

Chemoselective *N*-Benzenesulfonylation of Aliphatic Amines

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Chemoselective *N*-benzenesulfonylation of aliphatic amines using 2-(4-nitrobenzenesulfonyl)-4,5-dichloropyridazin-3(2*H*)-one (**2**) gave the corresponding 4-nitrobenzenesulfonamides in good or excellent yield. This method is a simple, mild and general procedure for the chemoselective *N*-benzenesulfonylation of aliphatic amines.

Key Words : Chemoselective *N*-benzenesulfonylation, 2-(4-Nitrobenzenesulfonyl)-4,5-dichloropyridazin-3-one, Pyridazinone

Introduction

Sulfonyl groups have been widely used to protect amines and other nitrogenous functionality in organic synthesis.¹ 4-Nitrobenzenesulfonyl groups as protecting/activating groups have also been frequently used for the preparation of secondary amines both in solution- and solid-phase synthesis.² One of the most attractive features of sulfonamides is their stability under various reaction conditions. Recently, the use of *N*-sulfonyl protecting groups has been improved by addition of new varieties amenable to some mild removal.³ Although sulfonyl chlorides are widely used for *N*-sulfonylation of amines, their drawbacks are the instability and the too strong reactivity. Also, selective *N*-sulfonylation of primary or secondary amine in the presence of secondary or tertiary amines in organic solvents is often required in laboratory. These problems and requirement led us to develop a selective, stable and mild *N*-benzenesulfonylating agent.

In connection with our research program for the synthesis of 5-alkylamino-4-halopyridazin-3-ones, we found the *N*-benzenesulfonyl-transfer potentiality of 2-(benzenesulfonyl)-4,5-dichloropyridazin-3-ones to amines.⁴ We investigated the application of 2-(benzenesulfonyl)-4,5-dichloropyridazin-3-ones as a novel *N*-benzenesulfonylating agent of amines. According to the literature,⁴ compound **2** show the excellent transfer potentiality among 2-(benzenesulfonyl) derivatives. 4-Nitrobenzenesulfonamide is also unique among the amino protective groups.^{3d} Therefore, we chose the compound **2** as a *N*-benzenesulfonylating agent.

In this paper, we would like to report a simple, mild and general procedure for the chemoselective *N*-benzenesulfonyla-

tion of aliphatic amines using **2**.

According to Kweons, *et al.*,⁴ the product distribution for the reaction of amines with **2** in the same solvent is dependent on the structure of amine, the basicity of amine, the substituent of benzene ring and the substituted position of benzene ring. Even though the solvent effect was not reported in their paper, the product distribution may be dependent on the dielectric constant of the solvent used. Therefore, we investigated firstly the reaction of gaseous ethylamine with **2** in some solvents with the different dielectric constants at room temperature. The results were summarized in Table 1. *N*-Sulfonylation of ethylamine (*g*) using **2** in *n*-hexane or cyclohexane selectively gave only *N*-ethyl-(4-nitrobenzene)sulfonamide (**3a**).

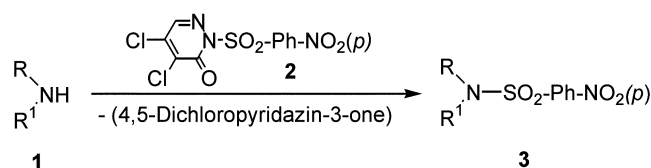
According to preliminary experiment, the selectivity of *N*-sulfonylation in diethyl ether and *n*-hexane was also dependent on the reaction temperature. The reaction, however, in refluxing cyclohexane yielded selectively *N*-sulfonylated amine. Therefore, we carried out *N*-benzenesulfonylation of some amines using **2** by suspension in refluxing cyclohexane. The results are summarized in Table 2. The reaction of primary amines involving only amino group with **2** in cyclohexane afforded only the corresponding sulfonamides **3** in 90-95% yields (Table 2, Entry 1-5).

Table 1. Reaction of ethylamine (*g*) with **2** in various solvents at room temperature.

Entry	Solvent	Time (h)	Product (%) ^a	
			Sulfonamide	5-EtNH-pyridazinone
1	<i>n</i> -hexane	21	97	—
2	cyclohexane	28	97	—
3	diethyl ether	5	94	— ^b
4	ethyl acetate	2	75	20
5	tetrahydrofuran	5	61	8
6	dichloromethane	3.5	91	8
7	acetonitrile	1.5	75	21
8	dimethylformamide	2	76	14

^aIsolated yield. 4,5-Dichloropyridazin-3-one was also isolated quantitatively.

^bDetection of trace on TLC plate.



Scheme 1

Table 2. *N*-Benzenesulfonylation of various aliphatic amines (1 equiv) with **2** (1.2 equiv) in refluxing cyclohexane

Entry	Amine	Time (h)	Sulfonamide 3 (%) ^a
1	Ethylamine (g)	9	3a (92) ^b
2	cyclohexylamine	2.5	3b (95)
3	benzylamine	2.5	3c (95)
4	4-methylbenzylamine	3	3d (95)
5	2,4-difluorobenzylamine	8.5	3e (90)
6	4-aminobenzylamine	9	3f (92) ^c
7	2-aminoethanol	01	3g (88) ^d
8	<i>N</i> -methylbenzylamine	25	3h (95)
9	piperidine	3	3i (98)
10	diethylamine	3	3j (98)
11	diethanolamine	2.5	3k (80)
12	2-methylpiperazine	9.5	3l (92) ^e

^aYield of isolated product. 4,5-Dichloropyridazin-3-one was also recovered quantitatively. ^bAt room temperature. ^cThe main product was *N*-(4-aminobenzyl)-4-nitrobenzenesulfonamide. 5-[(4-Aminobenzylamino)]-4-chloropyridazin-3-one was also isolated in 3% yield. ^d5-(2-Hydroxyethyl)amino-4-chloropyridazin-3-one was also isolated in 6% yield. ^eThe product is 4-(4-nitrobenzenesulfonyl)-2-methylpiperazine.

Treatment of 4-aminobenzylamine with **2** gave *N*-(4-aminobenzyl)-4-nitrobenzenesulfonamide (**3f**) (92%) and 5-[(4-aminobenzyl)amino]-4-chloropyridazin-3-one (3%) (Table 2, Entry 6). 2-Aminoethanol was also reacted with **2** in cyclohexane to give *N*-(2-hydroxyethyl)-4-nitrobenzenesulfonamide (**3g**) (88%) and 5-[(2-hydroxyethyl)amino]-4-chloropyridazin-3-one (6%) (Table 2, Entry 7). *N*-Sulfonylation of some secondary amines such as *N*-methylbenzylamine, piperidine, diethylamine and diethanolamine with **2** in cyclohexane selectively yielded only the corresponding sulfonamides **3h-3k** in 80-98% yields (Table 2, Entry 8-11). Also sulfonylation of 2-methylpiperazine with **2** gave selectively 4-(4-nitrobenzenesulfonyl)-2-methylpiperazine (**3l**) (Table 2, Entry 12). On the other hand, reaction of secondary bulky amines such as *N*-isopropylbenzylamine and dicyclohexylamine with **2** did not afford the corresponding sulfonamides. The sulfonylation of 4-aminobenzylamine, 2-aminoethanol and diethanolamine occurred chemoselectively at the aliphatic amino groups (Table 2, Entry 6, 7, 11).

We also investigated the selectivity in the sulfonylation of a mixture (1 : 1 mole ratio) of a hindered amine and a less-hindered amine with **2** (1 equivalent). According to preliminary test for the amine mixture, *N*-benzenesulfonylation of a mixture of amines with **2** under at 35-40 °C showed the excellent selectivity. Therefore, the competition reaction was carried out at 35-40 °C. The results are summarized in Table 3. In a mixture of a primary amine and a secondary amine, *N*-sulfonylation of the primary amine is more favorable than the secondary amine except for piperidine (Table 3, Entry 1, 2, 4, 5 and 6). In the case of a mixture of α -branched amine and a primary or a secondary amine, *N*-sulfonylation of the primary and the secondary amines is more selective than the α -branched primary amine (Table 3, Entry 7 and 8).

In all mixtures of a hindered amine and a less-hindered amine, *N*-sulfonylation occurs generally at the less-hindered

Table 3. Competition reaction of amines mixture with **2** in cyclohexane at 35-40 °C (amine-1/amine-2/**2** = 1 : 1 : 1 ratio)

Entry	Amine mixture	Time (h)	3 R R ¹ N	(%) ^a
1	EtNH ₂ / (Et) ₂ NH	17	EtNH (Et) ₂ N	3a (83) 3j (10)
2	C ₆ H ₁₁ NH ₂ / (C ₆ H ₁₁) ₂ NH	3 ^b	C ₆ H ₁₁ NH (C ₆ H ₁₁) ₂ N	3b (94) –
3	PhCH ₂ NH ₂ / PhNH ₂	85	PhCH ₂ NH PhNH	3c (96) –
4	C ₆ H ₁₁ NH ₂ / (CH ₂) ₅ NH ^c	106	C ₆ H ₁₁ NH (CH ₂) ₅ N	– 3i (98)
5	PhCH ₂ NH ₂ / PhCH ₂ NHMe	50	PhCH ₂ NH PhCH ₂ NMe	3c (51) 3h (48)
6	PhCH ₂ NH ₂ / PhCH ₂ NH(i-Pr)	73	PhCH ₂ NH PhCH ₂ N(i-Pr)	3c (94) –
7	PhCH ₂ NH ₂ / PhCH(Me)NH ₂	43	PhCH ₂ NH PhCH(Me)NH	3c (92) –
8	PhCH(Me)NH ₂ / PhCH ₂ NHMe	62	PhCH(Me)NH PhCH ₂ NMe	3m (11) 3h (85)

^aIsolated yields. 4,5-Dichloropyridazin-3-one was isolated quantitatively. ^bAt reflux temperature. ^cPiperidine.

amino group. The selectivity for *N*-sulfonylation of **2** with amines may be due to the steric bulkiness around N-S bond in compound **2**.⁵

In conclusion, compound **2** as a new sulfonylating reagent has several advantages as follows: (1) it is easily prepared from commercially available 4,5-dichloropyridazin-3-one in one step under mild condition, (2) it is stable in air and easy to handle, (3) it shows good chemoselectivity for aliphatic amino group, and (4) reusable 4,5-dichloropyridazin-3-one can also be recovered quantitatively.

Experimental Section

General remarks. TLC was performed on SiO₂ (silica gel 60 F254, Merck). The spots were located by UV light. Column chromatography was carried out on SiO₂ (silica gel 60, 70-230 mesh). Melting points were determined with a Thomas-Hoover capillary apparatus and were uncorrected. ¹H and ¹³C NMR spectra were obtained on a Bruker FT NMR-DRX 500 or Varian Inova 500 spectrometer and with chemical shift values reported in δ units (part per million) relative to an internal standard (tetramethylsilane). IR spectra were obtained on a Hitachi 270-50 or Mattson Genesis Series FT-IR spectrophotometer. Elemental analyses were performed with a Perkin Elmer 240C.

Typical procedure for the sulfonylation of amines. To a solution of **2**⁴ (1.2 mmol) and a suitable solvent (20 mL) was added amines (1.0 mmol, excess for ethyl amine (g) or an amine mixture (1 : 1 mole ratio)). The mixture was stirred until **2** was disappeared at room temperature, 35-40 °C or reflux temperature. After evaporating the solvent under reduced pressure, the resulting residue was purified by column chromatography on silica gel using dichloromethane as the eluent to afford the corresponding sulfonamides **3**.

***N*-Ethyl-4-nitrobenzenesulfonamide (3a):** mp 102-103 °C. IR (KBr): 3262, 3106, 2967, 2862, 1607, 1524, 1477, 1435, 1346, 1310, 1162, 1087, 1057, 1013, 949, 855, 798, 740, 683 cm⁻¹. ¹H NMR (CDCl₃): δ = 1.15 (t, 3H, *J* = 7.3 Hz), 3.09 (m, 2H), 4.85 (t, 1H, *J* = 5.6 Hz, D₂O exchangeable), 8.07 (m, 2H), 8.37 (m, 2H). ¹³C NMR (CDCl₃): δ = 15.2, 38.5, 124.4, 128.3, 146.1, 150.1. Anal. Calcd for C₈H₁₀N₂O₄S: C, 41.73; H, 4.38; N, 12.17; S, 13.92. Found: C, 41.88; H, 4.45; N, 12.32; S, 14.20.

***N*-Cyclohexyl-4-nitrobenzenesulfonamide (3b):** mp 135-137 °C. IR (KBr): 3288, 3124, 2946, 2929, 2856, 1609, 1526, 1448, 1349, 1333, 1299, 1159, 1073, 857, 740, 685 cm⁻¹. ¹H NMR (CDCl₃): δ = 1.20 (m, 5H), 1.54 (m, 1H), 1.65 (m, 2H), 1.77 (m, 2H), 3.22 (m, 1H), 4.89 (d, 1H, *J* = 7.7 Hz, D₂O exchangeable), 8.09 (d, 2H, *J* = 7.3 Hz), 8.36 (d, 2H, *J* = 7.1 Hz). ¹³C NMR (CDCl₃): δ = 24.6, 25.0, 34.0, 53.2, 124.4, 128.2, 147.5, 150.0. Anal. Calcd for C₁₂H₁₆N₂O₄S: C, 50.69; H, 5.67; N, 9.85; S, 11.28. Found: C, 50.73; H, 5.78; N, 9.96; S, 11.32.

***N*-Benzyl-4-nitrobenzenesulfonamide (3c):** mp 124-125 °C. IR (KBr): 3300, 3100, 3080, 1610, 1530, 1500, 1460, 1420, 1350, 1330, 1310, 1210, 1160, 1120, 1090, 1060, 1030, 1010, 980 cm⁻¹. ¹H NMR (CDCl₃ + DMSO-*d*₆): δ = 4.10 (d, 2H, *J* = 6.2 Hz), 7.21 (m, 5H + NH, D₂O exchangeable for NH), 7.99 (m, 2H), 8.26 (m, 3H). ¹³C NMR (CDCl₃ + DMSO-*d*₆): δ = 52.1, 129.2, 132.7, 133.1, 133.4, 133.6, 141.9, 152.2, 154.7. Anal. Calcd for C₁₃H₁₂N₂O₄S: C, 53.42; H, 4.14; N, 9.58; S, 10.97. Found: C, 53.57; H, 4.32; N, 9.78; S, 11.13.

***N*-(4-Methylbenzyl)-4-nitrobenzenesulfonamide (3d):** mp 161-162 °C. IR (KBr): 3310, 3130, 2950, 2880, 1620, 1540, 1440, 1360, 1340, 1320, 1170, 1100, 1080, 870, 820, 740, 690 cm⁻¹. ¹H NMR (CDCl₃): δ = 2.30 (s, 3H), 4.18 (d, 2H, *J* = 5.95 Hz), 4.88 (s, NH, D₂O exchangeable), 7.05 (m, 4H), 7.26 (s, 1H), 7.98 (m, 2H), 8.29 (d, 2H, *J* = 8.79 Hz). ¹³C NMR (CDCl₃): δ = 21.1, 47.2, 124.3, 127.9, 128.3, 129.5, 132.4, 138.2, 146.2, 150.0. Anal. Calcd for C₁₄H₁₄N₂O₄S: C, 54.89; H, 4.61; N, 9.14; S, 10.47. Found: C, 54.94; H, 4.72; N, 9.32; S, 10.54.

***N*-(2,4-Difluorobenzyl)-4-nitrobenzenesulfonamide (3e):** mp 106-107 °C. IR (KBr): 3310, 3100, 2970, 2870, 1610, 1530, 1440, 1360, 1340, 1280, 1160, 1070, 970, 860, 820, 790, 740 cm⁻¹. ¹H NMR (CDCl₃): δ = 4.26 (d, 2H, *J* = 6.21 Hz), 5.14 (t, NH, *J* = 5.88 Hz, D₂O exchangeable), 6.69 (m, 1H), 6.80 (m, 1H), 7.24 (m, 1H), 7.97 (d, 2H, *J* = 8.65), 8.29 (d, 2H, *J* = 8.62 Hz). ¹³C NMR (CDCl₃): δ = 41.10, 41.13, 103.90, 104.10, 104.30, 111.50, 111.60, 111.72, 111.75, 118.90, 119.00, 119.09, 119.12, 124.30, 128.30, 131.19, 131.24, 131.27, 131.3, 146.00, 150.10, 159.80, 159.90, 161.80, 161.90, 162.00, 163.90, 164.00. Anal. Calcd for C₁₃H₁₀N₂O₄SF₂: C, 47.56; H, 3.07; N, 8.53; S, 9.77. Found: C, 47.65; H, 3.22; N, 8.74; S, 9.87.

***N*-(4-Aminobenzyl)-4-nitrobenzenesulfonamide (3f):** mp 178-180 °C. IR (KBr): 3650, 3430, 3350, 3100, 3000, 2900, 2700, 1630, 1540, 1480, 1420, 1360, 1340, 1280, 1160, 1100 cm⁻¹. ¹H NMR (DMSO-*d*₆): δ = 3.86 (d, 2H, *J* = 6.0 Hz), 4.98 (bs, 2H, D₂O exchangeable), 6.42 (m, 2H), 6.82

(m, 2H), 7.99 (m, 2H), 8.26 (t, 1H, *J* = 6.0 Hz, D₂O exchangeable), 8.36 (m, 2H). ¹³C NMR (DMSO-*d*₆): δ = 46.6, 113.8, 123.9, 124.7, 128.4, 129.1, 147.0, 148.4, 149.7. Anal. Calcd for C₁₃H₁₃N₃O₄S: C, 50.81; H, 4.26; N, 13.67; S, 10.43. Found: C, 50.92; H, 4.32; N, 13.74; S, 10.53.

***N*-(2-Hydroxyethyl)-4-nitrobenzenesulfonamide (3g):** mp 119-121 °C. IR (KBr): 3350, 3160, 3150, 2900, 2000, 1840, 1620, 1540, 1500, 1420, 1360, 1320, 1180, 1100 cm⁻¹. ¹H NMR (DMSO-*d*₆): δ = 2.88 (m, 2H), 3.38 (m, 2H), 4.69 (t, 1H, *J* = 5.4 Hz, D₂O exchangeable), 7.99 (t, 1H, *J* = 5.8 Hz, D₂O exchangeable), 8.05 (m, 2H), 8.41 (m, 2H). ¹³C NMR (DMSO-*d*₆): δ = 45.5, 60.2, 124.9, 128.4, 146.8, 149.9. Anal. Calcd for C₈H₁₀N₂O₅S: C, 39.02; H, 4.09; N, 11.38; S, 13.02. Found: C, 39.22; H, 4.12; N, 11.41; S, 13.10.

***N*-Methyl-*N*-benzyl-4-nitrobenzenesulfonamide (3h):** mp 109-110 °C. IR (KBr): 3100, 3000, 2980, 1620, 1540, 1360, 1180, 1100, 980, 920, 860, 780, 760, 700 cm⁻¹. ¹H NMR (CDCl₃): δ = 2.69 (s, 3H), 4.22 (s, 2H), 7.31 (m, 5H), 8.02 (m, 2H), 8.38 (m, 2H). ¹³C NMR (CDCl₃): δ = 34.4, 54.1, 124.4, 128.3, 128.4, 128.6, 128.8, 134.9, 143.8, 150.1. Anal. Calcd for C₁₄H₁₄N₂O₄S: C, 54.89; H, 4.61; N, 9.14; S, 10.47. Found: C, 54.94; H, 4.79; N, 9.32; S, 10.75.

1-(4-Nitrobenzenesulfonyl)piperidine (3i): mp 168-170 °C. IR (KBr): 3113, 2949, 2934, 2852, 1607, 1530, 1471, 1345, 1310, 1175, 1091, 1052, 939, 856, 747, 706 cm⁻¹. ¹H NMR (CDCl₃): δ = 1.46 (m, 2H), 1.66 (m, 4H), 3.06 (t, 4H, *J* = 5.5 Hz), 7.95 (d, 2H, *J* = 6.9 Hz), 8.37 (d, 2H, *J* = 6.9 Hz). ¹³C NMR (CDCl₃): δ = 23.4, 25.2, 46.9, 76.8, 77.1, 77.3, 124.3, 128.8, 142.8, 150.1. Anal. Calcd for C₁₁H₁₄N₂O₄S: C, 48.88; H, 5.22; N, 10.36; S, 11.86. Found: C, 48.99; H, 5.34; N, 10.43; S, 11.93.

***N,N*-Diethyl-4-nitrobenzenesulfonamide (3j):** mp 131-132 °C. IR (KBr): 3113, 2949, 2934, 2852, 1607, 1530, 1471, 1345, 1310, 1175, 1091, 1052, 939, 856, 747, 706 cm⁻¹. ¹H NMR (CDCl₃): δ = 1.16 (t, 6H, *J* = 7.2 Hz), 3.30 (q, 4H, *J* = 7.2 Hz), 8.00 (d, 2H, *J* = 8.7 Hz), 8.35 (d, 2H, *J* = 8.7 Hz). ¹³C NMR (CDCl₃): δ = 14.2, 42.2, 124.4, 128.1, 146.6, 149.9. Anal. Calcd for C₁₀H₁₄N₂O₄S: C, 46.50; H, 5.46; N, 10.85; S, 12.41. Found: C, 46.67; H, 5.71; N, 10.91; S, 12.55.

***N,N*-Di(2-hydroxyethyl)-4-nitrobenzenesulfonamide (3k):** mp 125-127 °C. IR (KBr): 3450, 2950, 2900, 1600, 1580, 1500, 1350, 1300 cm⁻¹. ¹H NMR (DMSO-*d*₆): δ = 3.26 (t, 4H, *J* = 6.5 Hz and 6.0 Hz), 3.53 (t, 4H, *J* = 6.5 Hz and 6.0 Hz), 4.86 (bs, 2H, D₂O exchangeable), 8.08 (m, 2H), 8.40 (m, 2H). ¹³C NMR (DMSO-*d*₆): δ = 51.4, 60.2, 125.2, 129.2, 145.7, 150.3. Anal. Calcd for C₁₀H₁₄N₂O₆S: C, 41.37; H, 4.86; N, 9.65; S, 11.05. Found: C, 41.45; H, 4.92; N, 9.74; S, 11.11.

1-(4-Nitrobenzenesulfonyl)-2-methylpiperazine (3l): mp 138-140 °C. IR (KBr): 3400, 3100, 2950, 2900, 2850, 2750, 1600, 1510, 1440, 1345, 1245, 1175, 1115, 1100 cm⁻¹. ¹H NMR (DMSO-*d*₆): δ = 0.93 (d, 3H, *J* = 6.0 Hz), 1.86 (t, 1H, *J* = 10.0 Hz and 11.0 Hz), 2.20 (m, 1H), 2.66 (m, 2H), 2.88 (m, 1H), 3.23 (bs, 1H, D₂O exchangeable), 3.49 (m, 2H), 8.00 (m, 2H), 8.45 (m, 2H). ¹³C NMR (DMSO-*d*₆): δ = 19.1, 44.7, 46.2, 50.0, 52.5, 124.9, 129.3, 141.1, 150.3. Anal. Calcd for C₁₁H₁₅N₃O₄S: C, 46.30; H, 5.30; N, 14.73; S, 11.24. Found: C, 46.40; H, 5.42; N, 14.82; S, 11.35.

***N*-(α -Methylbenzyl)-4-nitrobenzenesulfonamide (3m):** mp 118-120 °C. IR (KBr): 3250, 3100, 3000, 1600, 1520, 1450, 1430, 1350, 1320, 1310, 1160, 1100 cm⁻¹. ¹H NMR (DMSO-*d*₆): δ = 1.28 (d, 3H, *J* = 7.0 Hz), 4.44 (m, 1H), 7.12 (m, 5H), 7.84 (m, 2H), 8.22 (m, 2H), 8.59 (d, 1H, *J* = 8.0 Hz, D₂O exchangeable). ¹³C NMR (DMSO-*d*₆): δ = 21.9, 51.7, 122.5, 124.5, 125.3, 126.3, 126.5, 141.0, 145.5, 147.5. Anal. Calcd for C₁₄H₁₄N₂O₄S: C, 54.89; H, 4.61; N, 9.14; S, 10.47. Found: C, 54.93; H, 4.72; N, 9.22; S, 10.51.

4-Chloro-5-ethylamino-2-(4-nitrobenzenesulfonyl)pyridazin-3-one: mp 202-204 °C. IR (KBr): 3310, 3114, 2980, 2936, 1656, 1622, 1529, 1435, 1347, 1326, 1193, 1104, 1008, 854, 742, 680 cm⁻¹. ¹H NMR (CDCl₃): δ = 1.54 (t, 3H, *J* = 7.0 Hz), 3.46 (m, 2H), 7.43 (bs, NH, D₂O exchangeable), 8.25 (d, 2H, *J* = 9.0 Hz), 8.28 (s, 1H), 8.44 (d, 2H, *J* = 9.0 Hz). ¹³C NMR (CDCl₃): δ = 15.5, 37.3, 101.9, 124.4, 130.3, 130.7, 142.0, 144.9, 150.8, 154.5. Anal. Calcd for C₁₂H₁₁N₄O₅SCl: C, 40.18; H, 3.09; N, 15.62; S, 8.94. Found: C, 40.33; H, 3.21; N, 15.80; S, 9.02.

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