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Efficient Synthesis of Highly Functionalized Cyclic Aminimides

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Supporting Information

Experimental procedures and characterization data for the preparation of compounds
12-22 and **23-29**.

¹H-NMR and ¹³C-NMR spectra were obtained using a Varian Gemini-300 (300 MHz for ¹H, and 75 MHz for ¹³C), or a Varian Inova-500 (500 MHz for ¹H, and 125 MHz for ¹³C) spectrometer. Chemical shifts are reported relative to tetramethylsilane peak (δ 0.00) or solvent peak (δ 7.26 for CDCl₃ in ¹H NMR, δ 77.2 for CDCl₃ in ¹³C NMR). Elemental analyses were performed by the Organic Chemistry Research Center at Sogang University using a Carlo Erba EA 1180 elemental analyzer. Electron spray ionization (ESI) low resolution mass spectra were obtained with a Waters Micromass ZQ mass spectrometer. IR spectra were obtained using a Thermo-Nicolet Avatar-320 IR spectrometer with a single bounce ATR accessory (Ge crystal). THF and ethyl ether were distilled from sodium benzophenone ketyl. Methylene chloride was distilled from calcium hydride. *N,N*-dimethylformamide (DMF) was dried over 4 Å molecular sieves. *N,N*-dimethylhydrazine, *N*-aminomorpholine, *N*-aminopiperidine, 1-amino-4-methylpiperazine were purchased from Aldrich. All other chemicals were used as received otherwise specified.

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General Procedure:

4-Hydroxy-1,1-dimethyl-pyrazolidin-3-one (12): A solution of epoxy methyl ester **8** (1.9 g, 18.6 mmol), dimethyl hydrazine (**A**) (1.70 mL, 22.4 mmol) in *i*-PrOH (5 mL) was stirred for 3 h at room temperature. The resulting white precipitates were filtered through a filter paper, washed with diethyl ether, and dried under vacuum to give the product as a white solid (2.3 g, 95%): m.p.: 266 °C; 1H NMR (500 MHz, D_2O) δ 4.67 (dd, $J = 7.5$ Hz and 5.5 Hz, 1H), 4.03 (dd, $J = 7.5$ Hz and 12.3 Hz, 1H), 3.64 (dd, $J = 5.5$ Hz and 12.3 Hz, 1H), 3.18 (s, 3H), 3.11 (s, 3H); ^{13}C NMR (125 MHz, D_2O) 179.85, 71.44, 69.93, 58.42, 57.38; IR (Ge ATR) 3085, 3027, 1606, 1342, 1305, 1259, 1112 cm^{-1} . Anal. Calcd for $C_5H_{10}N_2O_2$: C, 46.14; H, 7.74; N, 21.52. Found: C, 46.22; H, 7.36; N, 21.30.

3-Hydroxy-1,5-diaza-spiro[4.5]decan-2-one (13): Yield = 71%; m.p: 263 °C; 1H NMR (500 MHz, D_2O) δ 4.62 (dd, $J = 5.0$ and 7.5 Hz, 1H), 4.00 (dd, $J = 7.5$ and 12.5 Hz, 1H), 3.59 (dd, $J = 5.0$ and 12.5 Hz, 1H), 3.4~3.2 (m, 4H), 1.88 (m, 2H), 1.74 (m, 2H), 1.59 (m, 1H), 1.50 (m, 1H); ^{13}C NMR (125 MHz, D_2O) 179.80, 70.19, 67.39, 66.30, 21.68, 21.50, 20.48; IR (Ge ATR) 2945, 1565, 1454, 1388, 1316, 1106, 1050 cm^{-1} . Anal. Calcd for $C_8H_{14}N_2O_2$: C, 56.45; H, 8.29; N, 16.46. Found: C, 56.49; H, 8.50; N, 16.30.

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3-Hydroxy-8-oxa-1,5-diazaspiro[4,5]decane-2-one (14): Yield = 49%; m.p.: 252 °C; ^1H NMR (500 MHz, D_2O) δ 4.65 (dd, J = 5.5 and 7.5 Hz, 1H), 4.09 (dd, J = 7.5 and 12.5 Hz, 1H), 4.08 (m, 2H), 3.91 (ddd, J = 5.0, 5.0, and 5.0 Hz, 1H), 3.88 (ddd, J = 5.0 Hz, 5.0, and 5.0 Hz, 1H), 3.73 (dd, J = 5.5 and 12.5 Hz, 1H), 3.59 (ddd, J = 3.0, 7.5 and 12.0 Hz, 1H), 3.53 (ddd, J = 3.0, 8.0 and 12.5 Hz, 1H), 3.43 (m, 1H), 3.32 (m, 1H); ^{13}C NMR (125 MHz, D_2O) 180.32, 70.07, 68.70 (br), 65.71, 64.58, 62.40, 62.28; IR (Ge ATR) 3079, 2875, 1593, 1467, 1392, 1292, 1132, 1060 cm^{-1} . Anal. Calcd for $\text{C}_7\text{H}_{12}\text{N}_2\text{O}_3$: C, 48.83; H, 7.02; N, 16.27. Found: C, 48.53; H, 7.52; N, 16.13. LRMS (ESI): m/z 194.7 (195.1 calcd for $\text{C}_7\text{H}_{12}\text{N}_2\text{O}_3\text{Na}$ $[\text{M}+\text{Na}]^+$), 172.7 (173.1 calcd for $\text{C}_7\text{H}_{13}\text{N}_2\text{O}_3$ $[\text{M}+\text{H}]^+$).

3-Hydroxy-8-methyl-1,5,8-triaza-spiro[4.5]decan-2-one (15): yield = 45%; m.p: 211 °C; ^1H NMR (500 MHz, D_2O) δ 4.63 (dd, J = 5.3 and 7.5 Hz, 1H), 4.05 (dd, J = 7.5 and 12.5 Hz, 1H), 3.68 (dd, J = 5.3 and 12.5 Hz, 1H), 3.50 (br m, 1H), 3.44 (br m, 1H), 3.42 (br m, 1H), 3.34 (br m, 1H), 2.86 (br m, 2H), 2.72 (br m, 2H), 2.26 (s, 3H); ^{13}C NMR (125 MHz, D_2O) 180.17, 70.07, 65.59, 64.48, 49.39, 49.26, 43.99, 23.89; IR (Ge ATR) 3443, 2869, 1652, 1570, 1468, 1447, 1370, 1291, 1146, 1095 cm^{-1} . LRMS (ESI): m/z 207.8 (208.1 calcd for $\text{C}_8\text{H}_{15}\text{N}_3\text{O}_2\text{Na}$ $[\text{M}+\text{Na}]^+$), 185.8 (186.1 calcd for $\text{C}_8\text{H}_{16}\text{N}_3\text{O}_2$ $[\text{M}+\text{H}]^+$).

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4-Hydroxy-1,1,4-trimethyl-pyrazolidin-3-one (16): A solution of 2-methyloxirane-2-carboxylic acid methyl ester (**9**, 300 mg, 2.60 mmol), dimethyl hydrazine (236 μ L, 3.10 mmol) in *i*-PrOH (3 mL) was stirred at room temperature for 6 h. The resulting precipitates were collected by filtration, washed with diethyl ether, and dried under vacuum to give cyclic aminimide **16** as a white solid (251 mg, 67%): m.p: 252 $^{\circ}$ C; ^1H NMR (500 MHz, D_2O) δ 3.93 (d, $J = 13.0$ Hz, 1H), 3.76 (d, $J = 13.0$ Hz, 1H), 3.23 (s, 3H), 3.15 (s, 3H), 1.37 (s, 3H); ^{13}C NMR (125 MHz, D_2O) 180.89, 77.96, 74.56, 58.28, 57.73, 22.40; IR (film) 3065, 1620, 1605, 1472, 1388, 1208, 1175, 1147, 999 cm^{-1} . Anal. Calcd for $\text{C}_6\text{H}_{12}\text{N}_2\text{O}_2$: C, 49.98; H, 8.39; N, 19.43. Found: C, 49.80; H, 8.57; N, 19.58.

3-Hydroxy-3-methyl-8-oxa-1,5-diaza-spiro[4.5]decan-2-one (17): Yield = 53%; m.p: 277 $^{\circ}$ C; ^1H NMR (500 MHz, D_2O) δ 4.08 (m, 2H), 4.03 (d, $J = 13.0$ Hz, 1H), 3.88 (m, 2H), 3.75 (d, $J = 13.0$ Hz, 1H), 3.61 (ddd, $J = 3.0, 7.5$, and 13.0 Hz, 1H), 3.51 (m, 2H), 3.33 (m, 1H), 1.38 (s, 3H); ^{13}C NMR (125 MHz, D_2O) 181.39, 76.40, 73.27 (br), 65.57, 64.73, 62.38, 62.34, 22.41; IR (Ge ATR) 3102, 1588, 1476, 1408, 1389, 1294, 1166, 1127, 1058 cm^{-1} . LRMS (ESI): m/z 208.7 (209.1 calcd for $\text{C}_8\text{H}_{14}\text{N}_2\text{O}_3\text{Na}$ $[\text{M}+\text{Na}]^+$), 186.7 (187.1 calcd for $\text{C}_8\text{H}_{15}\text{N}_2\text{O}_3$ $[\text{M}+\text{H}]^+$).

(4-Hydroxy-1,1-dimethyl-3-oxo-pyrazolidin-4-yl)-acetic acid methyl ester (18): Yield = 62%; m.p: 210 $^{\circ}$ C; ^1H NMR (500 MHz, D_2O) δ 4.17 (d, $J = 22.0$ Hz, 1H), 4.10 (d, $J = 22.0$ Hz, 1H), 3.67 (s, 3H), 3.33 (s, 3H), 3.26 (s, 3H), 2.99 (d, $J = 28.5$ Hz,

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1H), 2.84 (d, $J = 28.5$ Hz, 1H); ^{13}C NMR (125 MHz, D_2O) 178.95, 172.35, 78.32, 73.27, 58.45, 57.72, 52.62, 40.10; IR (Ge ATR) 3032, 1730, 1622, 1349, 1223, 1202 cm^{-1} . Anal. Calcd for $\text{C}_8\text{H}_{14}\text{N}_2\text{O}_4$: C, 47.52; H, 6.98; N, 13.85. Found: C, 47.60; H, 7.11; N, 13.62.

(3-Hydroxy-2-oxo-8-oxa-1,5-diaza-spiro[4.5]dec-3-yl)-acetic acid methyl ester (19): Yield = 35%; m.p: 212 $^\circ$; ^1H NMR (500 MHz, D_2O) δ 4.18 (d, $J = 13.0$ Hz, 1H), 4.09 (m, 2H), 4.01 (d, $J = 13.0$ Hz, 1H), 3.90 (br d, $J = 13.5$ Hz, 2H), 3.64 (m, 1H), 3.63 (s, 3H), 3.56 (m, 2H), 3.35 (br d, $J = 12.5$ Hz, 1H), 2.93 (d, $J = 17.0$ Hz, 1H), 2.77 (d, $J = 17.0$ Hz, 1H); ^{13}C NMR (125 MHz, D_2O) 179.39, 172.32, 76.73, 72.14 (br), 65.83, 64.73, 62.44, 62.36, 52.63, 40.06; IR (KBr) 3427, 3135, 2958, 2928, 2903, 1725, 1604, 1448, 1431, 1356, 1225, 1203, 1127, 1036 cm^{-1} . Anal. Calcd for $\text{C}_{10}\text{H}_{16}\text{N}_2\text{O}_5$: C, 49.17; H, 6.60; N, 11.47. Found: C, 49.20; H, 6.75; N, 11.59.

3-Hydroxy-4-phenyl-8-oxa-1,5-diaza-spiro[4.5]decan-2-one (20): Yield = 75%; m.p: 199 $^\circ$; ^1H NMR (500 MHz, D_2O) δ 7.59-7.49 (m, 5H), 5.18 (d, $J = 9.3$ Hz, 1H), 4.76 (d, $J = 9.3$ Hz, 1H), 4.18 (ddd, $J = 13.0, 11.5$ and 1.5 Hz, 1H), 4.02 (ddd, $J = 2.0, 10.5$ and 12.5 Hz, 1H), 3.86 (dd, $J = 3.5, 13.0$ Hz, 1H), 3.70 (br d, $J = \sim 11$ Hz, 1H), 3.67 (dt, $J = 4$ and 12.5 Hz, 1H), 3.32 (d, $J = 13.0$ Hz, 1H), 3.01 (dd, $J = 1.5$ and 12.0 Hz, 1H), 2.95 (ddd, $J = 4.0, 12.5$ and 12.5 Hz, 1H); ^{13}C NMR (125 MHz, D_2O) 180.36, 131.77, 131.34, 129.87, 127.27, 84.84, 71.52, 62.36, 62.18, 61.48, 59.83; IR (KBr) 3422, 3096, 2954, 2929, 2891, 1590, 1472, 1459, 1446, 1402, 1392, 1289, 1125, 1072, 937 cm^{-1} . Anal. Calcd for $\text{C}_{13}\text{H}_{16}\text{N}_2\text{O}_3$: C, 62.89; H, 6.50; N, 11.28. Found: C, 62.92; H, 6.62; N, 11.13.

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2,3-Dibromo-propionic acid methyl ester: To a solution of methyl acrylate (10.0 g, 0.12 mmol) in CH_2Cl_2 (30 mL) was slowly added bromine (5.95 mL, 0.12 mmol) using a dropping funnel at 0 °C. The resulting solution was allowed to stir at 0 °C for 1 h. The reaction was quenched by adding a saturated sodium thiosulfate solution (20 mL). The resulting suspension was allowed to stir for 1 h at room temperature. The mixture was diluted with CH_2Cl_2 (10 mL) and extracted with CH_2Cl_2 (3 × 10 mL). The organic layers were washed with brine, dried over Na_2SO_4 , and concentrated. Purification of the crude product by flash chromatography (Hex:EtOAc = 3:1) on silica gel gave the product as a yellow liquid (27.5 g, 95%): ^1H NMR (300 MHz, CDCl_3) δ 4.45 (dd, J = 4.5 and 11.4 Hz, 1H), 3.93 (dd, J = 9.9 and 11.4 Hz, 1H), 3.85 (s, 3H), 3.68 (dd, J = 4.5 and 9.9 Hz, 1H).

1-(Toluene-4-sulfonyl)-aziridine-2-carboxylic acid methyl ester (21): To a suspension of 2,3-dibromo-propionic acid methyl ester (2.60 mL, 10.8 mmol) and K_2CO_3 (4.5 g, 32.5 mmol) in CH_3CN (15 mL) was added toluenesulfonamide (1.85 g, 10.8 mmol) at rt. Then the reaction mixture was heated at 80 °C overnight. The reaction was quenched by adding H_2O and the mixture was extracted with EtOAc (15 mL × 3). The combined organic layers were washed with brine, dried over Na_2SO_4 , and concentrated. The residue was purified by flash chromatography (Hex:EtOAc = 3:1) on silica gel to give the product as a yellow liquid **21** (1.5 g, 56%): ^1H NMR (500 MHz, CDCl_3) δ 7.83 (d, J = 8.4 Hz, 2H), 7.35 (d, J = 8.1 Hz, 2H), 3.72 (s, 3H), 3.33 (dd, J = 4.2 and 7.2 Hz, 1H), 2.75 (d, J = 7.2 Hz, 1H), 2.55 (d, J = 4.2 Hz, 1H), 2.44 (s, 3H); ^{13}C NMR (75 MHz, CDCl_3) 178.31, 142.89, 136.37, 129.41, 125.27, 52.48, 31.24, 20.90,

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18.43.

1-Methanesulfonyl-aziridine-2-carboxylic acid methyl ester (22): To a solution of 2,3-dibromo-propionic acid methyl ester (2.20 g, 9.02 mmol), K_2CO_3 (3.7 g, 27.1 mmol) in CH_3CN (10 mL) was added dropwise methanesulfonamide (858 mg, 9.02 mmol) at room temperature. Then the reaction mixture was stirred at 80 °C overnight. After the starting material disappeared judging from TLC analysis, the reaction was quenched with H_2O , extracted with EtOAc (15 mL $\times$ 3). The organic layers were washed with brine, dried over Na_2SO_4 , and concentrated. Then the residue was purified by flash chromatography (Hexane/EtOAc = 3/1) on silica gel to give **22** as a yellow liquid (945 mg, 52%): ^1H NMR (300 MHz, CDCl_3) δ 3.78 (s, 3H), 3.28 (dd, J = 4.2 and 7.2 Hz, 1H), 3.12 (s, 3H), 2.74 (dd, J = 2.1 and 7.2 Hz, 1H), 2.60 (dd, J = 2.1 and 4.2 Hz, 1H);

^{13}C NMR (75 MHz, CDCl_3) 179.23, 54.26, 42.11, 36.87, 22.46.

***N*-(5,5-Dimethyl-2-oxo-pyrrolidin-3-yl)-4-methyl-benzenesulfonamide (24):**

A solution of 1-(toluene-4-sulfonyl)-aziridine-2-carboxylic acid methyl ester (**21**, 230 mg, 0.9 mmol) and dimethyl hydrazine (82 μL , 1.1 mmol) in *i*-PrOH (1 mL) was stirred at rt for 5 h. The resulting precipitates were filtered, washed with diethyl ether, and dried under vacuum to give the product as a white solid (189 mg, 74%): m.p: 223 °C ^1H NMR (500 MHz, D_2O) δ 7.67 (d, J = 7.8 Hz, 2H), 7.35 (d, J = 7.8 Hz, 2H), 4.46 (t, J = 8.5 Hz, 1H), 3.89 (dd, J = 7.5 and 11.5 Hz, 1H), 3.34 (dd, J = 8.0 and 12.0 Hz, 1H), 3.09 (s, 3H), 3.02 (s, 3H), 2.32 (s, 3H); ^{13}C NMR (125 MHz, D_2O) 176.14, 145.73, 135.21, 130.43, 126.99, 68.10, 58.13, 57.16, 55.28, 20.88; IR (Ge ATR) 3036, 2840, 1636, 1598, 1327, 1162, 1094, 919 cm^{-1} . Anal. Calcd for $\text{C}_{12}\text{H}_{17}\text{N}_3\text{O}_3\text{S}$: C, 50.87; H,

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6.05; N, 14.83. Found: C, 50.72; H, 6.14; N, 14.95.

4-Methyl-N-(2-oxo-1,5-diaza-spiro[4.5]dec-3-yl)-benzenesulfonamide (25): A solution of 1-(toluene-4-sulfonyl)-aziridine-2-carboxylic acid methyl ester **21** (370 mg, 1.45 mmol) and *N*-aminopiperidine (188 μ L, 1.74 mmol) in *i*-PrOH (2 mL) was stirred at room temperature for 3 h. The resulting precipitates were isolated by filtration, washed with diethyl ether, and dried under vacuum to give **25** as a white solid (375 mg, 80%): m.p: 230 $^{\circ}$ C; 1H NMR (500 MHz, D_2O) δ 7.71 (d, J = 8.5 Hz, 2H), 7.39 (d, J = 8.5 Hz, 2H), 4.39 (t, J = 8.0 Hz, 1H), 3.87 (dd, J = 8.0 and 12.0 Hz, 1H), 3.26 (dd, J = 8.0 and 12.5 Hz, 1H), 3.23 (m, 2H), 3.18 (m, 2H), 2.35 (s, 3H), 1.83 (m, 2H), 1.60 (m, 2H), 1.48 (m, 2H); ^{13}C NMR (125 MHz, D_2O) 176.05, 145.76, 135.35, 130.45, 127.04, 67.36, 65.95, 64.65, 54.21, 21.60, 21.32, 20.89, 20.36; IR (Ge ATR) 3035, 2946, 1616, 1433, 1315, 1159, 1096 cm^{-1} . Anal. Calcd for $C_{15}H_{21}N_3O_3S$: C, 55.71; H, 6.54; N, 12.99. Found: C, 55.67; H, 6.51; N, 12.99.

4-Methyl-N-(2-oxo-8-oxa-1,5-diaza-spiro[4.5]dec-3-yl)-benzenesulfoamide (26): A solution of 1-(toluene-4-sulfonyl)-aziridine-2-carboxylic acid methyl ester **21** (226 mg, 0.89 mmol) and *N*-aminomorpholine (102.6 μ L, 1.06 mmol) in *i*-PrOH (1 mL) was stirred at rt for 6 h. The resulting precipitates were filtered, washed with diethyl ether, and dried under vacuum to give the product as a white solid (223 mg, 77%): m.p: 236 $^{\circ}$ C; 1H NMR (300 MHz, D_2O) δ 7.84 (d, J = 8.4 Hz, 2H), 7.52 (d, J = 7.8 Hz, 2H), 4.55 (t, J = 8.1 Hz, 1H), 4.11 (m, 3H), 3.94 (m, 2H), 3.57 (m, 3H), 3.39 (m, 2H), 2.48 (s, 3H); ^{13}C NMR (125 MHz, D_2O) 184.49, 145.80, 130.45, 129.11, 127.05, 95.01, 65.31, 64.61, 62.20, 54.01, 23.88, 20.89; IR (film) 3068, 2950, 1628, 1415, 1322, 1163 cm^{-1} . Anal. Calcd for $C_{14}H_{19}N_3O_4S$: C, 51.68; H, 5.89; N, 12.91; S, 9.85. Found: C, 51.56; H,

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6.26; N, 12.88; S, 10.06.

4-Methyl-N-(8-methyl-2-oxo-1,5,8-triaza-spiro[4.5]dec-3-yl)-

benzenesulfonamide (27): A solution of 1-(toluene-4-sulfonyl)-aziridine-2-carboxylic acid methyl ester **21** (415 mg, 1.63 mmol), 1-amino-4-methylpiperazine (235 μ L, 1.96 mmol) in *i*-PrOH (2 mL) was stirred at room temperature for 4 h. The resulting precipitates were isolated by filtration, washed with diethyl ether, and dried under vacuum to give cyclic aminimide **27** as a white solid (458 mg, 83%): m.p: 211 $^{\circ}$; 1H NMR (500 MHz, D_2O) δ 7.70 (d, J = 8.3 Hz, 2H), 7.38 (d, J = 8.3 Hz, 2H), 4.40 (t, J = 8.0 Hz, 1H), 3.92 (m, 1H), 3.38 (dd, J = 7.5 and 12.0 Hz, 1H), 3.29 (m, 4H), 2.81 (m, 2H), 2.65 (m, 2H), 2.34 (s, 3H), 2.23 (s, 3H); ^{13}C NMR (125 MHz, D_2O) 176.34, 145.77, 135.27, 130.46, 130.10, 127.05, 125.97, 65.26, 64.46, 54.06, 49.18, 43.96, 23.89, 20.89; IR (film) 3256, 3040, 2845, 2750, 1593, 1439, 1332, 1116, 1096 cm^{-1} . Anal. Calcd for $C_{15}H_{22}N_4O_3S$: C, 53.23; H, 6.55. Found: C, 53.16; H, 6.79. LRMS (ESI): m/z 360.9 (361.1 calcd for $C_{15}H_{22}N_4O_3SNa$ $[M+Na]^+$), 339.0 (339.14 calcd for $C_{15}H_{23}N_4O_3S$ $[M+H]^+$).

N-(1,1-Dimethyl-3-oxo-pyrrolidin-4-yl)-methanesulfonamide (28):

A solution of 1-methanesulfonylaziridine-2-carboxylic acid methyl ester **22** (245 mg, 1.36 mmol) and dimethyl hydrazine (125 μ L, 1.64 mmol) in *i*-PrOH (2 mL) was stirred at room temperature. The reaction mixture was stirred for 5 h. The mixture was filtered off, washed with diethyl ether and dried in a vacuum to give a product as a white solid (231 mg, 82%): m.p: 207 $^{\circ}$; 1H NMR (500 MHz, D_2O) δ 4.71 (t, J = 9.0 Hz, 1H), 4.32 (dd, J = 8.0 and 12.0 Hz, 1H), 3.68 (dd, J = 8.5 and 12 Hz, 1H), 3.21 (s, 3H), 3.15 (s, 3H), 3.10 (s, 3H); ^{13}C NMR (125 MHz, D_2O) 176.60, 68.70, 58.20, 57.24, 55.60, 40.75; IR (Ge ATR) 3331, 3137, 1616, 1400, 1367, 1311, 1154 cm^{-1} . Anal. Calcd for

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$C_6H_{13}N_3O_3S$: C, 34.77; H, 6.32; Found: C, 34.13; H, 6.14. LRMS (ESI): m/z 229.8 (230.1 calcd for $C_6H_{13}N_3O_3SNa$ $[M+Na]^+$).

***N*-(2-oxo-8-oxa-1,5-diaza-spiro[4,5]dec-3-yl)-methanesulfoamide (29):** To a solution of 1-methanesulfonyl-aziridine-2-carboxylic acid methyl ester (500 mg, 2.79 mmol), *N*-aminomorpholine (323 μ L, 1.06 mmol) in *i*-PrOH (3 mL) was stirred at room temperature for 6 h. The mixture was filtered off, washed with diethyl ether and dried in a vacuum to give a product as a white solid (536 mg, 77%): m.p: 219 $^{\circ}$ C; 1H NMR (500 MHz, D_2O) δ 4.67 (t, J = 7.5 Hz, 1H), 4.39 (dd, J = 8.5 and 12.5 Hz, 1H), 4.07 (m, 2H), 3.90 (m, 2H), 3.69 (dd, J = 8.5 and 12.0 Hz, 1H), 3.56 (m, 2H), 3.38 (m, 2H), 3.10 (s, 3H); ^{13}C NMR (125 MHz, D_2O) 176.94, 67.44, 65.39, 64.59, 62.32, 62.24, 54.31, 40.77; Anal. Calcd for $C_8H_{15}N_3O_4S$: C, 38.54; H, 6.06; N, 16.86. Found: C, 38.97; H, 5.81; N, 16.46.

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Crystallographic Data for 14

Data collection (14)

A dark orange crystal with approximate dimensions 0.30 x 0.22 x 0.10 mm³ was selected and attached to the tip of a glass fiber, transferred to a Bruker SMART diffractometer/CCD area detector and centered under liquid nitrogen in the beam at 233(2)K.

The crystal evaluation and data collection were performed on a Bruker CCD diffractometer with Mo K_α (λ = 0.71073) radiation employing 2kw sealed tube X-ray source operating at 1.6 kW and the diffractometer to crystal distance of 4.9701cm.

Preliminary orientation matrix and cell constants were determined from three series of ωscans at different starting angles. Each series consisted of 10 frames collected at intervals of 0.3°ωscan with the exposure time of 10 seconds per frame. The reflections were successfully indexed by an automated indexing routine built in the SMART program.¹

A total of 4681 data were harvested by collecting three sets of frames with 0.3°ω scans with an exposure time 10 seconds per frame. The highly redundant datasets were corrected for Lorentz and polarization effects, but no correction for crystal decay was applied.

Structure Solution and Refinement

The systematic absences in the diffraction data were consistent for the space group, Monoclinic, P2₁/c that yielded chemically reasonable³ and computationally stable results of refinement.⁴

A successful solution was obtained by the direct methods⁵ from the *E*-map. The remaining non-hydrogen atoms were located in an alternating series of least-squares cycles and difference Fourier maps. All non-hydrogen atoms were refined with anisotropic displacement coefficients. All hydrogen atoms were included in the structure factor calculation at idealized positions and were allowed to ride on the neighboring atoms with relative isotropic displacement coefficients. The final least-squares refinement of 109 parameters against 1895 data resulted in R₁(based on F_o for F_o > 4σ) and wR₂(based on F² for all data) of 0.0799 and 0.1774, respectively.

References

Supporting Information: Efficient Synthesis of Highly Functionalized Cyclic Aminimides; Moon, Han, and Kim May 31, 2005.

1. SMART Area-Detector Software Package, Bruker AXS, Inc., Madison WI, 1995.
2. SAINT; SAX Area-Detector Integration Program, version 4.050, Bruker AXS, Inc., Madison WI, 1995.
3. XPREP: part of the SHELXTL, Crystal Structure Determination Package, version 5.04, Bruker AXS, Inc., Madison WI, 1995.
4. SHELX-97 Sheldrick, G. M. Institut für Anorganische Chemie der Universität, Göttingen, F. R. G., 1997.
5. Beurskens, P. T. Admiral G. Beurskens, G. Bosman, W. P. Garcia-Granda, S. Gould, R. O. Smits, J. M. M. and Smkalla, C. DIRIF 92. The DIRIF Program System, Technical Report of the Crystallography Laboratory, University of Nijmegen, 1992.

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Ortep drawing of G012 : for clarity, hydrogens were omitted

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Table 1. Crystal data and structure refinement for **14**.

Identification code	14
Empirical formula	C ₇ H ₁₂ N ₂ O ₃
Formula weight	172.19
Temperature	233(2) K
Wavelength	0.71073 Å
Crystal system, space group	Monoclinic, P2(1)/c
Unit cell dimensions	a = 5.520(3) Å alpha = 90 deg. b = 8.698(4) Å beta = 98.120(7) deg. c = 16.224(7) Å gamma = 90 deg.
Volume	771.1(6) Å ³
Z, Calculated density	4, 1.483 Mg/m ³
Absorption coefficient	0.117 mm ⁻¹
F(000)	368
Crystal size	0.30 x 0.22 x 0.10 mm
Theta range for data collection	2.54 to 28.03 deg.
Limiting indices	-7<=h<=7, -7<=k<=11, -21<=l<=19
Reflections collected / unique	4681 / 1859 [R(int) = 0.0735]
Completeness to theta = 28.03	98.9 %
Max. and min. transmission	0.9884 and 0.9659
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	1859 / 0 / 109
Goodness-of-fit on F ²	1.066
Final R indices [I>2sigma(I)]	R ₁ = 0.0799, wR ₂ = 0.1549
R indices (all data)	R ₁ = 0.1351, wR ₂ = 0.1774
Largest diff. peak and hole	0.294 and -0.236 e. Å ⁻³

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Table 2. Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **14**. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
O(11)	3447(3)	1495(2)	3576(1)	29(1)
O(12)	8683(3)	732(2)	3412(1)	30(1)
N(1)	5844(4)	1863(2)	1716(1)	20(1)
N(2)	3799(4)	1609(3)	2193(1)	25(1)
O(8)	7517(4)	1424(2)	124(1)	33(1)
C(3)	4678(5)	1710(3)	2992(2)	21(1)
C(6)	6618(5)	318(3)	1425(2)	25(1)
C(5)	7834(5)	2661(3)	2303(2)	22(1)
C(10)	4947(5)	2800(3)	961(2)	27(1)
C(7)	8435(6)	488(4)	816(2)	31(1)
C(4)	7410(5)	2098(3)	3146(2)	21(1)
C(9)	6894(6)	2910(4)	395(2)	33(1)

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Table 3. Bond lengths [Å] and angles [deg] for **14**.

O(11)-C(3)	1.256(3)
O(12)-C(4)	1.416(3)
N(1)-N(2)	1.473(3)
N(1)-C(10)	1.495(3)
N(1)-C(6)	1.506(3)
N(1)-C(5)	1.516(3)
N(2)-C(3)	1.322(3)
O(8)-C(7)	1.421(4)
O(8)-C(9)	1.423(4)
C(3)-C(4)	1.531(4)
C(6)-C(7)	1.511(4)
C(5)-C(4)	1.503(4)
C(10)-C(9)	1.512(4)
N(2)-N(1)-C(10)	108.8(2)
N(2)-N(1)-C(6)	107.65(19)
C(10)-N(1)-C(6)	107.8(2)
N(2)-N(1)-C(5)	106.24(19)
C(10)-N(1)-C(5)	113.0(2)
C(6)-N(1)-C(5)	113.1(2)
C(3)-N(2)-N(1)	107.6(2)
C(7)-O(8)-C(9)	110.6(2)
O(11)-C(3)-N(2)	124.6(3)
O(11)-C(3)-C(4)	122.3(2)
N(2)-C(3)-C(4)	113.1(2)
N(1)-C(6)-C(7)	111.2(2)
C(4)-C(5)-N(1)	103.3(2)
N(1)-C(10)-C(9)	110.5(2)
O(8)-C(7)-C(6)	112.1(2)
O(12)-C(4)-C(5)	114.6(2)
O(12)-C(4)-C(3)	107.5(2)
C(5)-C(4)-C(3)	101.5(2)
O(8)-C(9)-C(10)	110.7(2)

Symmetry transformations used to generate equivalent atoms:

Supporting Information: Efficient Synthesis of Highly Functionalized Cyclic Aminimides; Moon, Han, and Kim May 31, 2005.

Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **14**. The anisotropic displacement factor exponent takes the form: $-2 \pi^2 [h^2 a^{*2} U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
O(11)	19(1)	41(1)	28(1)	1(1)	6(1)	-2(1)
O(12)	15(1)	34(1)	40(1)	8(1)	1(1)	1(1)
N(1)	14(1)	22(1)	25(1)	-2(1)	3(1)	1(1)
N(2)	15(1)	33(1)	27(1)	1(1)	5(1)	0(1)
O(8)	36(1)	38(1)	26(1)	-4(1)	6(1)	4(1)
C(3)	15(1)	21(2)	26(2)	0(1)	2(1)	1(1)
C(6)	26(2)	19(2)	29(2)	-3(1)	3(1)	2(1)
C(5)	14(1)	23(2)	28(2)	-3(1)	0(1)	-3(1)
C(10)	25(2)	29(2)	27(2)	0(1)	-1(1)	4(1)
C(7)	32(2)	35(2)	28(2)	-6(1)	5(1)	3(1)
C(4)	16(1)	23(2)	25(1)	0(1)	1(1)	-2(1)
C(9)	35(2)	35(2)	28(2)	2(1)	5(1)	-1(1)

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Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for **14**.

	x	y	z	U(eq)
H(12A)	10161	895	3472	45
H(6A)	5190	-234	1160	30
H(6B)	7352	-277	1901	30
H(5A)	9446	2369	2185	26
H(5B)	7670	3770	2263	26
H(10A)	4522	3824	1130	33
H(10B)	3488	2330	663	33
H(7A)	9932	937	1099	37
H(7B)	8827	-522	617	37
H(4A)	7745	2911	3565	25
H(9A)	6300	3540	-85	39
H(9B)	8341	3400	691	39

Supporting Information: Efficient Synthesis of Highly Functionalized Cyclic Aminimides; Moon, Han, and Kim May 31, 2005.

Table 6. Torsion angles [deg] for **14**.

C(10)-N(1)-N(2)-C(3)	-141.1(2)
C(6)-N(1)-N(2)-C(3)	102.3(2)
C(5)-N(1)-N(2)-C(3)	-19.1(3)
N(1)-N(2)-C(3)-O(11)	-177.2(2)
N(1)-N(2)-C(3)-C(4)	1.9(3)
N(2)-N(1)-C(6)-C(7)	170.9(2)
C(10)-N(1)-C(6)-C(7)	53.7(3)
C(5)-N(1)-C(6)-C(7)	-72.0(3)
N(2)-N(1)-C(5)-C(4)	28.4(3)
C(10)-N(1)-C(5)-C(4)	147.7(2)
C(6)-N(1)-C(5)-C(4)	-89.5(2)
N(2)-N(1)-C(10)-C(9)	-172.5(2)
C(6)-N(1)-C(10)-C(9)	-56.0(3)
C(5)-N(1)-C(10)-C(9)	69.8(3)
C(9)-O(8)-C(7)-C(6)	58.2(3)
N(1)-C(6)-C(7)-O(8)	-55.8(3)
N(1)-C(5)-C(4)-O(12)	89.7(3)
N(1)-C(5)-C(4)-C(3)	-25.7(3)
O(11)-C(3)-C(4)-O(12)	74.4(3)
N(2)-C(3)-C(4)-O(12)	-104.7(3)
O(11)-C(3)-C(4)-C(5)	-165.0(2)
N(2)-C(3)-C(4)-C(5)	15.9(3)
C(7)-O(8)-C(9)-C(10)	-60.2(3)
N(1)-C(10)-C(9)-O(8)	60.4(3)

Symmetry transformations used to generate equivalent atoms: