 2H), 2.96 (ddd, *J* = 16.0, 10.3 and 5.5 Hz, 2H), 3.52 (ddd, *J* = 13.2, 10.3 and 3.7 Hz, 2H), 3.84 (s, 6H), 3.85 (s, 6H), 4.02 (ddd, *J* = 13.2, 5.5 and 4.2 Hz, 2H), 4.69-4.71 (m, 2H), 6.56 (s, 2H), 6.61 (s, 2H) ppm; ^{13}C NMR ($CDCl_3$) δ 29.5, 34.7, 40.4, 55.1, 56.0, 56.2, 109.9, 111.5, 126.5, 128.7, 147.8, 148.1 ppm ; LR FAB MS: calcd for $[M+1]^-$ 474.2, found 474.4.

3,11-Dimethoxy-5,6,8,9,13b,17b-hexahydro-7-thia-6a,7a-diazabenzo[c]dinaphthaleno[1,2-a;1',2'-e]cycloheptene 7,7-dioxide (9a). Beginning with sulfamide **1a** (0.73 g) and aldehyde **8** (0.27 g), compound **9a** was obtained in 43% yield (0.40 g): mp 280-282 °C; IR (KBr) 1325, 1159 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 2.69-2.73 (m, 2H), 2.72-2.75 (m, 2H), 3.13-3.21 (m, 2H), 3.74-3.78 (m, 2H), 3.84 (s, 6H), 6.30 (s, 2H), 6.53 (dd, *J* = 5.8 and 3.7 Hz, 2H), 6.76 (d, *J* = 2.7 Hz, 2H), 6.80 (dd, *J* = 8.2 and 2.7 Hz, 2H), 7.00 (d, *J* = 8.2 Hz, 2H), 7.11 (dd, *J* = 5.8 and 3.2 Hz, 2H) ppm; ^{13}C NMR ($CDCl_3$) δ 29.2, 39.0, 55.4, 57.3, 112.5, 114.0, 126.3, 127.5, 129.3, 131.3, 136.1, 140.5, 159.0 ppm; LR FAB MS: calcd for $[M+1]^-$ 463.2, found 463.5.

2,3,11,12-Tetramethoxy-5,6,8,9,13b,17b-hexahydro-7-thia-6a,7a-diazabenzo[c]dinaphthaleno[1,2-a;1',2'-e]cycloheptene 7,7-dioxide (9b). Beginning with sulfamide **1b** (0.85 g) and aldehyde **8** (0.27 g), compound **9b** was obtained in 58% yield (0.61 g): mp 300-302 °C; IR (KBr) 1316, 1157 cm^{-1}; ^{1}H NMR ($CDCl_3$) δ 2.66 (dd, *J* = 15.6 and 4.6 Hz, 2H), 2.72 (ddd, *J* = 11.9, 11.3 and 4.6, Hz, 2H), 3.13 (ddd, *J* = 15.6, 11.3 and 7.3 Hz, 2H), 3.77 (dd, *J* = 11.9 and 7.3 Hz, 2H), 3.81 (s, 6H), 3.91 (s, 6H), 6.27 (s, 2H), 6.57 (s, 2H), 6.60 (dd, *J* = 5.6 and 3.7 Hz, 2H), 6.71 (s, 2H), 7.14 (dd, *J* = 5.7 and 3.2 Hz, 2H) ppm; ^{13}C NMR ($CDCl_3$) δ 28.6, 39.1, 56.0, 57.4, 110.8, 111.7, 125.8, 126.9, 127.6, 131.5, 140.3,

Table 2. Selected Bond lengths [Å] and Angles [deg] for **6a**[a]

Bond lengths			
S(1)-N(2)	1.602(3)	S(1)-N(1)	1.635(3)
N(1)-C(10)	1.477(4)	N(1)-C(1)	1.494(4)
N(2)-C(22)	1.464(4)	N(2)-C(13)	1.468(4)
C(1)-C(2)	1.517(4)	C(1)-C(11)	1.533(4)
C(3)-C(4)	1.378(5)	C(4)-C(5)	1.392(5)
C(11)-C(12)	1.530(4)	C(12)-C(13)	1.528(4)
C(13)-C(14)	1.516(4)	C(14)-C(15)	1.389(4)
C(14)-C(20)	1.392(4)	C(15)-C(16)	1.379(5)
C(21)-C(22)	1.507(4)	C(20)-C(21)	1.518(4)
Bond angles			
O(1)-S(1)-N(2)	106.87(15)	O(2)-S(1)-N(1)	105.07(15)
O(1)-S(1)-N(1)	112.99(14)	N(2)-S(1)-N(1)	102.19(13)
C(10)-N(1)-C(1)	119.0(2)	C(10)-N(1)-S(1)	114.6(2)
C(1)-N(1)-S(1)	117.4(2)	C(22)-N(2)-C(13)	115.5(3)
C(22)-N(2)-S(1)	123.3(2)	C(13)-N(2)-S(1)	120.8(2)
N(1)-C(1)-C(2)	111.8(2)	N(1)-C(1)-C(11)	108.5(3)
C(2)-C(1)-C(11)	109.6(23)	C(8)-C(9)-C(10)	109.0(3)
N(1)-C(10)-C(9)	110.1(3)	C(12)-C(11)-C(1)	117.8(3)
C(13)-C(12)-C(11)	115.5(3)	N(2)-C(13)-C(14)	109.3(2)
N(2)-C(13)-C(12)	110.0(3)	C(14)-C(13)-C(12)	112.9(3)
C(22)-C(21)-C(20)	112.5(3)	N(2)-C(22)-C(21)	108.5(3)

[a]Symmetry transformations used to generate equivalent atoms.

Table 3. Selected Bond Lengths [Å] and Angles [deg] for **9a**[a]

Bond lengths			
S(1)-O(1)	1.423(2)	S(1)-O(2)	1.427(2)
S(1)-N(2)	1.622(3)	S(1)-N(1)	1.642(3)
N(1)-C(10)	1.472(4)	N(1)-C(1)	1.485(4)
N(2)-C(17)	1.467(4)	N(2)-C(26)	1.477(4)
C(1)-C(2)	1.502(4)	C(1)-C(11)	1.537(4)
C(8)-C(9)	1.520(5)	C(2)-C(8)	1.400(4)
C(17)-C(18)	1.514(4)	C(9)-C(10)	1.528(5)
C(18)-C(24)	1.392(4)	C(11)-C(12)	1.408(4)
C(24)-C(25)	1.519(4)	C(12)-C(17)	1.538(4)
C(25)-C(26)	1.510(4)		
Bond Angles			
O(1)-S(1)-O(2)	120.29(14)	O(1)-S(1)-N(2)	105.87(14)
O(2)-S(1)-N(2)	107.88(13)	O(1)-S(1)-N(1)	105.15(13)
O(2)-S(1)-N(1)	106.90(14)	N(2)-S(1)-N(1)	110.62(14)
C(10)-N(1)-C(1)	111.8(2)	C(21)-O(4)-C(22)	116.8(3)
C(1)-N(1)-S(1)	115.4(2)	C(10)-N(1)-S(1)	120.2(2)
C(17)-N(2)-S(1)	118.5(2)	C(17)-N(2)-C(26)	113.9(2)
N(1)-C(1)-C(2)	106.8(2)	C(26)-N(2)-S(1)	122.1(2)
C(3)-C(2)-C(1)	120.9(3)	N(1)-C(1)-C(11)	112.8(2)
C(2)-C(8)-C(9)	120.6(3)	C(8)-C(2)-C(1)	120.7(3)
N(1)-C(10)-C(9)	109.0(3)	C(7)-C(8)-C(9)	119.9(3)
N(2)-C(17)-C(12)	112.2(2)	C(8)-C(9)-C(10)	114.8(3)
N(2)-C(26)-C(25)	107.1(2)	N(2)-C(17)-C(18)	108.5(2)

[a]Symmetry transformations used to generate equivalent atoms.

147.5, 148.6 ppm; LR FAB MS: calcd for $[M+1]^+$ 523.2, found 523.6.

X-ray Analysis of 7a and 9a. Details of the crystal data and summary of intensity data collection parameters for **7a** and **9a** are given in Table 1. Crystals were grown from chloroform solution stored at room temperature. Crystals were mounted on glass fibers in random orientations, and the data were collected on a Enraf-Nonius CAD4 diffractometer equipped with graphite-monochromated Mo-K_α 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[6] and least-squares refinement using SHELEX-97.[7] Anisotropic thermal parameters were used for all atoms except hydrogen. All the remaining hydrogen atoms were included in calculated positions. Some selected bond lengths and bond angles for **7a** and **9a** are shown in Table 2 and Table 3, respectively.

Supplementary material. Tables of full bond distances and bond angles, anisotropic thermal parameters, and atomic coordinates of hydrogen atoms are available from the corresponding author.

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