

## Supporting Information for:

### Selective Deprotection of Methanesulfonamides to Amines

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**General.**  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra were taken on a Varian Gemini-300 spectrometer at 300 and 75 MHz, respectively.  $\text{CDCl}_3$  was used as the solvent. Chemical shifts are reported in parts per million shift ( $\delta$  value) from  $\text{Me}_4\text{Si}$  ( $\delta$  0 ppm for  $^1\text{H}$ ) or based on the middle peak of the solvent ( $\text{CDCl}_3$ ) ( $\delta$  77.00 ppm for  $^{13}\text{C}$  NMR) as an internal standard. Signal patterns are indicated as br, broad; s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Coupling constants ( $J$ ) are given in Hertz. Infrared (IR) spectra were recorded on a JASCO A-100 spectrometer and are reported in wave numbers ( $\text{cm}^{-1}$ ). All reactions were carried out under argon, unless otherwise specified. Dry solvents (THF, diethyl ether, and  $\text{CH}_2\text{Cl}_2$ ) were purchased from Kanto Chemicals Co. (Japan). Chemicals were purified or dried in a standard manner, if necessary.

#### ***N,N*-Dioctylmethanesulfonamide (1).**

$^1\text{H}$  NMR  $\delta$  0.88 (t,  $J$  = 6.9 Hz, 6H), 1.20-1.36 (m, 20H), 1.58 (quintet,  $J$  = 6.9 Hz, 4H), 2.81 (s, 3H), 3.15 (t,  $J$  = 7.5 Hz, 4H).

$^{13}\text{C}$  NMR  $\delta$  14.05, 22.60, 26.72, 28.73, 29.18, 29.21, 31.75, 38.30, 47.81.

IR (neat) 2928, 2855, 1467, 1414, 1377, 1335, 1196, 1150, 1107, 1162, 961, 784, 723,  $523\text{ cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{17}\text{H}_{37}\text{NO}_2\text{S}$ : C, 63.90; H, 11.67. Found: C, 63.69; H, 11.75.

#### **Demesylation of 1. Dioctylamine (3).**

$^1\text{H}$  NMR  $\delta$  0.86 (t,  $J$  = 6.9 Hz, 6H), 0.88 (br s, 1H), 1.16-1.40 (m, 20H), 1.46 (quintet,  $J$  = 6.9 Hz, 4H), 2.56 (t,  $J$  = 7.2 Hz, 4H).

$^{13}\text{C}$  NMR  $\delta$  14.07, 22.64, 27.41, 29.25, 29.53, 30.19, 31.81, 50.16.

IR (neat) 3276, 2925, 2854, 2809, 1467, 1378, 1131,  $722\text{ cm}^{-1}$ .

These spectral properties and TLC mobility were in agreement with those of a commercially available authentic sample.

#### ***N*-Octyl-*N*-(10-undecenyl)methanesulfonamide (6).**

$^1\text{H}$  NMR  $\delta$  0.88 (t,  $J$  = 6.6 Hz, 3H), 1.20-1.44 (m, 22H), 1.50-1.64 (m, 4H), 2.04 (q,  $J$  = 7.2 Hz, 2H), 2.81 (s, 3H), 3.14 (t,  $J$  = 7.5 Hz, 4H), 4.89-5.03 (m, 2H), 5.81 (t/d/d,  $J$  = 6.6, 10.2, 17.1 Hz, 1H).

$^{13}\text{C}$  NMR  $\delta$  14.04, 22.59, 26.71 (2 peaks), 28.72 (2 peaks), 28.88, 29.05, 29.17, 29.20, 29.22, 29.36, 29.45, 31.75, 33.76, 38.21, 47.81 (2 peaks), 114.11, 139.16.

IR (neat) 3076, 2926, 2855, 1640, 1466, 1335, 1148, 961, 909, 785, 723,  $518\text{ cm}^{-1}$ .

Anal. Calcd for  $C_{20}H_{41}NO_2S$ : C, 66.80; H, 11.49. Found: C, 67.01; H, 11.19.

**Demesylation of 6. Octyl(10-undecenyl)amine.**

$^1H$  NMR  $\delta$  0.86 (t,  $J$  = 6.6 Hz, 3H), 1.13-1.58 (m, 26H), 1.40 (br s, 1H), 2.01 (q,  $J$  = 6.9 Hz, 2H), 2.57 (t,  $J$  = 7.2 Hz, 4H), 4.82-5.04 (m, 2H), 5.79 (t/d/d,  $J$  = 6.6, 10.2, 17.1 Hz, 1H).

$^{13}C$  NMR  $\delta$  14.03, 22.61, 27.37 (2 peaks), 28.88, 29.07, 29.23, 29.39, 29.50 (3 peaks), 30.08 (2 peaks), 31.79, 33.74, 50.07 (2 peaks), 114.04, 139.14.

IR (neat) 3320, 3076, 2925, 2854, 2810, 1641, 1617, 1466, 1377, 1339, 1131, 992, 909, 722  $cm^{-1}$ .

Anal. Calcd for  $C_{19}H_{39}N$ : C, 81.06; H, 13.96. Found: C, 81.34; H, 14.07.

These spectral properties were in agreement with those of the parent amine before mesylation.

***N*-(3,7-Dimethyl-6-octenyl)-*N*-octylmethanesulfonamide (7).**

$^1H$  NMR  $\delta$  0.88 (t,  $J$  = 7.2 Hz, 3H), 0.91 (d,  $J$  = 6.6 Hz, 3H), 1.09-1.72 (m, 17H), 1.60 (s, 3H), 1.68 (s, 3H), 1.98 (m, 2H), 2.81 (s, 3H), 3.14 (t,  $J$  = 7.5 Hz, 2H), 3.18 (t,  $J$  = 7.5 Hz, 2H), 5.07 (m, 1H).

$^{13}C$  NMR  $\delta$  14.03, 17.61, 19.34, 22.59, 25.35, 25.68, 26.69, 28.69, 29.16, 29.19, 30.24, 31.73, 35.64, 36.85, 38.32, 45.92, 47.68, 124.44, 131.40.

IR (neat) 2956, 2926, 2856, 1459, 1377, 1335, 1147, 960, 784  $cm^{-1}$ .

Anal. Calcd for  $C_{19}H_{39}NO_2S$ : C, 66.03; H, 11.37. Found: C, 65.94; H, 11.52.

**Demesylation of 7. (3,7-Dimethyl-6-octenyl)octylamine.**

$^1H$  NMR  $\delta$  0.88 (t,  $J$  = 6.6 Hz, 3H), 0.89 (d,  $J$  = 6.3 Hz, 3H), 1.08-1.72 (m, 17H), 1.40 (br s, 1H), 1.60 (s, 3H), 1.68 (s, 3H), 1.97 (m, 2H), 2.59 (t,  $J$  = 7.2 Hz, 4H), 5.09 (m, 1H).

$^{13}C$  NMR  $\delta$  14.06, 17.60, 19.60, 22.63, 25.46, 25.68, 27.40, 29.24, 29.57, 30.00, 30.68, 31.81, 37.13, 37.20, 47.92, 50.12, 124.80, 131.13.

IR (neat) 3320, 2956, 2925, 2854, 1684, 1617, 1457, 1377, 1339, 1129, 800, 722  $cm^{-1}$ .

Anal. Calcd for  $C_{18}H_{37}N$ : C, 80.82; H, 13.94. Found: C, 81.29; H, 13.52.

These spectral properties were in agreement with those of the parent amine before mesylation.

***N*-(10-Methoxydecyl)-*N*-octylmethanesulfonamide (8).**

$^1H$  NMR  $\delta$  0.88 (t,  $J$  = 7.2 Hz, 3H), 1.21-1.65 (m, 28H), 2.81 (s, 3H), 3.15 (t,  $J$  = 7.8 Hz, 4H), 3.32 (s, 3H), 3.36 (t,  $J$  = 6.6 Hz, 2H).

$^{13}C$  NMR  $\delta$  14.03, 22.59, 26.09, 26.70 (2 peaks), 28.71 (2 peaks), 29.17, 29.20, 29.21, 29.41, 29.43, 29.45, 29.61, 31.74, 38.27, 47.79 (2 peaks), 58.49, 72.91.

IR (neat) 2926, 2855, 1466, 1376, 1335, 1197, 1148, 1119, 962, 786, 723  $cm^{-1}$ .

Anal. Calcd for  $C_{20}H_{43}NO_3S$ : C, 63.61; H, 11.48. Found: C, 63.31; H, 11.52.

**Demesylation of 8. (10-Methoxydecanyl)octylamine.**

$^1H$  NMR  $\delta$  0.87 (t,  $J$  = 6.6 Hz, 3H), 1.21-1.61 (m, 28H), 1.50 (br s, 1H), 2.57 (t,  $J$  = 7.2 Hz, 4H), 3.32 (s, 3H), 3.35 (t,  $J$  = 6.6 Hz, 2H).

$^{13}C$  NMR  $\delta$  14.06, 22.63, 26.11, 27.40, 27.41, 29.25, 29.45, 29.50 (2 peaks), 29.54 (2 peaks), 29.62,

30.19 (2 peaks), 31.81, 50.15 (2 peaks), 58.49, 72.94.

IR (neat) 3310, 2925, 2854, 1466, 1378, 1196, 1121, 960, 723  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{19}\text{H}_{41}\text{NO}$ : C, 76.19; H, 13.80. Found: C, 75.77; H, 13.78.

These spectral properties were in agreement with those of the parent amine before mesylation.

***N*-(10-Chlorodecyl)-*N*-octylmethanesulfonamide (9).**

$^1\text{H}$  NMR  $\delta$  0.88 (t,  $J$  = 6.9 Hz, 3H), 1.21-1.65 (m, 26H), 1.76 (quintet,  $J$  = 6.9 Hz, 2H), 2.81 (s, 3H), 3.14 (t,  $J$  = 7.5 Hz, 4H), 3.53 (t,  $J$  = 6.6 Hz, 2H).

$^{13}\text{C}$  NMR  $\delta$  14.07, 22.61, 26.68, 26.72, 26.83, 28.72 (2 peaks), 28.81, 29.19 (2 peaks), 29.22, 29.33, 29.39, 31.75, 32.60, 38.26, 45.16, 47.78, 47.82.

IR (neat) 2928, 2855, 1466, 1375, 1335, 1148, 961, 785, 723, 651  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{19}\text{H}_{40}\text{ClNO}_2\text{S}$ : C, 59.73; H, 10.55. Found: C, 59.62; H, 10.86.

**Demesylation of 9. (10-Chlorodecyl)octylamine.**

$^1\text{H}$  NMR  $\delta$  0.87 (t,  $J$  = 6.6 Hz, 3H), 1.23-1.55 (m, 26H), 1.50 (br s, 1H), 1.76 (quintet,  $J$  = 6.9 Hz, 2H), 2.58 (t,  $J$  = 7.2 Hz, 4H), 3.52 (t,  $J$  = 6.6 Hz, 2H).

$^{13}\text{C}$  NMR  $\delta$  14.06, 22.63, 26.85, 27.37, 27.40, 28.85, 29.25, 29.37, 29.44, 29.49, 29.52, 30.30 (2 peaks), 31.81, 32.62, 45.13, 50.05, 50.07.

IR (neat) 3320, 2925, 2854, 1466, 1378, 1340, 1308, 1129, 723, 654  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{18}\text{H}_{38}\text{ClN}$ : C, 71.13; H, 12.60. Found: C, 70.86; H, 12.70.

These spectral properties were in agreement with those of the parent amine before mesylation.

***N,N*-Di(10-undecenyl)methanesulfonamide (10).**

$^1\text{H}$  NMR  $\delta$  1.20-1.44 (m, 24H), 1.51-1.65 (m, 4H), 2.04 (q,  $J$  = 7.2 Hz, 4H), 2.81 (s, 3H), 3.14 (t,  $J$  = 7.5 Hz, 4H), 4.89-5.03 (m, 4H), 5.81 (t/d/d,  $J$  = 6.6, 10.2, 17.1 Hz, 2H).

$^{13}\text{C}$  NMR  $\delta$  26.71, 28.73, 28.89, 29.06, 29.23, 29.36, 29.45, 33.76, 38.28, 47.80, 114.12, 139.16.

IR (neat) 3076, 2926, 2854, 1640, 1466, 1336, 1148, 993, 961, 908, 784  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{23}\text{H}_{45}\text{NO}_2\text{S}$ : C, 69.12; H, 11.35. Found: C, 69.22; H, 11.60.

**Demesylation of 10. Di(10-undecenyl)amine.**

$^1\text{H}$  NMR  $\delta$  1.20-1.54 (m, 28H), 1.40 (br s, 1H), 2.04 (q,  $J$  = 7.2 Hz, 4H), 2.58 (t,  $J$  = 7.2 Hz, 4H), 4.89-5.04 (m, 4H), 5.81 (t/d/d,  $J$  = 6.6, 10.2, 17.1 Hz, 2H).

$^{13}\text{C}$  NMR  $\delta$  27.41, 28.92, 29.11, 29.42, 29.53, 29.56, 30.18, 33.79, 50.15, 114.08, 139.23.

IR (neat) 3360, 3076, 2925, 2853, 1641, 1466, 1130, 992, 908, 722  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{22}\text{H}_{43}\text{N}$ : C, 82.17; H, 13.48. Found: C, 82.38; H, 13.28.

These spectral properties were in agreement with those of the parent amine before mesylation.

**2-(4-Chlorophenyl)-5-(methanesulfonyl)-2,5-diazabicyclo[2.2.1]heptane (11).**

$^1\text{H}$  NMR  $\delta$  1.96 (br d,  $J$  = 9.9 Hz, 1H), 2.08 (br d/d,  $J$  = 2.3, 9.9 Hz, 1H), 2.85 (s, 3H), 3.29 (br d,  $J$  = 9.0 Hz, 1H), 3.46 (d,  $J$  = 9.5 Hz, 1H), 3.49 (d,  $J$  = 9.5 Hz, 1H), 3.58 (d/d,  $J$  = 2.3, 9.0 Hz, 1H),

4.45 (br s, 1H), 4.56 (br s, 1H), 6.48 (d,  $J = 9.0$  Hz, 2H), 7.18 (d,  $J = 9.0$  Hz, 2H).

$^{13}\text{C}$  NMR  $\delta$  37.77, 39.12, 52.24, 57.19, 57.49, 59.68, 113.61 (2 carbons), 122.04, 129.28 (2 carbons), 144.90.

IR (nujol) 3089, 3043, 2997, 2923, 2825, 1596, 1499, 1469, 1362, 1335, 1220, 1175, 1140, 1097, 1073, 1021, 909, 810, 770, 736, 585, 546  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{12}\text{H}_{15}\text{ClN}_2\text{O}_2\text{S}$ : C, 50.26; H, 5.27. Found: C, 50.41; H, 5.36.

M.p. 200-201  $^{\circ}\text{C}$ .

#### **Demesylation of 11. 2-(4-Chlorophenyl)-2,5-diazabicyclo[2.2.1]heptane.**

$^1\text{H}$  NMR  $\delta$  1.53 (br s, 1H), 1.82 (br d,  $J = 9.3$  Hz, 1H), 1.93 (br d,  $J = 9.3$  Hz, 1H), 2.90 (d,  $J = 8.7$  Hz, 1H), 3.01 (d/d,  $J = 1.8, 9.9$  Hz, 1H), 3.08 (d,  $J = 9.9$  Hz, 1H), 3.62 (d/d,  $J = 2.1, 8.7$  Hz, 1H), 3.77 (br s, 1H), 4.24 (br s, 1H), 6.46 (d,  $J = 9.0$  Hz, 2H), 7.14 (d,  $J = 9.0$  Hz, 2H).

$^{13}\text{C}$  NMR  $\delta$  37.85, 50.14, 56.79, 57.47, 60.53, 113.97 (2 carbons), 121.28, 129.40 (2 carbons), 146.09.

IR (nujol) 3340, 2924, 2854, 1597, 1496, 1463, 1374, 1180, 1128, 1091, 1042, 968, 813, 761, 726, 637, 617, 511  $\text{cm}^{-1}$ .

These spectral properties were in agreement with those of the authentic amine prepared from its hydrobromide salt which is commercially available.

#### ***N*-Cyclohexyl-*N*-octylmethanesulfonamide (12).**

$^1\text{H}$  NMR  $\delta$  0.86 (t,  $J = 6.6$  Hz, 3H), 0.97-1.87 (m, 22H), 2.82 (s, 3H), 3.06 (t,  $J = 8.1$  Hz, 2H), 3.54 (t/t,  $J = 3.3, 10.8$  Hz, 1H).

$^{13}\text{C}$  NMR  $\delta$  14.00, 22.55, 25.23, 26.02 (2 carbons), 26.93, 29.17 (2 peaks), 31.69, 31.71, 32.10 (2 carbons), 40.29, 44.04, 57.86.

IR (neat) 2929, 2856, 1453, 1378, 1330, 1169, 1146, 1119, 1008, 960, 917, 894, 849, 814, 781, 734, 593  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{15}\text{H}_{31}\text{NO}_2\text{S}$ : C, 62.24; H, 10.79. Found: C, 62.00; H, 10.85.

#### **Demesylation of 12. Octylcyclohexylamine.**

$^1\text{H}$  NMR  $\delta$  0.87 (t,  $J = 6.6$  Hz, 3H), 0.97-1.93 (m, 22H), 1.70 (br s, 1H), 2.39 (t/t,  $J = 3.6, 10.5$  Hz, 1H), 2.56 (t,  $J = 7.5$  Hz, 2H).

$^{13}\text{C}$  NMR  $\delta$  14.06, 22.63, 25.11 (2 carbons), 26.20, 27.47, 29.25, 29.53, 30.50, 31.81, 33.66 (2 carbons), 47.08, 56.93.

IR (neat) 3309, 2925, 2853, 1450, 1367, 1347, 1259, 1133, 889, 845, 733  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{14}\text{H}_{29}\text{N}$ : C, 79.55; H, 13.83. Found: C, 79.88; H, 13.73.

These spectral properties were in agreement with those of the parent amine before mesylation.

#### ***N,N*-Dicyclohexylmethanesulfonamide (13).**

$^1\text{H}$  NMR  $\delta$  1.01-1.86 (m, 20H), 2.85 (s, 3H), 3.20-3.36 (m, 2H).

$^{13}\text{C}$  NMR  $\delta$  25.13 (2 carbons), 26.37 (4 carbons), 32.51 (4 carbons), 43.30, 57.60 (2 carbons).

IR (nujol) 2927, 2853, 1451, 1377, 1317, 1255, 1165, 1144, 1112, 1053, 983, 965, 895, 822, 774, 723, 641  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{13}\text{H}_{25}\text{NO}_2\text{S}$ : C, 60.19; H, 9.71. Found: C, 60.44; H, 10.19.

M.p. 100-100.5  $^{\circ}\text{C}$ .

#### **Demesylation of 13. Dicyclohexylamine.**

$^1\text{H}$  NMR  $\delta$  0.77 (br s, 1H), 0.89-1.94 (m, 20H), 2.52 (t/t,  $J = 3.9, 10.8$  Hz, 2H).

$^{13}\text{C}$  NMR  $\delta$  25.28 (2 carbons), 26.16, 34.33 (2 carbons), 52.98.

IR (neat) 3310, 2926, 2852, 1464, 1448, 1368, 1346, 1258, 1126, 888, 705  $\text{cm}^{-1}$ .

These spectral properties and TLC mobility were in agreement with those of a commercially available authentic sample.

#### ***N*-Cyclohexyl-*N*-cyclooctylmethanesulfonamide (14).**

$^1\text{H}$  NMR  $\delta$  1.08-2.10 (m, 24H), 2.86 (s, 3H), 3.24 (t/t,  $J = 3.3, 11.1$  Hz, 1H), 3.56 (t/t,  $J = 3.0, 10.5$  Hz, 1H).

$^{13}\text{C}$  NMR  $\delta$  25.19 (2 peaks), 26.12 (2 carbons), 26.20 (2 carbons), 26.39 (2 carbons), 32.69 (2 carbons), 34.35 (2 carbons), 43.02, 58.08, 58.16.

IR (neat) 2926, 2854, 1468, 1449, 1403, 1323, 1146, 1111, 1050, 1013, 992, 960, 894, 819, 774, 735, 621  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{15}\text{H}_{29}\text{NO}_2\text{S}$ : C, 62.67; H, 10.17. Found: C, 62.91; H, 10.28.

#### **Demesylation of 14. Cyclohexylcyclooctylamine.**

$^1\text{H}$  NMR  $\delta$  0.92-1.90 (m, 24H), 1.50 (br s, 1H), 2.46 (t/t,  $J = 3.6, 10.5$  Hz, 1H), 2.80 (br s, 1H).

$^{13}\text{C}$  NMR  $\delta$  24.19 (2 carbons), 25.30 (2 carbons), 25.80, 26.17, 27.27 (2 carbons), 33.23 (2 carbons), 34.14 (2 carbons), 53.41, 53.67.

IR (neat) 3309, 2923, 2851, 1466, 1447, 1365, 1347, 1258, 1112, 888, 733  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{14}\text{H}_{27}\text{N}$ : C, 80.31; H, 13.00. Found: C, 80.62; H, 12.95.

These spectral properties were in agreement with those of the parent amine before mesylation.

#### ***N*-[3-[*cis*-3,5-Dimethyl-4-(methanesulfonyl)piperazin-1-yl]propyl]carbazole (15).**

$^1\text{H}$  NMR  $\delta$  1.55 (d,  $J = 6.9$  Hz, 6H), 1.95-2.09 (m, 4H), 2.29 (t,  $J = 6.3$  Hz, 2H), 2.69 (d,  $J = 11.1$  Hz, 2H), 2.83 (s, 3H), 4.02 (m, 2H), 4.41 (t,  $J = 6.6$  Hz, 2H), 7.23 (m, 2H), 7.39-7.48 (m, 4H), 8.09 (d,  $J = 7.5$  Hz, 2H).

$^{13}\text{C}$  NMR  $\delta$  22.41 (2 carbons), 25.78, 40.32, 40.49, 48.87 (2 carbons), 55.23, 58.30 (2 carbons), 108.53 (2 carbons), 118.85 (2 carbons), 120.36 (2 carbons), 122.81 (2 carbons), 125.58 (2 carbons), 140.41 (2 carbons).

IR (neat) 3050, 2924, 2854, 2784, 1594, 1484, 1452, 1377, 1326, 1212, 1179, 1133, 1006, 970, 768, 750, 723, 528  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{22}\text{H}_{29}\text{N}_3\text{O}_2\text{S}$ : C, 66.13; H, 7.32. Found: C, 66.06; H, 6.98.

**Demesylation of 15. 9-[3-(*cis*-3,5-dimethyl-1-piperazinyl)propyl]carbazole.**

$^1\text{H}$  NMR  $\delta$  0.98 (d,  $J$  = 6.6 Hz, 6H), 1.34 (br s, 1H), 1.44 (d,  $J$  = 11 Hz, 2H), 1.96 (quintet,  $J$  = 6.6 Hz, 2H), 2.19 (t,  $J$  = 6.6 Hz, 2H), 2.64 (d/d,  $J$  = 2.1, 11 Hz, 2H), 2.83-2.96 (m, 2H), 4.29 (t,  $J$  = 6.6 Hz, 2H), 7.19 (m, 2H), 7.36-7.44 (m, 4H), 8.05 (d,  $J$  = 7.5 Hz, 2H).

$^{13}\text{C}$  NMR  $\delta$  19.76 (2 carbons), 25.63, 40.25, 50.47 (2 carbons), 54.78, 60.43 (2 carbons), 108.70 (2 carbons), 118.57 (2 carbons), 120.08 (2 carbons), 122.63 (2 carbons), 125.31 (2 carbons), 140.31 (2 carbons).

IR (nujol) 3195, 3051, 2926, 2806, 2763, 1626, 1591, 1485, 1453, 1369, 1344, 1319, 1250, 1207, 1154, 1140, 1073, 882, 851, 781, 753, 727, 555  $\text{cm}^{-1}$ .

These spectral properties were in agreement with those of the parent amine prepared from its dihydrochloride salt which is commercially available.

***N*-Ethyl-*N*-[6-(*N*-ethyl-*N*-methanesulfonylamino)hexyl]benzenesulfonamide (16).**

$^1\text{H}$  NMR  $\delta$  1.07 (t,  $J$  = 7.2 Hz, 3H), 1.18 (t,  $J$  = 7.2 Hz, 3H), 1.26-1.35 (m, 4H), 1.47-1.63 (m, 4H), 2.80 (s, 3H), 3.07-3.30 (m, 8H), 7.44-7.58 (m, 3H), 7.78 (m, 2H).

$^{13}\text{C}$  NMR  $\delta$  13.94, 14.05, 26.10, 26.13, 28.49, 28.58, 38.36, 42.26, 42.58, 46.96, 47.31, 126.89 (2 carbons), 128.94 (2 carbons), 132.22, 140.07.

IR (neat) 3064, 2974, 2936, 2865, 1466, 1446, 1382, 1329, 1149, 1091, 963, 921, 775, 739, 694, 579  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{17}\text{H}_{30}\text{N}_2\text{O}_4\text{S}_2$ : C, 52.28; H, 7.74. Found: C, 52.36; H, 7.51.

***N*-Ethyl-*N*-[6-(*N*-ethyl-*N*-methanesulfonylamino)hexyl]trifluoromethanesulfonamide (17).**

$^1\text{H}$  NMR  $\delta$  1.19 (t,  $J$  = 7.2 Hz, 3H), 1.23 (t,  $J$  = 7.2 Hz, 3H), 1.29-1.39 (m, 4H), 1.53-1.69 (m, 4H), 2.80 (s, 3H), 3.14 (t,  $J$  = 7.5 Hz, 2H), 3.25 (q,  $J$  = 7.2 Hz, 2H), 3.33 (br s, 2H), 3.43 (q,  $J$  = 7.2 Hz, 2H).

$^{13}\text{C}$  NMR  $\delta$  13.78, 13.99, 25.82, 25.94, 28.07, 28.53, 38.27, 42.30, 43.11, 46.88, 47.55, 119.96 (q,  $J$  = 321.8 Hz,  $\text{CF}_3$ ).

IR (neat) 2979, 2940, 2866, 1385, 1331, 1263, 1225, 1185, 1146, 1000, 963, 926, 774, 727, 693, 595  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{12}\text{H}_{25}\text{F}_3\text{N}_2\text{O}_4\text{S}_2$ : C, 37.68; H, 6.59. Found: C, 37.91; H, 6.24.

***tert*-Butyl *N*-ethyl-*N*-[6-(*N*-ethyl-*N*-methanesulfonylamino)hexyl]carbamate (18).**

$^1\text{H}$  NMR  $\delta$  1.06 (t,  $J$  = 7.2 Hz, 3H), 1.18 (t,  $J$  = 7.2 Hz, 3H), 1.23-1.35 (m, 4H), 1.43 (s, 9H), 1.49 (quintet,  $J$  = 7.2 Hz, 2H), 1.57 (quintet,  $J$  = 7.2 Hz, 2H), 2.79 (s, 3H), 3.10-3.27 (m, 4H), 3.13 (t,  $J$  = 7.5 Hz, 2H), 3.24 (q,  $J$  = 7.2 Hz, 2H).

$^{13}\text{C}$  NMR  $\delta$  13.63, 14.06, 26.38, 26.45, 28.43 ( $\text{Me}_3\text{C}$  and one more carbon), 28.72, 38.43, 41.64, 42.24, 46.42, 47.03, 78.86, 155.40.

IR (neat) 2974, 2933, 2863, 1689 (C=O), 1479, 1418, 1365, 1332, 1288, 1254, 1148, 1071, 964, 921, 865, 774, 732  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{16}\text{H}_{34}\text{N}_2\text{O}_4\text{S}$ : C, 54.83; H, 9.78. Found: C, 54.53; H, 9.72.

***N*-(Methanesulfonyl)-*N'*-(benzenesulfonyl)-1,4-piperazine (19).**

$^1\text{H}$  NMR  $\delta$  2.78 (s, 3H), 3.14 (t,  $J$  = 4.8 Hz, 4H), 3.33 (t,  $J$  = 4.8 Hz, 4H), 7.53-7.68 (m, 3H), 7.75 (m, 2H).

$^{13}\text{C}$  NMR  $\delta$  35.13, 45.22 (2 carbons), 45.73 (2 carbons), 127.62 (2 carbons), 129.32 (2 carbons), 133.34, 135.31.

IR (nujol) 3010, 2924, 2854, 1457, 1338, 1318, 1269, 1166, 1128, 1101, 945, 786, 745, 688, 579  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{11}\text{H}_{16}\text{N}_2\text{O}_4\text{S}_2$ : C, 43.40; H, 5.30. Found: C, 43.31; H, 5.70.

M.p. 197-198  $^{\circ}\text{C}$ .

***N*-(*tert*-Butoxycarbonyl)-*N'*-methanesulfonyl-1,4-piperazine (20).**

$^1\text{H}$  NMR  $\delta$  1.45 (s, 9H), 2.76 (s, 3H), 3.16 (t,  $J$  = 5.1 Hz, 4H), 3.52 (t,  $J$  = 5.1 Hz, 4H).

$^{13}\text{C}$  NMR  $\delta$  28.27 ( $\text{Me}_3\text{C}$ ), 34.55, 43.24 (2 carbons), 45.59 (2 carbons), 80.45, 154.20.

IR (nujol) 2923, 2856, 1693 (C=O), 1458, 1406, 1322, 1281, 1248, 1157, 1144, 1123, 1105, 969, 941, 787  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{10}\text{H}_{20}\text{N}_2\text{O}_4\text{S}$ : C, 45.44; H, 7.63. Found: C, 45.28; H, 7.35.

M.p. 110-111  $^{\circ}\text{C}$ .

***N*-Ethyl-*N*-[6-(ethylamino)hexyl]benzenesulfonamide (21).**

$^1\text{H}$  NMR  $\delta$  1.09 (t,  $J$  = 7.2 Hz, 3H), 1.16 (t,  $J$  = 7.2 Hz, 3H), 1.21-1.41 (m, 4H), 1.45-1.62 (m, 4H), 1.55 (br s, 1H), 2.64 (t,  $J$  = 7.2 Hz, 2H), 2.70 (q,  $J$  = 7.2 Hz, 2H), 3.13 (t,  $J$  = 7.5 Hz, 2H), 3.22 (q,  $J$  = 7.2 Hz, 2H), 7.40-7.58 (m, 3H), 7.81 (m, 2H).

$^{13}\text{C}$  NMR  $\delta$  14.02, 14.79, 26.55, 26.92, 28.66, 29.59, 42.64, 43.96, 47.49, 49.36, 127.07 (2 carbons), 128.96 (2 carbons), 132.17, 140.61.

IR (neat) 3340, 2969, 2932, 2858, 1446, 1380, 1335, 1155, 1091, 1026, 931, 739, 693, 580  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{16}\text{H}_{28}\text{N}_2\text{O}_2\text{S}$ : C, 61.50; H, 9.03. Found: C, 61.23; H, 9.48.

These spectral properties were in agreement with those of the parent amine before mesylation.

***N*-Ethyl-*N*-[6-(ethylamino)hexyl]trifluoromethanesulfonamide (22).**

$^1\text{H}$  NMR  $\delta$  1.11 (t,  $J$  = 7.2 Hz, 3H), 1.25 (t,  $J$  = 7.2 Hz, 3H), 1.30-1.41 (m, 4H), 1.37 (br s, 1H), 1.50 (quintet,  $J$  = 7.2 Hz, 2H), 1.64 (quintet,  $J$  = 7.2 Hz, 2H), 2.60 (t,  $J$  = 7.2 Hz, 2H), 2.65 (q,  $J$  = 7.2 Hz, 2H), 3.34 (br s, 2H), 3.45 (q,  $J$  = 7.2 Hz, 2H).

$^{13}\text{C}$  NMR  $\delta$  13.92, 15.29, 26.39, 26.94, 28.37, 30.08, 43.27, 44.15, 47.90, 49.66, 120.18 (q,  $J$  = 321.8 Hz,  $\text{CF}_3$ ).

IR (neat) 3314, 2932, 2859, 1387, 1226, 1183, 1133, 1035, 946, 768, 726, 692, 593  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{11}\text{H}_{23}\text{F}_3\text{N}_2\text{O}_2\text{S}$ : C, 43.41; H, 7.62. Found: C, 43.30; H, 7.27.

***tert*-Butyl *N*-ethyl-*N*-[6-(ethylamino)hexyl]carbamate (23).**

$^1\text{H}$  NMR  $\delta$  1.08 (t,  $J$  = 7.2 Hz, 3H), 1.11 (t,  $J$  = 7.2 Hz, 3H), 1.23-1.57 (m, 8H), 1.45 (s, 9H), 1.47

(br s, 1H), 2.59 (t,  $J = 7.2$  Hz, 2H), 2.64 (q,  $J = 7.2$  Hz, 2H), 3.07-3.28 (m, 4H).

$^{13}\text{C}$  NMR  $\delta$  13.65, 15.34, 26.84, 27.22, 28.49 ( $\text{Me}_3\text{C}$  and one more carbon), 30.21, 41.64, 44.19, 46.58, 49.85, 78.88, 155.47.

IR (neat) 3315, 2971, 2930, 2858, 2815, 1695 (C=O), 1479, 1457, 1417, 1365, 1285, 1255, 1162, 1069, 964, 865, 773  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{15}\text{H}_{32}\text{N}_2\text{O}_2$ : C, 66.13; H, 11.84. Found: C, 65.85; H, 12.07.

These spectral properties were in agreement with those of the parent amine before mesylation.

#### ***N*-(Benzenesulfonyl)-1,4-piperazine (24).**

$^1\text{H}$  NMR  $\delta$  1.47 (br s, 1H), 2.84-2.97 (m, 8H), 7.46-7.60 (m, 3H), 7.71 (m, 2H).

$^{13}\text{C}$  NMR  $\delta$  45.17 (2 carbons), 46.75 (2 carbons), 127.62 (2 carbons), 128.89 (2 carbons), 132.69, 135.38.

IR (nujol) 3322, 3279, 3096, 3060, 2924, 2857, 1455, 1346, 1320, 1168, 1126, 1095, 1072, 947, 886, 819, 742, 692, 577  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_{10}\text{H}_{14}\text{N}_2\text{O}_2\text{S}$ : C, 53.08; H, 6.24. Found: C, 53.13; H, 5.91.

M.p. 106.5-108  $^{\circ}\text{C}$ .

These spectral properties were in agreement with those of the parent amine before mesylation.

#### ***N*-(*tert*-Butoxycarbonyl)-1,4-piperazine (25).**

$^1\text{H}$  NMR  $\delta$  1.46 (s, 9H), 1.71 (br s, 1H), 2.81 (t,  $J = 5.1$  Hz, 4H), 3.39 (t,  $J = 5.1$  Hz, 4H).

$^{13}\text{C}$  NMR  $\delta$  28.30 ( $\text{Me}_3\text{C}$ ), 44.47 (2 carbons), 45.78 (2 carbons), 79.40, 154.71.

IR (nujol) 3324, 2925, 2870, 2816, 2735, 1700 (C=O), 1419, 1362, 1317, 1244, 1174, 1121, 1092, 1054, 1006, 928, 866, 812, 768, 570  $\text{cm}^{-1}$ .

Anal. Calcd for  $\text{C}_9\text{H}_{18}\text{N}_2\text{O}_2$ : C, 58.04; H, 9.74. Found: C, 58.22; H, 9.36.

M.p. 42-43  $^{\circ}\text{C}$ .