Stereoconvergent Arylations and Alkenylations of Unactivated Alkyl Electrophiles: The Catalytic Enantioselective Synthesis of Secondary Sulfonamides and Sulfones

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I. General Information

The following reagents were purchased and used as received: NiCl₂·glyme (Strem), ZnI₂ (Strem), and Cp₂ZrHCl (Strem). Ligands L₁ (available from Aldrich) and L₆ were prepared according to a literature procedure.¹ Grignard reagents were prepared from aryl bromides and magnesium turnings (Strem) or from aryl iodides and i-PrMgCl (Aldrich; 2.0 M in THF); on occasion, we have found purchased Grignard reagents to be less suitable. THF was deoxygenated and dried by sparging with argon followed by passage through an activated alumina column (S. G. Water) prior to use. All reactions were carried out in oven-dried glassware under an inert atmosphere.

¹H NMR data and ¹³C NMR data were collected on a VARIAN 500 MHz spectrometer at ambient temperature. HPLC analyses were carried out on an Agilent 1100 series system with Daicel CHIRALPAK® columns or Daicel CHIRALCEL® columns (internal diameter 4.6 mm, column length 250 mm, particle size 5 μm or 3 μm). GC analyses were carried out on an Agilent 6890 series system with an HP-5 column (length 30 m, I.D. 0.25 mm).

(1) Choi, J.; Fu, G. C. J. Am. Chem. Soc. 2012, 134, 9102–9105.
II. Preparation of Electrophiles

These procedures have not been optimized.

Representative experimental procedure for the preparation of α-bromosulfonamides.

LDA was prepared by the dropwise addition of n-BuLi (1.6 M in hexanes; 13.8 mL, 22 mmol) to a solution of i-Pr₂NH (3.36 mL, 24.0 mmol) in THF (71 mL) in a 500-mL round-bottom flask at −78 °C. The reaction mixture was stirred at 0 °C for 15 min, and then it was cooled to −78 °C. A solution of the 1-bromomethanesulfonamide (20.0 mmol; prepared according to a literature procedure from bromomethanesulfonyl chloride and a secondary amine\(^2\)) in THF (40.0 mL) was added over 15 min to the LDA solution at −78 °C. The mixture was stirred for 30 min, and then a solution of the alkyl bromide (26.0 mmol) in THF (43.3 mL) was added over 15 min. The solution was stirred at −78 °C for 2 h, and then it was allowed to slowly warm to r.t. The reaction mixture was stirred at r.t. for 12 h, and then the reaction was quenched by the addition of saturated aqueous NH₄Cl (100 mL). The mixture was extracted with Et₂O (3 × 50 mL), and the combined organic layers were rinsed with brine (50 mL), dried over MgSO₄, and concentrated.

**1-Bromo-N,N-dimethylpentane-1-sulfonamide.** The title compound was prepared from 1-bromo-N,N-dimethylmethanesulfonamide (5.00 g, 24.7 mmol) and 1-bromobutane (3.45 mL, 32.2 mmol). The product was purified by column chromatography (3%→20% ethyl acetate/hexanes): 3.00 g (47%). Colorless oil.

\(^1\)H NMR (500 MHz, CDCl₃) δ 4.81 (dd, 1H, \(J = 10.7, 3.1\) Hz), 3.03 (s, 6H), 2.34 (dddd, 1H, \(J = 14.4, 10.0, 5.5, 3.1\) Hz), 2.10–2.02 (m, 1H), 1.70–1.61 (m, 1H), 1.47–1.30 (m, 3H), 0.94 (t, 3H, \(J = 7.2\) Hz).

\(^13\)C NMR (126 MHz, CDCl₃) δ 63.3, 38.8, 32.9, 29.2, 21.9, 13.9.

FT-IR (neat) 2958, 2873, 2814, 1483, 1458, 1435, 1414, 1380, 1342, 1287, 1237, 1203, 1171, 1145, 1106, 1064, 973, 930, 782, 750, 734 cm\(^{-1}\).

MS (EI) \(m/z\) (M⁺) calcd for C₇H₁₆BrNO₂S: 257, found: 257.

(2) Gao, F.; Yan, X.; Zahr, O.; Larsen, A.; Vong, K.; Auclair, K. *Bioorg. Med. Chem. Lett.* 2008, 18, 5518–5522.

(3) Brienne, M.-J.; Varech, D.; Leclercq, M.; Jacques, J.; Radembino, N.; Dessalles, M.-C.; Mahuzier, G.; Gueyouche, C.; Bories, C. Loiseau, P.; Gayral, P. *J. Med. Chem.* 1987, 30, 2232–2239.
1-Bromo-N-methyl-N-phenylpentane-1-sulfonamide. The title compound was prepared from 1-bromo-N-methyl-N-phenylmethanesulfonamide (3.82 g, 14.5 mmol) and 1-bromobutane (2.02 mL, 18.8 mmol). The product was purified by column chromatography on silica gel (2%→15% ethyl acetate/hexanes) and then on C-18 silica gel (10%→100% acetonitrile/water): 3.60 g (78%). Colorless oil.

$^1$H NMR (500 MHz, CDCl$_3$) δ 7.50–7.48 (m, 2H), 7.43–7.39 (m, 2H), 7.35–7.31 (m, 1H), 4.74 (dd, 1H, $J = 10.5, 3.1$ Hz), 3.52 (s, 3H), 2.27 (dddd, 1H, $J = 14.5, 10.2, 5.3, 3.1$ Hz), 2.11–2.03 (m, 1H), 1.66–1.58 (m, 1H), 1.40–1.24 (m, 3H), 0.89 (t, 3H, $J = 7.2$ Hz).

$^{13}$C NMR (126 MHz, CDCl$_3$) δ 140.8, 129.7, 128.1, 127.3, 63.3, 42.0, 32.7, 29.1, 21.9, 13.9.

FT-IR (neat) 3062, 3039, 2957, 2931, 2872, 1595, 1493, 1466, 1453, 1351, 1270, 1237, 1183, 1143, 1106, 1068, 1026, 917, 886, 767, 725 cm$^{-1}$.

MS (ESI) m/z (M$^+$+H) calcd for C$_{12}$H$_{19}$BrNO$_2$S: 320, found: 320.

N-Benzyl-1-bromo-N-methylpentane-1-sulfonamide. The title compound was prepared from N-benzyl-1-bromo-N-methylmethanesulfonamide (3.75 g, 13.5 mmol) and 1-bromobutane (1.88 mL, 17.5 mmol). The product was purified by column chromatography (2%→15% ethyl acetate/hexanes): 2.04 g (45%). Light-yellow oil.

$^1$H NMR (500 MHz, CDCl$_3$) δ 7.39–7.35 (m, 4H), 7.34–7.30 (m, 1H), 4.84 (dd, 1H, $J = 10.7, 3.1$ Hz), 4.61 (d, 1H, $J = 14.8$ Hz), 4.36 (d, 1H, $J = 14.8$ Hz), 2.88 (s, 3H), 2.40 (dddd, 1H, $J = 14.4, 10.0, 5.6, 3.1$ Hz), 2.15–2.07 (m, 1H), 1.72–1.64 (m, 1H), 1.50–1.31 (m, 3H), 0.95 (t, 3H, $J = 7.2$ Hz).

$^{13}$C NMR (126 MHz, CDCl$_3$) δ 135.8, 128.9, 128.3, 128.3, 64.1, 55.5, 35.5, 32.9, 29.3, 21.9, 13.9.

FT-IR (neat) 3088, 3064, 3031, 2958, 2931, 2872, 1605, 1587, 1496, 1467, 1455, 1338, 1278, 1212, 1196, 1151, 1106, 1077, 1029, 994, 944, 910, 858, 787, 733 cm$^{-1}$.

MS (ESI) m/z (M$^+$+H) calcd for C$_{13}$H$_{21}$BrNO$_2$S: 334, found: 334.

1-((1-Bromopentyl)sulfonyl)pyrrolidine. The title compound was prepared from 1-((bromomethyl)sulfonyl)pyrrolidine (3.02 g, 13.2 mmol) and 1-bromobutane (1.85 mL, 17.2 mmol). The product was purified by column chromatography (2%→15% ethyl acetate/hexanes): 1.96 g (52%). Light-yellow oil.
$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 4.84 (dd, 1H, $J = 10.7, 3.1$ Hz), 3.62–3.56 (m, 2H), 3.49–3.43 (m, 2H), 2.36 (dddd, 1H, $J = 14.4, 10.0, 5.6, 3.1$ Hz), 2.11–2.03 (m, 1H), 1.99–1.94 (m, 4H), 1.70–1.61 (m, 1H), 1.48–1.30 (m, 3H), 0.94 (t, 3H, $J = 7.2$ Hz).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 63.7, 49.4, 32.7, 29.3, 26.1, 21.9, 13.9.

FT-IR (neat) 2957, 2872, 1461, 1334, 1238, 1200, 1148, 1076, 1014, 929, 781 cm$^{-1}$.

MS (EI) $m/z$ (M$^+$) calcd for C$_9$H$_{18}$BrNO$_2$: 283, found: 283.

4-((1-Bromopentyl)sulfonyl)morpholine. The title compound was prepared from 4-((bromomethyl)sulfonyl)morpholine (3.01 g, 12.3 mmol) and 1-bromobutane (1.72 mL, 16.0 mmol). The product was purified by column chromatography (2%→20% ethyl acetate/hexanes): 1.28 g (35%). White solid.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 4.72 (dd, 1H, $J = 10.7, 3.1$ Hz), 3.75–3.68 (m, 4H), 3.50–3.42 (m, 4H), 2.32 (dddd, 1H, $J = 14.3, 9.9, 5.5, 3.0$ Hz), 2.05–1.97 (m, 1H), 1.67–1.58 (m, 1H), 1.45–1.27 (m, 3H), 0.92 (t, 3H, $J = 7.2$ Hz).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 67.0, 63.8, 47.3, 32.8, 29.1, 21.8, 13.8.

FT-IR (neat) 2959, 2925, 2860, 1467, 1460, 1450, 1434, 1347, 1328, 1299, 1261, 1237, 1204, 1153, 1114, 1074, 1014, 958, 846, 778, 732 cm$^{-1}$.

MS (EI) $m/z$ (M$^+$) calcd for C$_9$H$_{18}$BrNO$_3$: 299, found: 299.

1-Bromo-\(N,N\)-dimethylnon-8-ene-1-sulfonamide. The title compound was prepared from 1-bromo-\(N,N\)-dimethylmethanesulfonamide (1.76 g, 8.71 mmol) and 8-bromo-1-octene (1.90 mL, 11.3 mmol). The product was purified by column chromatography (2%→20% ethyl acetate/hexanes): 1.30 g (35%). Colorless oil.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 5.80 (ddt, 1H, $J = 16.9, 10.2, 6.7$ Hz), 5.00 (ddt, 1H, $J = 17.1, 2.2, 1.6$ Hz), 4.94 (ddt, 1H, $J = 10.2, 2.2, 1.2$ Hz), 4.81 (dd, 1H, $J = 10.6, 3.1$ Hz), 3.03 (s, 6H), 2.33 (dddd, 1H, $J = 14.3, 10.0, 5.8, 3.1$ Hz), 2.10–2.02 (m, 3H), 1.71–1.63 (m, 1H), 1.48–1.25 (m, 7H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 139.0, 114.5, 63.3, 38.8, 33.8, 33.1, 28.8, 28.6, 27.1.

FT-IR (neat) 3075, 2923, 2852, 1640, 1479, 1454, 1414, 1340, 1285, 1204, 1143, 1063, 971, 907, 783 cm$^{-1}$.

MS (ESI) $m/z$ (M$^+$+H) calcd for C$_{11}$H$_{23}$BrNO$_2$: 312, found: 312.
1-Bromo-5-((tert-butyldimethylsilyl)oxy)-N,N-dimethylpentane-1-sulfonamide. A 250-mL round-bottom flask was charged with 1-bromo-N,N-dimethylmethanesulfonamide (0.808 g, 4.00 mmol) and toluene (24 mL). tert-Butyl(4-iodobutoxy)dimethylsilane (5.03 g, 16.0 mmol), aqueous NaOH (50% w/v; 24 mL), and benzyltriethylammonium chloride (0.911 g, 4.00 mmol) were added to the solution at r.t. The resulting mixture was stirred at r.t. for 24 h, and then water (50 mL) was added. The organic phase was separated, and the aqueous solution was extracted with ethyl acetate (2 × 25 mL). The combined organic layers were dried over MgSO\(_4\) and concentrated. The product was purified by column chromatography (hexanes→30% ethyl acetate/hexanes): 1.21 g (78%). Colorless oil.

\(^1\)H NMR (500 MHz, CDCl\(_3\)) δ 4.82 (dd, 1H, J = 10.7, 3.1 Hz), 3.62 (t, 2H, J = 6.1 Hz), 3.02 (s, 6H), 2.38–2.32 (m, 1H), 2.13–2.04 (m, 1H), 1.79–1.70 (m, 1H), 1.67–1.45 (m, 3H), 0.89 (s, 9H), 0.05 (s, 6H).

\(^13\)C NMR (126 MHz, CDCl\(_3\)) δ 63.2, 62.6, 38.8, 33.0, 31.8, 26.1, 23.7, 18.5, −5.2.

FT-IR (neat) 2952, 2929, 2885, 2856, 1471, 1462, 1389, 1343, 1287, 1256, 1205, 1146, 1127, 1106, 1006, 973, 939, 836, 812, 776, 740 cm\(^{-1}\).

MS (ESI) m/z (M\(^+\)+H) calcd for C\(_{13}\)H\(_{31}\)BrNO\(_3\)SSi: 388, found: 388.

1-Bromo-N,N-dimethyl-5-(thiophen-2-yl)pentane-1-sulfonamide. The title compound was prepared from 1-bromo-N,N-dimethylmethanesulfonamide (3.00 g, 14.8 mmol) and 2-(4-bromobutyl)thiophene (4.23 g, 19.3 mmol). The product was purified by column chromatography (3%→20% ethyl acetate/hexanes): 1.44 g (29%). Light-yellow solid.

\(^1\)H NMR (500 MHz, CDCl\(_3\)) δ 7.12 (dd, 1H, J = 5.1, 1.2 Hz), 6.92 (dd, 1H, J = 5.1, 3.4 Hz), 6.79 (dddd, 1H, J = 3.3, 1.0, 1.0, 1.0 Hz), 4.80 (dd, 1H, J = 10.5, 3.2 Hz), 3.02 (s, 6H), 2.92–2.82 (m, 2H), 2.40–2.34 (m, 1H), 2.14–2.07 (m, 1H), 1.80–1.69 (m, 3H), 1.56–1.47 (m, 1H).

\(^13\)C NMR (126 MHz, CDCl\(_3\)) δ 144.8, 126.9, 124.4, 123.2, 63.0, 38.8, 33.0, 30.9, 29.7, 26.6.

FT-IR (neat) 2935, 2857, 1480, 1454, 1414, 1340, 1286, 1203, 1180, 1145, 1063, 972, 850, 784 cm\(^{-1}\).

MS (ESI) m/z (M\(^+\)+H) calcd for C\(_{11}\)H\(_{19}\)BrNO\(_2\)S: 340, found: 340.

1-Bromo-1-cyclopentyl-N,N-dimethylmethanesulfonamide. The title compound was prepared from 1-bromo-N,N-dimethylmethanesulfonamide (3.03 g, 15.0 mmol) and cyclopentyl
4-methylbenzenesulfonate (4.69 g, 19.5 mmol). The product was purified by column chromatography (10% ethyl acetate/hexanes): 668 mg (16%). Colorless oil.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 4.99 (d, 1H, $J = 4.8$ Hz), 3.00 (s, 6H), 2.66–2.59 (m, 1H), 1.99–1.88 (m, 2H), 1.75–1.55 (m, 5H), 1.54–1.45 (m, 1H).

$^13$C NMR (126 MHz, CDCl$_3$) $\delta$ 68.5, 41.8, 38.7, 31.8, 30.0, 25.6, 25.5.

FT-IR (neat) 2947, 2869, 2812, 1481, 1452, 1413, 1333, 1284, 1205, 1180, 1142, 1063, 969, 898, 862, 786 cm$^{-1}$.

MS (El) $m/z$ (M$^+$–Br) calcd for C$_{8}$H$_{16}$NO$_{2}$S: 190, found: 190.

![1-Bromo-N,N-dicyclohexylpentane-1-sulfonamide](image)

**1-Bromo-N,N-dicyclohexylpentane-1-sulfonamide.** The title compound was prepared from 1-bromo-N,N-dicyclohexylmethanesulfonamide (2.10 g, 6.21 mmol) and 1-bromobutane (0.867 mL, 8.07 mmol). The product was purified by column chromatography (1%–8% ethyl acetate/hexanes): 2.06 g (84%). Colorless oil.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 4.57 (dd, 1H, $J = 10.6$, 2.9 Hz), 3.38–3.33 (br m, 2H), 2.35 (dddd, 1H, $J = 14.3$, 10.1, 5.3, 2.9 Hz), 2.07–2.00 (m, 1H), 1.95–1.91 (m, 2H), 1.86–1.59 (m, 13H), 1.45–1.23 (m, 7H), 1.09 (qt, 2H, $J = 13.1$, 3.5 Hz), 0.92 (t, 3H, $J = 7.2$ Hz).

$^13$C NMR (126 MHz, CDCl$_3$) $\delta$ 66.6, 59.3, 33.9, 33.3, 32.4, 29.5, 26.5, 25.4, 22.0, 13.9.

FT-IR (neat) 2931, 2855, 1467, 1454, 1401, 1381, 1329, 1275, 1256, 1235, 1188, 1166, 1142, 1101, 1074, 1048, 1027, 997, 982, 929, 917, 895, 856, 847, 824, 801, 774, 760, 749, 733 cm$^{-1}$.

MS (El) $m/z$ (M$^+$) calcd for C$_{17}$H$_{32}$BrNO$_{2}$S: 393, found: 393.

![N-Benzyl-1-bromo-N-phenyl-5-(thiophen-2-yl)pentane-1-sulfonamide](image)

**N-Benzyl-1-bromo-N-phenyl-5-(thiophen-2-yl)pentane-1-sulfonamide.** The title compound was prepared from N-benzyl-1-bromo-N-phenylmethanesulfonamide (2.70 g, 7.94 mmol) and 2-(4-bromobuty1)thiophene (2.26 g, 10.3 mmol). The product was purified by column chromatography on silica gel (2%->12% ethyl acetate/hexanes) and then preparative HPLC on C-18 silica gel (80%->100% acetonitrile/water; water was doped with 0.1% AcOH): 0.881 g (23%). White solid.

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.34–7.27 (m, 5H), 7.26–7.20 (m, 5H), 7.11 (dd, 1H, $J = 5.1$, 1.2 Hz), 6.90 (dd, 1H, $J = 5.1$, 3.4 Hz), 6.76 (dddd, 1H, $J = 3.2$, 1.0, 1.0, 1.0 Hz), 5.34 (d, 1H, $J = 14.8$ Hz), 4.75 (dd, 1H, $J = 10.5$, 3.1 Hz), 4.69 (d, 1H, $J = 14.9$ Hz), 2.88–2.78 (m, 2H), 2.38–2.31 (m, 1H), 2.21–2.13 (m, 1H), 1.79–1.65 (m, 3H), 1.51–1.43 (m, 1H).

$^13$C NMR (126 MHz, CDCl$_3$) $\delta$ 144.8, 138.1, 136.3, 129.6, 129.4, 128.7, 128.6, 128.5, 127.9, 126.9, 124.4, 123.2, 63.3, 58.9, 32.7, 30.9, 29.6, 26.6.
FT-IR (neat) 3064, 3031, 2932, 2858, 1594, 1492, 1454, 1439, 1348, 1214, 1178, 1150, 1093, 1066, 1028, 917, 868, 822, 781 cm$^{-1}$.

MS (ESI) $m/z$ (M$^+$+H) calcd for C$_{22}$H$_{25}$BrNO$_2$S$_2$: 478, found: 478.

1-Bromo-N,N-dimethylhex-5-ene-1-sulfonamide. The title compound was prepared from 1-bromo-N,N-dimethylmethanesulfonamide (4.00 g, 19.8 mmol) and 5-bromo-1-pentene (3.05 mL, 25.7 mmol). The product was purified by column chromatography (3%→15% ethyl acetate/hexanes): 2.29 g (43%). Colorless oil.

$^1$H NMR (500 MHz, CDCl$_3$) δ 5.79 (ddt, 1H, $J = 16.9, 10.2, 6.7$ Hz), 5.05 (dq, 1H, $J = 17.1, 1.7$ Hz), 5.01 (ddt, 1H, $J = 10.2, 1.9, 1.2$ Hz), 4.82 (dd, 1H, $J = 10.5, 3.2$ Hz), 3.02 (s, 6H), 2.35 (dddt, 1H, $J = 14.5, 10.2, 6.0, 3.2$ Hz), 2.19–2.04 (m, 3H), 1.84–1.75 (m, 1H), 1.60–1.51 (m, 1H).

$^{13}$C NMR (126 MHz, CDCl$_3$) δ 137.5, 115.8, 63.0, 38.8, 32.8, 32.7, 26.4.

FT-IR (neat) 3076, 2918, 1640, 1482, 1415, 1341, 1285, 1204, 1143, 1063, 970, 912, 856, 786, 738 cm$^{-1}$.

MS (ESI) $m/z$ (M$^+$+H) calcd for C$_8$H$_{17}$BrNO$_2$: 270, found: 270.

(E)-1-Bromo-N,N-dimethylhex-5-ene-1-sulfonamide-6-d. The title compound was prepared from 1-bromo-N,N-dimethylmethanesulfonamide (762 mg, 3.77 mmol) and (E)-pent-4-en-1-yl-5-d 4-methylbenzenesulfonate (1.18 g, 4.90 mmol). The product was purified by column chromatography (2%→20% ethyl acetate/hexanes): 408 mg (40%). Colorless oil.

$^1$H NMR (500 MHz, CDCl$_3$) δ 5.78 (dt, 1H, $J = 16.9, 6.5$ Hz), 5.05–5.00 (m, 1H), 4.82 (dd, 1H, $J = 10.5, 3.2$ Hz), 3.01 (s, 6H), 2.37–2.30 (m, 1H), 2.18–2.03 (m, 3H), 1.83–1.74 (m, 1H), 1.59–1.50 (m, 1H).

$^{13}$C NMR (126 MHz, CDCl$_3$) δ 137.4, 115.5 (t, $J = 24$ Hz), 63.1, 38.8, 32.8, 32.7, 26.4.

FT-IR (neat) 3028, 2949, 2862, 2264, 1621, 1483, 1455, 1435, 1414, 1342, 1287, 1204, 1183, 1144, 1064, 972, 868, 785, 744 cm$^{-1}$.

MS (ESI) $m/z$ (M$^+$+H) calcd for C$_8$H$_{16}$DBrNO$_2$: 271, found: 271.
Representative experimental procedure for the preparation of α-bromosulfones. The target molecules were prepared according to literature procedures from α-bromoketones.\textsuperscript{4,5} A 100-mL round-bottom flask was charged with the α-bromo-β-keto-sulfone (10.0 mmol) and aqueous KOH (30\% w/v; 50 mL), and the mixture was stirred at r.t. for 48 h. When the reaction was complete (monitored by TLC), the reaction mixture was extracted with dichloromethane (3 \times 30 mL). The combined organic layers were dried over MgSO\textsubscript{4} and concentrated.

\textbf{1-Bromo-1-(methylsulfonyl)pentane.} The title compound was prepared from 2-bromo-2-(methylsulfonyl)-1-phenylhexan-1-one (12.0 g, 36.0 mmol). The product was purified by column chromatography (hexanes→20\% ethyl acetate/hexanes): 8.08 g (98\%). White solid.

\textsuperscript{1}H NMR (500 MHz, CDCl\textsubscript{3}) \(\delta\) 4.61 (dd, 1H, \(J = 11.0, 3.0\) Hz), 3.09 (s, 3H), 2.43 (dddd, 1H, \(J = 14.4, 9.9, 5.7, 3.0\) Hz), 1.96 (dddd, 1H, \(J = 14.2, 11.1, 9.5, 4.4\) Hz), 1.72–1.64 (m, 1H), 1.50–1.31 (m, 3H), 0.94 (t, 3H, \(J = 7.2\) Hz).

\textsuperscript{13}C NMR (126 MHz, CDCl\textsubscript{3}) \(\delta\) 64.3, 37.6, 30.1, 29.2, 21.8, 13.8.

FT-IR (neat) 3010, 2958, 2932, 2873, 1467, 1454, 1434, 1413, 1381, 1311, 1237, 1208, 1140, 1121, 1106, 956, 928, 815, 771, 748, 735 cm\textsuperscript{-1}.

MS (ESI) \textit{m/z} (M\textsuperscript{+}+H) calcd for C\textsubscript{6}H\textsubscript{14}BrO\textsubscript{2}S: 229, found: 229.

\textbf{(Bromo(methylsulfonyl)methyl)cyclohexane.} The bromination of 2-cyclohexyl-2-(methylsulfonyl)-1-phenylethan-1-one was conducted at 60 °C, and extra KBr and H\textsubscript{2}O\textsubscript{2} were added until the reaction was complete. The title compound was prepared from 2-bromo-2-cyclohexyl-2-(methylsulfonyl)-1-phenylethan-1-one (8.13 g, 22.6 mmol). The reaction was run at 40 °C for 96 h. The product was purified by column chromatography on silica gel (5\%→30\% ethyl acetate/hexanes) and then on C-18 silica gel (10\%→100\% acetonitrile/water): 1.69 g (29\%). White solid.

\textsuperscript{4} Suryakiran, N.; Reddy, T. S.; Ashalatha, K.; Lakshman, M.; Venkateswarlu, Y. \textit{Tetrahedron Lett.} \textbf{2006}, \textit{47}, 3853–3856.

\textsuperscript{5} Suryakiran, N.; Prabhakar, P.; Reddy, T. S.; Mahesh, K. C.; Rajesh, K.; Venkateswarlu, Y. \textit{Tetrahedron Lett.} \textbf{2007}, \textit{48}, 877–881.
was purified by column chromatography (5% aqueous mixture was extracted with dichloromethane at r.t.
water (50 mL) were concentrated, and the mixture was extracted with dichloromethane (30 mL) 20×5 mm, 20%)

1H NMR (500 MHz, CDCl₃) δ 4.60 (d, 1H, J = 2.7 Hz), 3.10 (s, 3H), 2.41–2.35 (m, 1H), 2.08–2.04 (m, 1H), 1.84–1.76 (m, 2H), 1.72–1.67 (m, 1H), 1.64–1.61 (m, 1H), 1.48–1.30 (m, 4H), 1.21–1.12 (m, 1H).

13C NMR (126 MHz, CDCl₃) δ 71.0, 39.9, 37.4, 31.8, 28.5, 26.0, 25.6, 25.3.

FT-IR (neat) 3011, 2930, 1452, 1411, 1370, 1310, 1240, 1171, 1138, 1090, 1080, 1060, 1032, 968, 922, 896, 885, 848, 792, 774, 728 cm⁻¹.

MS (ESI) m/z (M⁺+H) calcd for C₉H₁₆BrO₂S: 255, found: 255.

**Benzyl benzyl(7-bromo-7-(methylsulfonyl)heptyl)carbamate.** The title compound was prepared from benzyl benzyl(7-bromo-7-(methylsulfonyl)-8-oxo-8-phenyloctyl)carbamate (3.08 g, 5.13 mmol). The product was purified by column chromatography on silica gel (10%→50% ethyl acetate/hexanes) and then on C-18 silica gel (10%→100% acetonitrile/water): 1.57 g (62%). Viscous colorless oil.

1H NMR (500 MHz, CD₂Cl₂) δ 7.39–7.20 (br m, 10H), 5.18–5.14 (m, 2H), 4.68–4.60 (m, 1H), 4.50 (s, 2H), 3.28–3.20 (m, 2H), 3.06 (s, 3H), 2.40–2.30 (br m, 1H), 1.97–1.86 (br m, 1H), 1.70–1.25 (br m, 8H).

13C NMR (126 MHz, CD₂Cl₂) δ 156.9, 156.4, 138.6, 137.6, 128.82, 128.78, 128.2, 128.05, 127.98, 127.5, 67.3, 64.7, 50.8, 50.5, 47.4, 46.7, 37.8, 30.7, 28.6, 28.3, 27.9, 27.2, 26.7.

FT-IR (neat) 3087, 3062, 3030, 2930, 2858, 1692, 1605, 1585, 1496, 1467, 1453, 1421, 1365, 1315, 1230, 1140, 1119, 1072, 1028, 955, 915, 819, 768, 733 cm⁻¹.

MS (ESI) m/z (M⁺+H) calcd for C₅₁H₆₁BrNO₅S: 496, found: 496.

**1-Bromo-1-(tert-butylsulfonyl)pentane.** A mixture of 2-bromo-1-phenylhexan-1-one (5.10 g, 20.0 mmol), 2-methyl-2-propanethiol (1.80 g, 20.0 mmol), benzyltriethylammonium bromide (0.272 g, 1.00 mmol), and NaOH (3.00 g, 75.0 mmol) in dichloromethane (40 mL) and water (40 mL) in a 250-mL round-bottom flask was stirred at r.t. for 8 h. Then, water (100 mL) was added, and the mixture was extracted with dichloromethane (3×50 mL). The combined organic layers were dried over MgSO₄ and concentrated. The residue was dissolved in MeOH (50 mL) and water (50 mL), and then oxone® (30.7 g, 100 mmol) was added. The reaction mixture was stirred at r.t. overnight, and most of the MeOH was removed under reduced pressure. The resulting aqueous mixture was extracted with dichloromethane (3×30 mL). The combined organic layers were dried over MgSO₄ and concentrated. 2-(tert-Butylsulfonyl)-1-phenylhexan-1-one was purified by column chromatography (5%→60% ethyl acetate/hexanes): 5.34 g (90%). White solid.
2-Bromo-2-(tert-butylsulfonyl)-1-phenylhexan-1-one was prepared from 2-(tert-butylsulfonyl)-1-phenylhexan-1-one following the described procedure.

The title compound was prepared from 2-bromo-2-(tert-butylsulfonyl)-1-phenylhexan-1-one (2.30 g, 6.13 mmol). The reaction was conducted at 40 °C. The product was purified by column chromatography (hexanes→20% ethyl acetate/hexanes): 1.41 g (85%). White solid.

1H NMR (500 MHz, CDCl₃) δ 4.84 (dd, 1H, J = 10.5, 3.0 Hz), 2.43 (dddd, 1H, J = 14.5, 10.2, 5.3, 2.9 Hz), 2.11–2.03 (m, 1H), 1.75–1.66 (m, 1H), 1.55 (s, 9H), 1.48–1.30 (m, 3H), 0.94 (t, 3H, J = 7.2 Hz).

13C NMR (126 MHz, CDCl₃) δ 63.3, 59.2, 30.8, 28.8, 25.2, 22.0, 13.9.

FT-IR (neat) 2959, 2933, 2873, 1479, 1467, 1399, 1366, 1305, 1192, 1167, 1118, 1104, 1020, 986, 964, 929, 801, 733 cm⁻¹.

MS (ESI) m/z (M⁺+Na) calcd for C₉H₁₉BrNaO₂S: 293, found: 293.

PhS₄n-BuBr

((1-Bromopentyl)sulfonyl)benzene. The title compound was prepared from 2-bromo-1-phenyl-1-(phenylsulfonyl)hexan-1-one (12.0 g, 30.4 mmol). The reaction was conducted at 60 °C. The product was purified by column chromatography (hexanes→20% ethyl acetate/hexanes): 8.50 g (96%). White solid.

1H NMR (500 MHz, CDCl₃) δ 7.98–7.95 (m, 2H), 7.72–7.68 (m, 1H), 7.61–7.57 (m, 2H), 4.70 (dd, 1H, J = 11.1, 2.9 Hz), 2.41 (dddd, 1H, J = 14.3, 9.9, 5.8, 2.9 Hz), 1.89 (ddddd, 1H, J = 14.1, 11.1, 9.4, 4.4 Hz), 1.67–1.58 (m, 1H), 1.45–1.26 (m, 3H), 0.91 (t, 3H, J = 7.2 Hz).

13C NMR (126 MHz, CDCl₃) δ 135.5, 134.6, 130.2, 129.2, 66.0, 31.0, 29.2, 21.8, 13.8.

FT-IR (neat) 3065, 2958, 2933, 2872, 1584, 1478, 1466, 1447, 1381, 1324, 1309, 1236, 1203, 1149, 1133, 1083, 1024, 999, 929, 792, 778, 746 cm⁻¹.

MS (ESI) m/z (M⁺+H) calcd for C₁₁H₁₆BrO₂S: 291, found: 291.

III. Enantioselective Arylations

General Procedure. An oven-dried 8-mL vial equipped with a magnetic stir bar was capped with a PTFE-lined septum cap, cooled under vacuum, and then filled with nitrogen. ZnI₂ (290 mg, 0.910 mmol) was added to the vial, and the vial was then immediately placed under vacuum and refilled with nitrogen (three cycles). Next, THF (2.73 mL) was added to the vial, followed by a solution of ArMgBr (prepared according to a literature procedure;¹ 1.00 M in THF; 0.910 mL, 0.910 mmol). The mixture was stirred at r.t. for 30 min. An oven-dried 20-mL vial equipped with a magnetic stir bar was charged with NiCl₂-glyme (15.4 mg, 0.070 mmol), (R,R)-L¹ (30.4 mg, 0.091 mmol), and the electrophile (0.70 mmol). The vial was sealed with a PTFE-lined septum cap, placed under vacuum, and then filled with nitrogen; this cycle was repeated three times. THF (4.14 mL) was added, and the mixture was stirred at r.t. for 20 min, at which
time it had become homogenous. Both vials were wrapped with electrical tape, attached with nitrogen-filled balloons, and cooled to –20 °C for 15 min. The heterogeneous mixture of the nucleophile was then transferred by syringe over 2 min to the vial that contained the electrophile. The nitrogen-filled balloon was removed, and the septum cap was covered with grease. The reaction mixture was stirred at –20 °C for 24 h, and then the reaction was quenched by the addition of ethanol (0.70 mL). The solution was allowed to warm to r.t., and then it was filtered through a pad of silica (eluted with Et₂O). The filtrate was concentrated, and the residue was purified by column chromatography.

A second run was conducted with (S,S)-L1.

(S)-N,N-Dimethyl-1-phenylpentane-1-sulfonamide (Table 2, Entry 1). 1-Bromo-N,N-dimethylpentane-1-sulfonamide (181 mg, 0.700 mmol) and phenylzinc iodide (0.910 mmol) were used. The product was purified by column chromatography (20% → 25% Et₂O/hexanes). Light-yellow solid. First run: 159 mg (89%, 96% ee). Second run: 162 mg (91%, 96% ee).

The ee was determined by HPLC on a CHIRALCEL OD-H column (1% i-PrOH/hexanes, 1.0 mL/min) with tᵣ = 10.9 min (major), 13.4 min (minor).

1H NMR (500 MHz, CDCl₃) δ 7.42–7.34 (m, 5H), 4.08 (dd, 1H, J = 11.3, 3.8 Hz), 2.53 (s, 6H), 2.34 (dddd, 1H, J = 13.7, 10.2, 6.5, 3.8 Hz), 2.15 (dddd, 1H, J = 13.6, 11.4, 10.0, 5.1 Hz), 1.38–1.23 (m, 2H), 1.22–1.09 (m, 2H), 0.84 (t, 3H, J = 7.3 Hz).

13C NMR (126 MHz, CDCl₃) δ 133.9, 129.6, 129.0, 128.9, 67.7, 37.8, 29.6, 28.9, 22.4, 13.9.

FT-IR (neat) 3017, 2952, 2930, 2872, 1497, 1455, 1326, 1305, 1288, 1204, 1137, 1109, 1064, 973, 820, 808 cm⁻¹.

MS (ESI) m/z (M⁺Na) calcd for C₁₃H₂₁NNaO₂S: 278, found: 278.

[α]D²⁵ = −30° (c = 1.02, CHCl₃).

(S)-N-Methyl-N,1-diphenylpentane-1-sulfonamide (Table 2, Entry 2). 1-Bromo-N-methyl-N-phenylpentane-1-sulfonamide (224 mg, 0.700 mmol) and phenylzinc iodide (0.910 mmol) were used. The product was purified by column chromatography (10% Et₂O/hexanes). Light-yellow solid. First run: 211 mg (95%, 93% ee). Second run: 211 mg (95%, 95% ee).

The ee was determined by HPLC on a CHIRALCEL OD-H column (2% i-PrOH/hexanes, 1.0 mL/min) with tᵣ = 10.5 min (major), 11.7 min (minor).

1H NMR (500 MHz, CDCl₃) δ 7.41–7.35 (m, 5H), 7.31–7.27 (m, 2H), 7.21–7.18 (m, 1H), 7.17–7.14 (m, 2H), 4.11 (dd, 1H, J = 11.4, 3.7 Hz), 2.88 (s, 3H), 2.32 (dddd, 1H, J = 13.6, 10.1, 6.5, 3.7
Hz), 2.14 (dddd, 1H, J = 13.4, 11.4, 9.9, 5.2 Hz), 1.34–1.18 (m, 2H), 1.17–1.03 (m, 2H), 0.80 (t, 3H, J = 7.3 Hz).

$^{13}$C NMR (126 MHz, CDCl$_3$) δ 141.7, 133.7, 129.9, 129.1, 129.0, 128.8, 126.5, 125.8, 68.2, 39.2, 30.0, 28.9, 22.4, 13.9.

FT-IR (neat) 3063, 3030, 2957, 2932, 2872, 1596, 1493, 1455, 1423, 1380, 1342, 1266, 1179, 1143, 1108, 1067, 1028, 1003, 969, 917, 880, 801, 765 cm$^{-1}$.

MS (ESI) m/z (M$^+$+Na) calcd for C$_{19}$H$_{23}$NNaO$_2$: 340, found: 340. [$\alpha$]$^D_{25}$ = −105° (c = 1.01, CHCl$_3$).

(S)-N-Benzyl-N-methyl-1-phenylpentane-1-sulfonamide (Table 2, Entry 3). N-Benzyl-1-bromo-N-methylpentane-1-sulfonamide (234 mg, 0.700 mmol) and phenylzinc iodide (0.910 mmol) were used. The product was purified by column chromatography (7% ethyl acetate/hexanes). Light-yellow solid. First run: 219 mg (94%, 94% ee). Second run: 221 mg (95%, 93% ee).

The ee was determined by HPLC on a CHIRALPAK AD-H column (2% i-PrOH/hexanes, 1.0 mL/min) with t$_r$ = 25.8 min (major), 28.9 min (minor).

$^1$H NMR (500 MHz, CDCl$_3$) δ 7.42–7.36 (m, 5H), 7.31–7.23 (m, 3H), 7.22–7.18 (m, 2H), 4.12 (dd, 1H, J = 11.3, 3.8 Hz), 4.01 (d, 1H, J = 14.7 Hz), 3.68 (br d, 1H, J = 11.0 Hz), 2.42 (s, 3H), 2.38 (dddd, 1H, J = 13.7, 10.1, 6.2, 3.8 Hz), 2.21 (dddd, 1H, J = 13.5, 11.3, 9.8, 5.2 Hz), 1.42–1.26 (m, 2H), 1.26–1.12 (m, 2H), 0.85 (t, 3H, J = 7.3 Hz).

$^{13}$C NMR (126 MHz, CDCl$_3$) δ 136.3, 133.9, 129.7, 129.0, 128.9, 128.6, 128.3, 127.9, 68.5, 54.2, 34.6, 29.6, 29.0, 22.4, 13.9.

FT-IR (neat) 3063, 3030, 2954, 2930, 2870, 1495, 1454, 1363, 1327, 1214, 1149, 1133, 1075, 1003, 944, 890, 807, 760 cm$^{-1}$.

MS (ESI) m/z (M$^+$+Na) calcd for C$_{19}$H$_{23}$NNaO$_2$: 354, found: 354. [$\alpha$]$^D_{25}$ = −54° (c = 1.03, CHCl$_3$).

(S)-1-((1-Phenylpentyl)sulfonyl)pyrrolidine (Table 2, Entry 4). 1-((1-Bromopentyl)sulfonyl)pyrrolidine (199 mg, 0.700 mmol) and phenylzinc iodide (0.910 mmol) were used. The product was purified by column chromatography (10% ethyl acetate/hexanes). White solid. First run: 166 mg (84%, 96% ee). Second run: 170 mg (86%, 96% ee).

The ee was determined by HPLC on a CHIRALPAK AD-H column (2% i-PrOH/hexanes, 1.0 mL/min) with t$_r$ = 13.8 min (minor), 20.2 min (major).
$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.43–7.39 (m, 2H), 7.39–7.33 (m, 3H), 4.11 (dd, 1H, $J$ = 11.3, 3.8 Hz), 3.21–3.13 (m, 2H), 2.84–2.77 (m, 2H), 2.33 (dddd, 1H, $J$ = 13.8, 10.1, 6.4, 3.8 Hz), 2.17 (dddd, 1H, $J$ = 13.5, 11.3, 9.6, 5.2 Hz), 1.74–1.67 (m, 2H), 1.67–1.58 (m, 2H), 1.39–1.24 (m, 2H), 1.24–1.10 (m, 2H), 0.84 (t, 3H, $J$ = 7.3 Hz).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 134.3, 129.7, 128.8, 128.7, 67.7, 48.2, 29.2, 29.0, 25.9, 22.4, 13.9.

FT-IR (neat) 3436, 2957, 2887, 2872, 2857, 1498, 1467, 1456, 1325, 1294, 1240, 1198, 1143, 1128, 1084, 1015, 829, 806, 728 cm$^{-1}$.

MS (ESI) m/z (M$^+$+Na) calcd for C$_{15}$H$_{23}$NNaO$_3$: 304, found: 304.

$[\alpha]^D_{25}$ = −51° (c = 0.97, CHCl$_3$).

(S)-4-((1-Phenylpentyl)sulfonyl)morpholine (Table 2, Entry 5). 4-((1-Bromopentyl)sulfonyl)morpholine (210 mg, 0.700 mmol) and phenylzinc iodide (0.910 mmol) were used. The product was purified by column chromatography (20% ethyl acetate/hexanes). White solid. First run: 197 mg (95%, 98% ee). Second run: 186 mg (89%, 95% ee).

The ee was determined by HPLC on a CHIRALCEL OD-H column (3% $i$-PrOH/hexanes, 1.0 mL/min) with $t_r$ = 13.9 min (major), 16.7 min (minor).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.44–7.35 (m, 5H), 4.02 (dd, 1H, $J$ = 11.3, 3.8 Hz), 3.56–3.52 (m, 2H), 3.48–3.43 (m, 2H), 3.06–3.02 (m, 2H), 2.75 (br s, 2H), 2.34 (dddd, 1H, $J$ = 13.8, 10.1, 6.3, 3.8 Hz), 2.13 (dddd, 1H, $J$ = 13.4, 11.3, 9.8, 5.1 Hz), 1.39–1.23 (m, 2H), 1.23–1.08 (m, 2H), 0.84 (t, 3H, $J$ = 7.3 Hz).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 133.5, 129.7, 129.2, 129.0, 68.5, 67.0, 46.3, 29.7, 28.9, 22.4, 13.9.

FT-IR (neat) 2955, 2923, 2859, 1496, 1455, 1336, 1323, 1257, 1214, 1152, 1128, 1076, 955, 924, 848, 803 cm$^{-1}$.

MS (ESI) m/z (M$^+$+Na) calcd for C$_{15}$H$_{23}$NNaO$_3$: 320, found: 320.

$[\alpha]^D_{25}$ = −34° (c = 1.02, CHCl$_3$).

(S)-N,N-Dimethyl-1-phenylnonen-8-ene-1-sulfonamide (Table 2, Entry 6). 1-Bromo-N,N-dimethyl-1-phenylnonen-8-ene-1-sulfonamide (219 mg, 0.700 mmol) and phenylzinc iodide (0.910 mmol) were used. The product was purified by column chromatography (5%→10% ethyl acetate/hexanes). Light-yellow solid. First run: 192 mg (89%, 95% ee). Second run: 189 mg (87%, 95% ee).

The ee was determined by HPLC on a CHIRALCEL OD-H column (1% $i$-PrOH/hexanes, 1.0 mL/min) with $t_r$ = 12.8 min (major), 20.6 min (minor).
\(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.41–7.34 (m, 5H), 5.76 (dddd, 1H, \(J = 16.9, 10.2, 6.7, 6.7\) Hz), 4.96 (dddd, 1H, \(J = 17.1, 2.2, 1.6, 1.6\) Hz), 4.91 (dddd, 1H, \(J = 10.2, 2.3, 1.2, 1.2\) Hz), 4.08 (dd, 1H, \(J = 11.3, 3.9\) Hz), 2.53 (s, 6H), 2.30 (dddd, 1H, \(J = 13.7, 10.2, 6.5, 3.9\) Hz), 2.19–2.11 (m, 1H), 2.01–1.96 (m, 2H), 1.35–1.11 (m, 8H).

\(^13\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 139.1, 133.9, 129.6, 129.0, 128.9, 114.4, 67.7, 37.8, 32.4, 29.7, 26.0, 23.1, 18.4, –5.2.

FT-IR (neat) 3062, 2924, 2853, 1640, 1497, 1468, 1456, 1414, 1327, 1208, 1137, 1066, 977, 912, 824 cm\(^{-1}\).

MS (ESI) \(m/z\) (M\(^{+}\)+Na) calcd for C\(_{17}\)H\(_{27}\)NNaO\(_2\): 332, found: 332. \([\alpha]_{D}^{25} = -19.2^\circ\) (c = 0.98, CHCl\(_3\)).

\((S)-5-((\text{tert-Butyldimethylsilyl})\text{oxy})-N,N\text{-dimethyl-1-phenylpentane-1-sulfonamide}\) (Table 2, Entry 7). 1-Bromo-5-((\text{tert-Butyldimethylsilyl})\text{oxy})-N,N\text{-dimethylpentane-1-sulfonamide} (272 mg, 0.700 mmol) and phenylzinc iodide (0.910 mmol) were used. The product was purified by column chromatography (2\%–20\% ethyl acetate/hexanes). White solid. First run: 248 mg (92\%, >99\% ee). Second run: 250 mg (93\%, 98\% ee).

The ee was determined by HPLC on a CHIRALCEL OD-H column (2\% i-PrOH/hexanes, 1.0 mL/min) with \(t_r = 8.2\) min (major), 11.0 min (minor).

\(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta\) 7.41–7.33 (m, 5H), 4.09 (dd, 1H, \(J = 11.3, 3.9\) Hz), 3.56–3.49 (m, 2H), 2.53 (s, 6H), 2.35–2.28 (m, 1H), 2.21–2.13 (m, 1H), 1.57–1.42 (m, 2H), 1.27–1.18 (m, 2H), 0.82 (s, 9H), –0.02 (s, 6H).

\(^13\)C NMR (126 MHz, CDCl\(_3\)) \(\delta\) 133.7, 129.6, 129.0, 128.9, 67.6, 62.7, 37.8, 32.4, 29.7, 26.0, 23.1, 18.4, –5.2.

FT-IR (neat) 3065, 2931, 2897, 2860, 1458, 1385, 1359, 1329, 1280, 1257, 1200, 1143, 1132, 1110, 1092, 966, 900, 872, 833, 808, 779, 736 cm\(^{-1}\).

MS (ESI) \(m/z\) (M\(^{+}\)+Na) calcd for C\(_{19}\)H\(_{35}\)NNaO\(_3\)Si: 408, found: 408. \([\alpha]_{D}^{25} = -15.0^\circ\) (c = 0.98, CHCl\(_3\)).

\((S)-N,N\text{-Dimethyl-1-phenyl-5-(thiophen-2-yl)pentane-1-sulfonamide}\) (Table 2, Entry 8). 1-Bromo-\(N,N\text{-dimethyl-5-(thiophen-2-yl)pentane-1-sulfonamide}\) (238 mg, 0.700 mmol) and phenylzinc iodide (1.05 mmol) were used. The product was purified by column chromatography (10\%–15\% ethyl acetate/hexanes). Yellow solid. First run: 128 mg (54\%, 90\% ee). Second run: 131 mg (55\%, 91\% ee).
The ee was determined by HPLC on a CHIRALCEL OD-H column (5% i-PrOH/hexanes, 1.0 mL/min) with $t_r = 18.3$ min (major), 22.8 min (minor).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.41–7.34 (m, 5H), 7.08 (dd, 1H, $J = 5.1, 1.2$ Hz), 6.88 (dd, 1H, $J = 5.1, 3.4$ Hz), 6.71 (ddddd, 1H, $J = 3.3, 1.0, 1.0, 1.0$ Hz), 4.08 (dd, 1H, $J = 11.2, 3.9$ Hz), 2.82–2.70 (m, 2H), 2.52 (s, 6H), 2.39–2.32 (m, 1H), 2.23–2.15 (m, 1H), 1.74–1.61 (m, 2H), 1.35–1.21 (m, 2H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 145.1, 133.8, 129.6, 129.0, 128.9, 126.8, 124.2, 123.0, 67.6, 37.8, 31.4, 29.65, 29.59, 26.2.

FT-IR (neat) 3064, 2932, 2856, 1495, 1480, 1454, 1331, 1282, 1200, 1140, 1062, 1030, 967, 849, 820 cm$^{-1}$.

MS (EI) $m/z$ (M$^+$+Na) calcd for C$_{17}$H$_{23}$NNaO$_2$S$_2$: 360, found: 360.

$[\alpha]_{25}^{D} = -9.4^\circ$ (c = 0.99, CHCl$_3$).

(S)-1-Cyclopentyl-$N,N$-dimethyl-$1$-phenylmethanesulfonamide (Table 2, Entry 9). 1-Bromo-1-cyclopentyl-$N,N$-dimethylmethanesulfonamide (189 mg, 0.700 mmol) and phenylzinc iodide (1.05 mmol) were used. The product was purified by column chromatography (first purification: 10% ethyl acetate/hexanes; second purification: 12%→100% dichloromethane/hexanes). White solid. First run: 86 mg (46%, >99% ee). Second run: 79 mg (42%, >99% ee).

The ee was determined by HPLC on a CHIRALCEL OD-H column (1% i-PrOH/hexanes, 1.0 mL/min) with $t_r = 12.7$ min (major), 14.6 min (minor).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.41–7.38 (m, 2H), 7.37–7.31 (m, 3H), 3.92 (d, 1H, $J = 10.3$ Hz), 2.78–2.69 (m, 1H), 2.43 (s, 6H), 2.29–2.22 (m, 1H), 1.75–1.67 (m, 1H), 1.66–1.60 (m, 1H), 1.59–1.41 (m, 4H), 1.03–0.95 (m, 1H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 135.2, 129.7, 128.69, 128.67, 73.2, 41.8, 37.6, 32.3, 32.1, 25.5, 24.1.

FT-IR (neat) 3090, 3064, 3025, 2960, 2871, 2812, 1496, 1479, 1452, 1323, 1293, 1206, 1188, 1131, 1081, 1063, 1030, 1003, 969, 911, 872, 848, 807, 732 cm$^{-1}$.

MS (EI) $m/z$ (M$^+$–SO$_2$NMe$_2$) calcd for C$_{12}$H$_{15}$: 159, found: 159.

$[\alpha]_{D}^{25} = -43^\circ$ (c = 1.04, CHCl$_3$).

(S)-(1-(Methy1sulfonyl)pentyl)benzene (Table 3, Entry 1). 1-Bromo-1-(methylsulfonyl)pentane (160 mg, 0.700 mmol) and phenylzinc iodide (0.910 mmol) were used. The product was purified by column chromatography (20%→30% ethyl acetate/hexanes). White solid. First run: 150 mg (95%, 94% ee). Second run: 153 mg (97%, 94% ee).
The ee was determined by HPLC on a CHIRALCEL OD-H column (5% *i*-PrOH/hexanes, 1.0 mL/min) with $t_r = 17.6$ min (major), 20.8 min (minor).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.42–7.37 (m, 5H), 3.99 (dd, 1H, $J = 11.5, 3.7$ Hz), 2.59 (s, 3H), 2.45–2.37 (m, 1H), 2.12 (ddddd, 1H, $J = 13.6, 11.5, 9.6, 5.3$ Hz), 1.41–1.26 (m, 2H), 1.25–1.14 (m, 2H), 0.85 (t, 3H, $J = 7.1$ Hz).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 133.4, 129.5, 129.28, 129.27, 70.4, 38.7, 28.9, 26.7, 22.4, 13.9.

FT-IR (neat) 3088, 3065, 3051, 3011, 2931, 2869, 1496, 1468, 1456, 1417, 1379, 1292, 1277, 1263, 1211, 1158, 1130, 1107, 1072, 1036, 966, 936, 904, 805, 722 cm$^{-1}$.

MS (ESI) $m/z$ (M$^+$+Na) calcld for C$_{12}$H$_{18}$NaO$_2$S: 249, found: 249.

$[\alpha]_D^{25} = -6.2^\circ$ (c = 1.00, CHCl$_3$).

(S)-(Cyclohexyl(methylsulfonyl)methyl)benzene (Table 3, Entry 2).

(Bromo(methylsulfonyl)methyl)cyclohexane (179 mg, 0.700 mmol) and phenylzinc iodide (1.05 mmol) were used. The product was purified by column chromatography (10%→15% ethyl acetate/hexanes). White solid. First run: 145 mg (82%, 99% ee). Second run: 148 mg (84%, 99% ee).

The ee was determined by HPLC on a CHIRALPAK AD-H column (4% *i*-PrOH/hexanes, 1.0 mL/min) with $t_r = 16.7$ min (minor), 25.8 min (major).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.43–7.36 (m, 5H), 3.87 (d, 1H, $J = 7.9$ Hz), 2.53–2.45 (m, 1H), 2.46 (s, 3H), 2.29–2.24 (m, 1H), 1.80–1.74 (m, 1H), 1.67–1.56 (m, 3H), 1.42–1.33 (m, 1H), 1.28–1.18 (m, 2H), 1.14–1.05 (m, 1H), 0.93–0.85 (m, 1H).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 133.9, 129.8, 129.2, 129.1, 75.9, 41.4, 38.1, 32.4, 30.6, 26.11, 26.06, 26.0.

FT-IR (neat) 3004, 2930, 2853, 1496, 1454, 1413, 1378, 1348, 1319, 1302, 1292, 1244, 1221, 1170, 1127, 1076, 1036, 970, 896, 854, 804, 742 cm$^{-1}$.

MS (ESI) $m/z$ (M$^+$+Na) calcld for C$_{14}$H$_{20}$NaO$_2$S: 275, found: 275.

$[\alpha]_D^{25} = -40^\circ$ (c = 1.06, CHCl$_3$).

Benzyl (S)-benzyl(7-(methylsulfonyl)-7-phenylheptyl)carbamate (Table 3, Entry 3). Benzyl benzyl(7-bromo-7-(methylsulfonyl)heptyl)carbamate (199 mg, 0.400 mmol) and phenylzinc iodide (0.520 mmol) were used. The product was purified by column chromatography on silica gel (25% ethyl acetate/hexanes) and then preparative HPLC on C-18 silica gel (80%→100% acetonitrile/water; water was doped with 0.1% AcOH). Viscous colorless oil. First run: 149 mg (75%, 89% ee). Second run: 145 mg (73%, 91% ee).
The ee was determined by HPLC on a CHIRALCEL OD-H column (20% i-PrOH/hexanes, 1.0 mL/min) with t_r = 31.5 min (major), 40.0 min (minor).

^1^H NMR (500 MHz, CD_2Cl_2) δ 7.44–7.17 (m, 15H), 5.14–5.12 (m, 2H), 4.45 (s, 2H), 4.01–3.95 (m, 1H), 3.22–3.15 (m, 2H), 2.59 (s, 3H), 2.35–2.23 (br m, 1H), 2.12–1.99 (br m, 1H), 1.48–1.40 (br m, 2H), 1.35–1.09 (br m, 6H).

^13^C NMR (126 MHz, CD_2Cl_2) δ 156.9, 156.3, 138.7, 137.6, 133.6, 129.9, 129.40, 129.36, 128.8, 128.7, 128.2, 128.01, 127.95, 127.5, 70.3, 67.3, 50.8, 50.4, 47.4, 46.7, 38.9, 29.2, 28.4, 27.9, 27.4, 26.9, 26.8.

FT-IR (neat) 3088, 3063, 3031, 3007, 2931, 2858, 1697, 1605, 1586, 1496, 1454, 1422, 1366, 1305, 1232, 1137, 1086, 1071, 1029, 954, 916, 801 cm^-1.

MS (ESI) m/z (M^+H) calcd for C_{29}H_{36}NO_4S: 494, found: 494.

[α]_D^{25} = −0.037° (c = 4.1, CHCl_3).

(S)-(1-(tert-Butylsulfonyl)pentyl)benzene (Table 3, Entry 4). 1-Bromo-1-(tert-butylsulfonyl)pentane (190 mg, 0.700 mmol) and phenylzinc iodide (0.910 mmol) were used. The product was purified by column chromatography (15% ethyl acetate/hexanes). White solid. First run: 179 mg (95%, 99% ee). Second run: 182 mg (97%, 98% ee).

The ee was determined by HPLC on a CHIRALPAK IB-3 column (1% i-PrOH/hexanes, 1.0 mL/min) with t_r = 8.4 min (major), 9.9 min (minor).

^1^H NMR (500 MHz, CDCl_3) δ 7.47–7.45 (m, 2H), 7.39–7.32 (m, 3H), 4.14 (dd, 1H, J = 11.6, 3.3 Hz), 2.47 (dddd, 1H, J = 13.6, 10.6, 6.2, 3.3 Hz), 2.06 (dddd, 1H, J = 13.4, 11.6, 10.2, 4.9 Hz), 1.40–1.30 (m, 1H), 1.29–1.21 (m, 1H), 1.16 (s, 9H), 1.15–1.01 (m, 2H), 0.82 (t, 3H, J = 7.3 Hz).

^13^C NMR (126 MHz, CDCl_3) δ 134.9, 129.6, 129.0, 128.9, 65.3, 62.1, 28.9, 28.7, 24.4, 22.4, 13.9.

FT-IR (neat) 3082, 2966, 2872, 1497, 1466, 1455, 1366, 1279, 1190, 1115, 1100, 782 cm^{-1}.

MS (ESI) m/z (M^+Na) calcd for C_{15}H_{24}NaO_2S: 291, found: 291.

[α]_D^{25} = −20.3° (c = 1.01, CHCl_3).

(S)-((1-Phenylpentyl)sulfonyl)benzene (Table 3, Entry 5). ((1-Bromopentyl)sulfonyl)benzene (204 mg, 0.700 mmol) and phenylzinc iodide (0.910 mmol) were used. The product was purified by column chromatography (10%→20% Et_2O/hexanes). White solid. First run: 195 mg (97%, 86% ee). Second run: 193 mg (96%, 83% ee).

The ee was determined by HPLC on a CHIRALCEL OD-H column (1% i-PrOH/hexanes, 1.0 mL/min) with t_r = 13.6 min (major), 18.7 min (minor).
\[ ^1H \text{NMR (500 MHz, CDCl}_3 \delta 7.54-7.49 (m, 3H), 7.38-7.34 (m, 2H), 7.29-7.26 (m, 1H), 7.24-7.20 (m, 2H), 7.10-7.07 (m, 2H), 4.01 (dd, 1H, } J = 11.6, 3.6 \text{ Hz), 2.46-2.39 (m, 1H), 2.20-2.10 (m, 1H), 1.38-1.23 (m, 2H), 1.22-1.13 (m, 2H), 0.83 (t, 3H, } J = 7.3 \text{ Hz).} \]

\[ ^13C \text{NMR (126 MHz, CDCl}_3 \delta 137.6, 133.5, 132.6, 130.0, 129.2, 128.8, 128.7, 128.6, 71.8, 29.0, 27.1, 22.4, 13.9.} \]

FT-IR (neat) 2952, 2926, 2857, 1584, 1496, 1467, 1455, 1447, 1379, 1316, 1304, 1294, 1214, 1147, 1084, 1070, 1037, 968, 800, 758, 713 cm\(^{-1}\).

MS (ESI) \( m/z \) (M\(^+\)+Na) calcd for C\(_{17}\)H\(_{20}\)NaO\(_2\)S: 311, found: 311.

\([\alpha]^{25}_D = -78^\circ \) (c = 1.08, CHCl\(_3\)).

\( (S)\)-\( N\)-\( N\)-Dimethyl-1-(4-tolyl)pentane-1-sulfonamide (Table 4, Entry 1). \( 1\)-Bromo-\( N\)-\( N\)-dimethylpentane-1-sulfonamide (181 mg, 0.700 mmol) and 4-tolylzinc iodide (0.910 mmol) were used. The product was purified by column chromatography (20\% Et\(_2\)O/hexanes). Light-yellow oil. First run: 172 mg (91\%, 96\% ee). Second run: 165 mg (87\%, 95\% ee).

The ee was determined by HPLC on a CHIRALCEL OD-H column (2\% i-PrOH/hexanes, 1.0 mL/min) with \( t_r \) = 8.1 min (major), 10.1 min (minor).

\[ ^1H \text{NMR (500 MHz, CDCl}_3 \delta 7.30-7.27 (m, 2H), 7.19-7.17 (m, 2H), 4.05 (dd, 1H, } J = 11.3, 3.8 \text{ Hz), 2.54 (s, 6H), 2.36 (s, 3H), 2.29 (dddd, 1H, } J = 13.7, 10.1, 6.4, 3.8 \text{ Hz), 2.12 (dddd, 1H, } J = 13.5, 11.4, 9.7, 5.3 \text{ Hz), 1.38-1.22 (m, 2H), 1.22-1.09 (m, 2H), 0.83 (t, 3H, } J = 7.3 \text{ Hz).} \]

\[ ^13C \text{NMR (126 MHz, CDCl}_3 \delta 138.8, 130.8, 129.6, 129.5, 67.4, 37.8, 29.6, 28.9, 22.4, 21.3, 13.9.} \]

FT-IR (neat) 3025, 2956, 2932, 2872, 2811, 1515, 1479, 1457, 1413, 1380, 1331, 1283, 1204, 1141, 1107, 1062, 1022, 968, 843, 832, 716 cm\(^{-1}\).

MS (ESI) \( m/z \) (M\(^+\)+Na) calcd for C\(_{14}\)H\(_{23}\)NNaO\(_2\)S: 292, found: 292.

\([\alpha]^{25}_D = -30^\circ \) (c = 0.99, CHCl\(_3\)).

\( (S)\)-\( N\)-\( N\)-Dimethyl-1-(4-(trifluoromethyl)phenyl)pentane-1-sulfonamide (Table 4, Entry 2). An oven-dried 8-mL vial equipped with a magnetic stir bar was capped with a PTFE-lined septum cap, cooled under vacuum, and filled with nitrogen. 4-Iodobenzotrifluoride (248 mg,
0.910 mmol) and THF (1.35 mL) were added to the vial, followed by the dropwise addition over 1 min of i-PrMgCl (1.92 M in THF; 0.474 mL, 0.910 mmol), and the resulting mixture was stirred at r.t. for 1 h. An oven-dried 4-mL vial equipped with a magnetic stir bar was capped with a PTFE-lined septum cap, cooled under vacuum, and filled with nitrogen. ZnI₂ (290 mg, 0.910 mmol) was added into the vial. The vial was immediately evacuated and refilled with nitrogen (three cycles), and then THF (1.82 mL) was added to the vial. The solution of ZnI₂ was transferred by syringe to the Grignard reagent, and then the reaction mixture was stirred at r.t. for 30 min.

1-Bromo-N,N-dimethylpentane-1-sulfonamide (181 mg, 0.700 mmol) and (4-(trifluoromethyl)phenyl)zinc iodide (0.910 mmol) were used. The product was purified by column chromatography (20% Et₂O/hexanes). Light-yellow solid. First run: 209 mg (92%, 98% ee). Second run: 216 mg (95%, 98% ee).

The ee was determined by HPLC on a CHIRALCEL OD-H column (3% i-PrOH/hexanes, 1.0 mL/min) with tᵣ = 8.8 min (major), 12.0 min (minor).

¹H NMR (500 MHz, CDCl₃) δ 7.65 (d, 2H, J = 8.2 Hz), 7.55 (d, 2H, J = 8.2 Hz), 4.14 (dd, 1H, J = 11.4, 3.8 Hz), 2.58 (s, 6H), 2.32 (dddd, 1H, J = 13.9, 10.3, 6.3, 3.9 Hz), 2.14 (ddddd, 1H, J = 13.7, 11.4, 10.1, 4.9 Hz), 1.38–1.24 (m, 2H), 1.22–1.06 (m, 2H), 0.84 (t, 3H, J = 7.3 Hz).

¹³C NMR (126 MHz, CDCl₃) δ 138.2 (d, J_CF = 1.3 Hz), 131.1 (q, J_CF = 32.7 Hz), 130.0, 125.8 (q, J_CF = 3.7 Hz), 123.6 (q, J_CF = 272.2 Hz), 67.3, 37.8, 29.6, 28.8, 22.3, 13.8.

FT-IR (neat) 2958, 2875, 1325, 1167, 1122, 1069, 1019, 968, 856, 727 cm⁻¹.

MS (ESI) m/z (M⁺+Na) calcd for C₁₄H₂₀F₃NNaO₂S: 346, found: 346.

[α]²⁵_D = −23.3° (c = 1.02, CHCl₃).

**Ethyl (S)-4-(1-(N,N-dimethylsulfamoyl)pentyl)benzoate (Table 4, Entry 3).** An oven-dried 8-mL vial equipped with a magnetic stir bar was capped with a PTFE-lined septum cap, cooled under vacuum, and then filled with nitrogen. Ethyl 4-iodobenzoate (251 mg, 0.910 mmol) was added to the vial, and then the vial was evacuated and refilled with nitrogen (three cycles). Next, THF (1.31 mL) was added to the vial, and the vial was wrapped with electrical tape and fitted with a nitrogen-filled balloon. Then, the reaction mixture was cooled to −20 °C. i-PrMgCl (1.78 M in THF; 0.511 mL, 0.910 mmol) was added over 1 min, and then the mixture was stirred at −20 °C for 2 h. An oven-dried 4-mL vial equipped with a magnetic stir bar was capped with a PTFE-lined septum cap, cooled under vacuum, and filled with nitrogen. ZnI₂ (291 mg, 0.910 mmol) was added to the vial. The vial was immediately placed under vacuum and then filled with nitrogen. This evacuation-refill cycle was repeated three times, and then THF (1.82 mL) was added to the vial. The solution of ZnI₂ was transferred by syringe to the Grignard reagent,
and then the reaction mixture was stirred at –20 °C for 30 min. The reaction mixture was allowed to warm to r.t. and stirred for an additional 30 min.

1-Bromo-\(N,N\)-dimethylpentane-1-sulfonamide (181 mg, 0.700 mmol) and (4-(ethoxycarbonyl)phenyl)zinc iodide (0.910 mmol) were used. The product was purified by column chromatography (20% ethyl acetate/hexanes). Colorless oil. First run: 229 mg (>99%, 97% ee). Second run: 229 mg (>99%, 97% ee).

The ee was determined by HPLC on a CHIRALCEL OD-H column (5% \(i\)-PrOH/hexanes, 1.0 mL/min) with \(t_r = 8.7\) min (major), 11.4 min (minor).

\(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta 8.07–8.05 (m, 2H), 7.50–7.48 (m, 2H), 4.38 (q, 2H, \(J = 7.1\) Hz), 4.14 (dd, 1H, \(J = 11.3, 3.8\) Hz), 2.55 (s, 6H), 2.32 (dddd, 1H, \(J = 14.0, 10.2, 6.2, 3.8\) Hz), 2.16 (ddddd, 1H, \(J = 13.6, 11.3, 10.0, 4.8\) Hz), 1.40 (t, 3H, \(J = 7.1\) Hz), 1.37–1.21 (m, 2H), 1.21–1.05 (m, 2H), 0.82 (t, 3H, \(J = 7.3\) Hz).

\(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta 166.2, 139.0, 131.1, 130.0, 129.6, 67.5, 61.4, 37.9, 29.6, 28.9, 22.4, 14.5, 13.9.

FT-IR (neat) 2956, 2934, 2872, 2813, 1718, 1611, 1576, 1507, 1477, 1457, 1417, 1367, 1334, 1278, 1182, 1143, 1110, 1063, 1021, 968, 867, 799, 776, 753, 712 cm\(^{-1}\).

MS (ESI) \(m/z (M^+ – SO_2NMe_2)\) calcd for C\(_{14}\)H\(_{19}\)O\(_2\): 219, found: 219.

\([\alpha]^{25}_D = -36°\) (c = 1.00, CHCl\(_3\)).

\((S)-1-(3\text{-Methoxyphenyl})-\(N,N\)-dimethylpentane-1-sulfonamide (Table 4, Entry 4). 1-Bromo-\(N,N\)-dimethylpentane-1-sulfonamide (181 mg, 0.700 mmol) and (3-methoxyphenyl)zinc iodide (0.910 mmol) were used. The product was purified by column chromatography (15% ethyl acetate/hexanes). White solid. First run: 175 mg (88%, 95% ee). Second run: 174 mg (87%, 96% ee).

The ee was determined by HPLC on a CHIRALCEL OD-H column (3% \(i\)-PrOH/hexanes, 1.0 mL/min) with \(t_r = 10.5\) min (major), 12.9 min (minor).

\(^1\)H NMR (500 MHz, CDCl\(_3\)) \(\delta 7.30–7.27 (m, 1H), 6.99–6.97 (m, 2H), 6.90–6.88 (m, 1H), 4.05 (dd, 1H, \(J = 11.3, 3.8\) Hz), 3.82 (s, 3H), 2.56 (s, 6H), 2.29 (ddddd, 1H, \(J = 13.6, 10.2, 6.6, 3.8\) Hz), 2.12 (ddddd, 1H, \(J = 13.5, 11.3, 9.8, 5.2\) Hz), 1.38–1.23 (m, 2H), 1.23–1.10 (m, 2H), 0.84 (t, 3H, \(J = 7.3\) Hz).

\(^{13}\)C NMR (126 MHz, CDCl\(_3\)) \(\delta 159.9, 135.4, 129.8, 122.0, 115.2, 114.2, 67.6, 55.5, 37.8, 29.7, 28.9, 22.4, 13.9.

FT-IR (neat) 3002, 2956, 2873, 2839, 1718, 1611, 1576, 1507, 1477, 1457, 1417, 1367, 1334, 1278, 1182, 1143, 1110, 1063, 1021, 968, 867, 799, 776, 753, 712 cm\(^{-1}\).

MS (ESI) \(m/z (M^+ + Na)\) calcd for C\(_{14}\)H\(_{23}\)NNaO\(_3\)S: 308, found: 308.

\([\alpha]^{25}_D = -28°\) (c = 1.00, CHCl\(_3\)).

S–20
(S)-1-(2-Methoxyphenyl)-N,N-dimethylpentane-1-sulfonamide (Table 4, Entry 5). 1-Bromo-N,N-dimethylpentane-1-sulfonamide (181 mg, 0.700 mmol) and (2-methoxyphenyl)zinc iodide (1.05 mmol) were used. The product was purified by column chromatography on silica gel (10%→15% ethyl acetate/hexanes) and then on C-18 silica gel (10%→100% acetonitrile/water). Light-yellow oil. First run: 125 mg (63%, 96% ee). Second run: 128 mg (64%, 96% ee).

The ee was determined by HPLC on a CHIRALPAK AS-H column (5% i-PrOH/hexanes, 1.0 mL/min) with $t_r = 16.6$ min (major), 19.3 min (minor).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.59 (dd, 1H, $J = 7.8, 1.7$ Hz), 7.30 (ddd, 1H, $J = 8.2, 7.4, 1.7$ Hz), 7.00 (ddd, 1H, $J = 7.6, 7.6, 1.1$ Hz), 6.91 (dd, 1H, $J = 8.3, 1.1$ Hz), 4.87 (dd, 1H, $J = 11.4, 3.9$ Hz), 3.87 (s, 3H), 2.51 (s, 6H), 2.32 (ddddd, 1H, $J = 13.7, 10.2, 6.4, 3.9$ Hz), 2.13–2.05 (m, 1H), 1.36–1.22 (m, 2H), 1.22–1.07 (m, 2H), 0.83 (t, 3H, $J = 7.3$ Hz).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 157.7, 129.7, 129.6, 122.2, 121.1, 110.5, 57.3, 55.8, 37.6, 29.7, 28.6, 22.4, 13.9.

FT-IR (neat) 3070, 3005, 2957, 2873, 1601, 1587, 1494, 1463, 1442, 1380, 1330, 1290, 1247, 1202, 1142, 1124, 1090, 1052, 1026, 967, 796, 765, 726 cm$^{-1}$.

MS (ESI) $m/z$ (M$^+$+Na) calcd for C$_{14}$H$_{23}$NNaO$_3$S: 308, found: 308.

$[\alpha]_D^{25} = +40^\circ$ (c = 1.03, CHCl$_3$).

(S)-N,N-Dimethyl-1-(o-tolyl)pentane-1-sulfonamide (Table 4, Entry 6). 1-Bromo-N,N-dimethylpentane-1-sulfonamide (181 mg, 0.700 mmol) and o-tolylzinc iodide (1.05 mmol) were used. The product was purified by column chromatography (5%→10% ethyl acetate/hexanes). Light-yellow oil. First run: 148 mg (78%, 97% ee). Second run: 149 mg (79%, 97% ee).

The ee was determined by HPLC on a CHIRALCEL OD-H column (2% i-PrOH/hexanes, 1.0 mL/min) with $t_r = 9.8$ min (major), 11.9 min (minor).

$^1$H NMR (500 MHz, CDCl$_3$) $\delta$ 7.62–7.58 (m, 1H), 7.25–7.19 (m, 3H), 4.43 (dd, 1H, $J = 11.3, 3.8$ Hz), 2.60 (s, 6H), 2.39 (s, 3H), 2.33 (ddddd, 1H, $J = 13.6, 10.1, 6.1, 3.8$ Hz), 2.16–2.08 (m, 1H), 1.37–1.22 (m, 2H), 1.21–1.06 (m, 2H), 0.83 (t, 3H, $J = 7.3$ Hz).

$^{13}$C NMR (126 MHz, CDCl$_3$) $\delta$ 137.4, 132.3, 130.7, 128.4, 128.3, 126.6, 63.2, 38.0, 30.7, 28.7, 22.6, 20.1, 13.9.

S-21
FT-IR (neat) 3064, 3023, 2957, 2872, 2813, 1604, 1493, 1461, 1380, 1283, 1178, 1141, 1119, 1063, 967, 834, 802 cm\(^{-1}\).

MS (ESI) \text{m/z} (M^+ + Na) calcd for C\(_{14}\)H\(_{23}\)NNaO\(_2\)S: 292, found: 292.
\([\alpha]\)\text{D}\(^{25}\) = +7.9° (c = 1.05, CHCl\(_3\)).

\(\text{Me}_2\text{N} - \text{Bu}
\text{O} \text{Et}(\text{S})-\text{1-1} -(2\text{-Ethylphenyl})-\text{N,N-dimethylpentane-1-sulfonamide (Table 4, Entry 7)}.

1-Bromo-N,N-dimethylpentane-1-sulfonamide (181 mg, 0.700 mmol), (2-ethylphenyl)zinc iodide (1.40 mmol), NiCl\(_2\)-glyme (30.8 mg, 0.140 mmol), and (R,R)-L1 (60.9 mg, 0.182 mmol) were used. The product was purified by column chromatography (first purification: 10% ethyl acetate/hexanes; second purification: 15%\(\rightarrow\)90% dichloromethane/hexanes). Light-yellow oil. First run: 178 mg (90%, 97% ee). Second run: 165 mg (83%, 97% ee).

The ee was determined by HPLC on a CHIRALPAK IC column (15% i-PrOH/hexanes, 1.0 mL/min) with \(t_r\) = 16.9 min (minor), 22.7 min (major).

\(^1\text{H NMR} (500 \text{ MHz, CD}_2\text{Cl}_2) \delta 7.57-7.54 \text{ (m, 1H), 7.30-7.22 \text{ (m, 3H), 4.46 \text{ (dd, 1H, } J = 11.1, 4.0 \text{ Hz), 2.79 (dq, 1H, } J = 14.9, 7.5 \text{ Hz), 2.68 (dq, 1H, } J = 15.2, 7.6 \text{ Hz), 2.62 (s, 6H), 2.29-2.22 \text{ (m, 1H), 2.15-2.07 \text{ (m, 1H), 1.39-1.24 \text{ (m, 2H), 1.22 (t, 3H, } J = 7.6 \text{ Hz), 1.24-1.15 \text{ (m, 1H), 1.13-1.04 \text{ (m, 1H), 0.84 (t, 3H, } J = 7.3 \text{ Hz).}}}

\(^{13}\text{C NMR} (126 \text{ MHz, CD}_2\text{Cl}_2) \delta 144.1, 131.9, 129.2, 128.7, 128.5, 126.5, 62.8, 38.0, 30.8, 29.3, 26.0, 23.0, 15.7, 13.9.

FT-IR (neat) 3063, 3021, 2959, 2933, 2873, 2813, 1490, 1455, 1378, 1330, 1282, 1201, 1177, 1141, 1120, 1062, 968, 803, 760 cm\(^{-1}\).

MS (ESI) \text{m/z} (M^+ + Na) calcd for C\(_{15}\)H\(_{25}\)NNaO\(_2\)S: 306, found: 306.
\([\alpha]\)\text{D}\(^{25}\) = +8.9° (c = 1.03, CHCl\(_3\)).

\(\text{tert-Butyl (S)-5-(1-(N,N-dimethylsulfamoyl)pentyl)-1H-indole-1-carboxylate (Table 4, Entry 8).}\) An oven-dried 8-mL vial equipped with a magnetic stir bar was capped with a PTFE-lined septum cap, cooled under vacuum, and filled with nitrogen. \(\text{tert-Butyl 5-iodo-1H-indole-1-carboxylate (360 mg, 1.05 mmol) was added to the vial, and then the vial was evacuated and refilled with nitrogen (three cycles). THF (1.56 mL) was added to the vial, and the vial was}
wrapped with electrical tape and fitted with a nitrogen-filled balloon. Then, the reaction mixture was cooled to −20 °C. i-PrMgCl (1.93 M in THF; 0.544 mL, 1.05 mmol) was added over 1 min, and the mixture was stirred at −20 °C for 2 h. An oven-dried 4-mL vial equipped with a magnetic stir bar was capped with a PTFE-lined septum cap, cooled under vacuum, and filled with nitrogen. ZnI₂ (338 mg, 1.06 mmol) was added to the vial. The vial was immediately placed under vacuum and then filled with nitrogen. This evacuation-refill cycle was repeated three times, and then THF (2.10 mL) was added to the vial. The solution of ZnI₂ was transferred by syringe to the Grignard reagent, and then the reaction mixture was stirred at −20 °C for 30 min. The reaction mixture was allowed to warm to r.t. and stirred for an additional 30 min.

1-Bromo-N,N-dimethylpentane-1-sulfonamido (181 mg, 0.70 mmol) and (1-(tert-butoxycarbonyl)-1H-indol-5-yl)zinc iodide (1.05 mmol) were used. The product was purified by column chromatography on silica gel (10%→15% ethyl acetate/hexanes) and then on C-18 silica gel (10%→100% acetonitrile/water). Yellow solid. First run: 180 mg (65%, 88% ee). Second run: 200 mg (72%, 90% ee).

The ee was determined by HPLC on a CHIRALCEL OD-H column (2% i-PrOH/hexanes, 1.0 mL/min) with tᵣ = 11.7 min (major), 15.4 min (minor).

³¹C NMR (126 MHz, CDCl₃) δ 149.7, 135.4, 130.9, 128.0, 126.9, 125.7, 121.9, 115.4, 107.4, 84.2, 67.6, 37.9, 28.9, 28.3, 22.4, 13.9.

FT-IR (neat) 3152, 3120, 2956, 2934, 2873, 1736, 1536, 1470, 1445, 1374, 1351, 1329, 1256, 1218, 1193, 1164, 1138, 1107, 1084, 1042, 1024, 968, 841, 768, 729 cm⁻¹.

MS (ESI) m/z (M⁺+Na) calcd for C₂₀H₂₉N₂NaO₄S: 417, found: 417.

[α]D₂⁵ = −23.7° (c = 1.04, CHCl₃).

(S)-1-Methoxy-2-(1-(methylsulfonfyl)pentyl)benzene (Table 4, Entry 9). 1-Bromo-1-(methylsulfonfyl)pentane (160 mg, 0.700 mmol) and (2-methoxyphenyl)zinc iodide (0.910 mmol) were used. The product was purified by column chromatography (20%→25% ethyl acetate/hexanes). Colorless oil. First run: 148 mg (82%, 96% ee). Second run: 154 mg (86%, 96% ee).

The ee was determined by HPLC on a CHIRALCEL OD-H column (5% i-PrOH/hexanes, 1.0 mL/min) with tᵣ = 17.6 min (minor), 18.9 min (major).

¹H NMR (500 MHz, CDCl₃) δ 7.52 (dd, 1H, J = 7.8, 1.7 Hz), 7.34 (ddd, 1H, J = 8.3, 7.4, 1.7 Hz), 7.04 (ddd, 1H, J = 7.6, 7.6, 1.1 Hz), 6.93 (dd, 1H, J = 8.3, 1.1 Hz), 4.81 (dd, 1H, J = 11.5, 3.9 Hz),
3.87 (s, 3H), 2.58 (s, 3H), 2.40 (ddddd, 1H, J = 13.5, 9.6, 6.9, 3.9 Hz), 2.05 (ddddd, 1H, J = 13.5, 11.5, 9.4, 5.3 Hz), 1.38–1.24 (m, 2H), 1.23–1.12 (m, 2H), 0.84 (t, 3H, J = 7.3 Hz).

$^{13}$C NMR (126 MHz, CDCl$_3$) δ 157.6, 130.1, 129.2, 121.7, 121.6, 110.9, 60.4, 55.9, 38.6, 28.7, 25.9, 22.4, 13.9.

FT-IR (neat) 3009, 2957, 2872, 1601, 1587, 1494, 1464, 1440, 1412, 1380, 1296, 1247, 1192, 1164, 1137, 1090, 1051, 1025, 956, 792, 755 cm$^{-1}$.

MS (ESI) m/z (M$^+$+Na) calcd for C$_{13}$H$_{20}$NaO$_2$S: 279, found: 279. 

$[\alpha]_{25}^{25}D$ = +61° (c = 1.00, CHCl$_3$).

(S)-1-Methyl-2-(1-(methylsulfonyl)pentyl)benzene (Table 4, Entry 10). 1-Bromo-1-(methylsulfonyl)pentane (160 mg, 0.700 mmol) and o-tolylzinc iodide (0.910 mmol) were used. The product was purified by column chromatography (15%→20% ethyl acetate/hexanes). Colorless oil. First run: 137 mg (81%, 97% ee). Second run: 134 mg (80%, 97% ee).

The ee was determined by HPLC on a CHIRALPAK AS-H column (10% i-PrOH/hexanes, 1.0 mL/min) with $t_r$ = 18.4 min (minor), 28.0 min (major).

$^1$H NMR (500 MHz, CD$_2$Cl$_2$) δ 7.52–7.50 (m, 1H), 7.30–7.23 (m, 3H), 4.37 (dd, 1H, J = 11.4, 3.7 Hz), 2.61 (s, 3H), 2.40 (s, 3H), 2.37–2.31 (m, 1H), 2.12–2.04 (m, 1H), 1.40–1.24 (m, 2H), 1.24–1.10 (m, 2H), 0.84 (t, 3H, J = 7.3 Hz).

$^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) δ 138.4, 132.0, 131.2, 129.0, 127.9, 127.1, 65.0, 38.7, 29.1, 28.5, 22.8, 20.3, 13.9.

FT-IR (neat) 3025, 2957, 2931, 2872, 1493, 1464, 1411, 1380, 1294, 1224, 1208, 1177, 1138, 1113, 1051, 958, 825, 796, 771, 736 cm$^{-1}$.

MS (ESI) m/z (M$^+$+Na) calcd for C$_{13}$H$_{20}$NaO$_2$S: 263, found: 263. 

$[\alpha]_{25}^{25}D$ = +24.2° (c = 0.99, CHCl$_3$).

(S)-1-Ethyl-2-(1-(methylsulfonyl)pentyl)benzene (Table 4, Entry 11). 1-Bromo-1-(methylsulfonyl)pentane (160 mg, 0.700 mmol), (2-ethylphenyl)zinc iodide (1.40 mmol), NiCl$_2$·glyme (30.8 mg, 0.140 mmol), and (R,R)-L1 (60.9 mg, 0.182 mmol) were used. The product was purified by column chromatography (15% ethyl acetate/hexanes). Light-yellow oil. First run: 145 mg (81%, 98% ee). Second run: 146 mg (82%, 98% ee).
The ee was determined by HPLC on a CHIRALPAK AS-H column (10% i-PrOH/hexanes, 1.0 mL/min) with t_r = 13.1 min (minor), 22.1 min (major).

^1^H NMR (500 MHz, CDCl_3) δ 7.52–7.51 (m, 1H), 7.34–7.26 (m, 3H), 4.41 (dd, 1H, J = 11.2, 3.9 Hz), 2.82–2.75 (m, 1H), 2.73–2.66 (m, 1H), 2.62 (s, 3H), 2.35 (dddd, 1H, J = 13.5, 11.0, 5.7, 3.8 Hz), 2.12–2.04 (m, 1H), 1.41–1.20 (m, 3H), 1.23 (t, 3H, J = 7.6 Hz), 1.19–1.09 (m, 1H), 0.85 (t, 3H, J = 7.2 Hz).

^13^C NMR (126 MHz, CDCl_3) δ 144.4, 131.2, 129.6, 129.1, 127.9, 126.9, 64.5, 38.8, 29.3, 28.7, 26.2, 23.0, 15.7, 13.9.

FT-IR (neat) 3063, 3026, 2960, 2932, 2873, 1491, 1453, 1411, 1379, 1294, 1218, 1176, 1138, 1113, 1061, 958, 831, 797, 757 cm\(^{-1}\).

MS (ESI) m/z (M\(^+\)+Na) calcd for C_{14}H_{22}NaO_{2}S: 277, found: 277.

\([\alpha]^{25}_{D} = +24.1^\circ\) (c = 1.01, CHCl_3).

(S,E)-N,N-Dimethyl-1-phenylhex-5-ene-1-sulfonamide-6-d (eq 3). White solid. The ee was determined by HPLC on a CHIRALCEL OD-H column (1% i-PrOH/hexanes, 1.0 mL/min) with t_r = 13.9 min (major), 17.4 min (minor).

^1^H NMR (500 MHz, CDCl_3) δ 7.42–7.34 (m, 5H), 5.70 (dt, 1H, J = 17.0, 6.5 Hz), 4.95 (dt, 1H, J = 17.1, 1.6 Hz), 4.09 (dd, 1H, J = 11.2, 3.9 Hz), 2.53 (s, 6H), 2.32 (dddd, 1H, J = 13.9, 10.3, 6.4, 3.9 Hz), 2.20–2.12 (m, 1H), 2.10–1.98 (m, 2H), 1.36–1.22 (m, 2H).

^13^C NMR (126 MHz, CDCl_3) δ 137.8, 134.0, 129.6, 129.0, 128.9, 115.0 (t, J = 24 Hz), 67.8, 37.8, 33.3, 29.5, 26.2.

FT-IR (neat) 3088, 3065, 3024, 2926, 2860, 2822, 2261, 1623, 1496, 1480, 1456, 1436, 1326, 1292, 1256, 1200, 1140, 1064, 1043, 984, 970, 917, 906, 822, 799, 778, 745 cm\(^{-1}\).

MS (EI) m/z (M\(^+\)-SO_2NMe_2) calcd for C_{12}H_{14}D: 160, found: 160.

\([\alpha]^{25}_{D} = -34^\circ\) (c = 0.99, CHCl_3); 96% ee.

(S)-N,N-Dimethyl-1-phenylhex-5-ene-1-sulfonamide (Figure 1). White solid. The ee was determined by HPLC on a CHIRALCEL OD-H column (1% i-PrOH/hexanes, 1.0 mL/min) with t_r = 14.1 min (major), 17.8 min (minor).

^1^H NMR (500 MHz, CDCl_3) δ 7.42–7.34 (m, 5H), 5.70 (ddt, 1H, J = 17.0, 10.3, 6.7 Hz), 4.98–4.92 (m, 2H), 4.09 (dd, 1H, J = 11.2, 3.9 Hz), 2.53 (s, 6H), 2.32 (dddd, 1H, J = 14.1, 10.3, 6.3, 3.9 Hz), 2.20–2.12 (m, 1H), 2.10–1.98 (m, 2H), 1.36–1.22 (m, 2H).

^13^C NMR (126 MHz, CDCl_3) δ 138.0, 134.0, 129.6, 129.0, 128.9, 115.3, 67.8, 37.8, 33.4, 29.5, 26.2.
FT-IR (neat) 3067, 3033, 2908, 2868, 2821, 1640, 1497, 1480, 1455, 1417, 1329, 1282, 1199, 1141, 1063, 1043, 993, 966, 916, 906, 870, 814, 781, 746, 735 cm⁻¹.
MS (EI) m/z (M⁺−SO₂NMe₂) calcd for C₁₂H₁₅: 159, found: 159.
[α]²⁵° D = −33° (c = 0.82, CHCl₃); 97% ee.

**syn-2-Benzyl-Ν,Ν-dimethylcyclopentane-1-sulfonamide (Figure 1).** White solid.

\(^{1}H\) NMR (500 MHz, CD₂Cl₂) δ 7.30–7.26 (m, 2H), 7.20–7.16 (m, 3H), 3.54 (dd, 1H, J = 8.7, 8.7, 6.3 Hz), 3.31–3.25 (m, 1H), 2.89 (s, 6H), 2.60–2.52 (m, 2H), 2.15–2.07 (m, 1H), 2.04–1.97 (m, 1H), 1.95–1.87 (m, 1H), 1.66–1.44 (m, 3H).

\(^{13}C\) NMR (126 MHz, CD₂Cl₂) δ 141.8, 129.4, 128.6, 126.2, 62.8, 44.7, 37.8, 35.7, 29.7, 26.8, 22.7.

FT-IR (neat) 3084, 3060, 3024, 2922, 2874, 2850, 2806, 1602, 1583, 1495, 1473, 1452, 1332, 1273, 1195, 1136, 1073, 1058, 1029, 958, 845, 822, 727 cm⁻¹.

MS (EI) m/z (M⁺) calcd for C₁₄H₂₁NO₂S: 267, found: 267.

**anti-2-Benzyl-Ν,Ν-dimethylcyclopentane-1-sulfonamide (Figure 1).** Colorless oil.

\(^{1}H\) NMR (500 MHz, CD₂Cl₂) δ 7.32–7.28 (m, 2H), 7.22–7.19 (m, 3H), 3.20 (ddd, 1H, J = 8.9, 6.1, 6.1 Hz), 2.97 (dd, 1H, J = 12.6, 4.9 Hz), 2.78 (s, 6H), 2.65–2.53 (m, 2H), 2.08–1.95 (m, 2H), 1.82–1.74 (m, 1H), 1.73–1.61 (m, 2H), 1.42–1.35 (m, 1H).

\(^{13}C\) NMR (126 MHz, CD₂Cl₂) δ 140.7, 129.6, 128.7, 126.5, 64.5, 43.6, 41.3, 37.8, 32.1, 28.5, 24.9.

FT-IR (neat) 3084, 3060, 3025, 2917, 2849, 1602, 1583, 1494, 1461, 1453, 1315, 1199, 1136, 1082, 1059, 1029, 960, 882, 849, 733 cm⁻¹.

MS (EI) m/z (M⁺) calcd for C₁₄H₂₁NO₂S: 267, found: 267.

IV. Enantioselective Alkenylations

**General Procedure.** Cp₂ZrHCl (Schwartz’s reagent; 258 mg, 1.00 mmol) was added to an oven-dried 4-mL vial equipped with a magnetic stir bar, and then the vial was capped with a PTFE-lined septum cap. The vial was evacuated and refilled with nitrogen (three cycles). 1,2-Dimethoxyethane (1.00 ml) was added to the vial, followed by the alkyne (1.00 mmol). The reaction mixture was stirred at r.t. for 1.5 h, at which time it had become homogenous. An oven-dried 20-mL vial equipped with a magnetic stir bar was charged with NiCl₂·glyme (11.0 mg, 0.050 mmol), (3R,8S)–L₆ (23.3 mg, 0.065 mmol), and the electrophile (0.500 mmol). The vial
was sealed with a PTFE-lined septum cap, placed under vacuum, and then filled with nitrogen. This evacuation-refill cycle was repeated three times. 1,2-Dimethoxyethane (2.57 mL) was added, and the mixture was stirred at r.t. for 1 h. The solution of the nucleophile was transferred by syringe over 2 min to the vial that contained the electrophile. The reaction mixture was stirred at r.t. for 24 h, and then the reaction was quenched by the addition of ethanol (0.50 mL). The solution was filtered through a pad of silica (eluted with Et₂O). The filtrate was concentrated, and the resulting residue was purified by column chromatography.

A second run was conducted with (3S,8R)-L6.

(S,E)-N,N-Dimethyl-1-phenyloct-2-ene-4-sulfonamide (Table 5, Entry 1). 1-Bromo-N,N-dimethylpentane-1-sulfonamide (129 mg, 0.500 mmol) and (E)-(3-phenylprop-1-en-1-yl)zirconium reagent (1.00 mmol) were used. The product was purified by column chromatography on silica gel (15% ethyl acetate/hexanes). Colorless oil. First run: 116 mg (79%, 91% ee). Second run: 122 mg (83%, 90% ee).

The ee was determined by HPLC on a CHIRALCEL OJ-H column (5% i-PrOH/hexanes, 1.0 mL/min) with t_r = 18.2 min (major), 20.9 min (minor).

1H NMR (500 MHz, CDCl₃) δ 7.32–7.28 (m, 2H), 7.24–7.20 (m, 1H), 7.18–7.15 (m, 2H), 5.87 (dddd, 1H, J = 15.3, 6.9, 6.9, 0.4 Hz), 5.44 (dddd, 1H, J = 15.3, 9.7, 1.5, 1.5 Hz), 3.57 (ddd, 1H, J = 10.5, 10.5, 3.5 Hz), 3.49–3.39 (m, 2H), 2.82 (s, 6H), 2.04–1.97 (m, 1H), 1.75–1.68 (m, 1H), 1.41–1.18 (m, 4H), 0.89 (t, 3H, J = 7.1 Hz).

13C NMR (126 MHz, CDCl₃) δ 139.3, 136.9, 128.7, 128.6, 126.5, 126.5, 125.1, 65.8, 39.1, 38.3, 28.8, 28.7, 22.4, 14.0.

FT-IR (neat) 3061, 3027, 2954, 2930, 2871, 1663, 1603, 1494, 1453, 1379, 1329, 1281, 1198, 1139, 1076, 1062, 1029, 966, 804, 747, 730 cm⁻¹.

MS (EI) m/z (M⁺–SO₂NMe₂) calcd for C₉₄H₂₉: 187, found: 187.

[α]D²⁵ = +22.9° (c = 1.01, CHCl₃).

(S,E)-N,N-Dicyclohexyl-1-phenyloct-2-ene-4-sulfonamide (Table 5, Entry 2). 1-Bromo-N,N-dicyclohexylpentane-1-sulfonamide (197 mg, 0.500 mmol) and (E)-(3-phenylprop-1-en-1-yl)zirconium reagent (1.00 mmol) were used. The product was purified by column chromatography on silica gel (5% Et₂O/hexanes) and then on C-18 silica gel (10%→100%
acetonitrile/water). Viscous light-yellow oil. First run: 179 mg (83%, 95% ee). Second run: 180 mg (83%, 94% ee).

The ee was determined by HPLC on a CHIRALPAK AD-H column (1% i-PrOH/hexanes, 0.6 mL/min) with $t_r = 16.6$ min (major), 17.7 min (minor).

$^1$H NMR (500 MHz, CD$_2$Cl$_2$) $\delta$ 7.31–7.28 (m, 2H), 7.22–7.17 (m, 3H), 5.80 (ddddd, 1H, $J = 15.3, 7.4, 6.0, 0.4$ Hz), 5.42 (ddddd, 1H, $J = 15.4, 9.8, 1.5, 1.5$ Hz), 3.48–3.38 (m, 2H), 3.33 (dddd, 1H, $J = 10.8, 10.0, 3.2$ Hz), 3.17–3.10 (m, 2H), 2.04–1.97 (m, 1H), 1.79–1.56 (m, 15H), 1.41–1.16 (m, 8H),

$^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) $\delta$ 140.1, 136.6, 128.9, 128.8, 126.6, 126.0, 69.0, 58.4, 39.2, 34.0, 33.1, 29.5, 29.3, 26.99, 26.97, 25.8, 22.7, 14.1.

FT-IR (neat) 3084, 3062, 3027, 2931, 2855, 1603, 1495, 1466, 1453, 1401, 1381, 1322, 1274, 1256, 1188, 1164, 1139, 1108, 1074, 1047, 1028, 981, 895, 854, 823, 750 cm$^{-1}$.

MS (ESI) m/z (M$^+$+Na) calcld for C$_{26}$H$_{41}$NNaO$_3$S: 454, found: 454.

$[\alpha]^{25}_D = -6.9^\circ$ (c = 1.02, CHCl$_3$).

\[ \text{(S,E)-1-(( tert-Butyldiphenylsilyl)oxy)-N,N-dicyclohexyl-3-en-5-sulfonamide (Table 5, Entry 3).} \]

1-Bromo-N,N-dicyclohexylpentane-1-sulfonamide (197 mg, 0.500 mmol) and (E)-4-((tert-butyldiphenylsilyl)oxy)but-1-en-1-yl)zirconium reagent (1.00 mmol) were used. The product was purified by column chromatography on silica gel (3% ethyl acetate/hexanes) and then on C-18 silica gel (10%→100% acetonitrile/water). Viscous light-yellow oil. First run: 254 mg (81%, 95% ee). Second run: 259 mg (83%, 94% ee).

The ee was determined by HPLC on a CHIRALPAK AD-H column (0.5% i-PrOH/hexanes, 0.8 mL/min) with $t_r = 14.6$ min (minor), 17.9 min (major).

$^1$H NMR (500 MHz, CD$_2$Cl$_2$) $\delta$ 7.67–7.64 (m, 4H), 7.45–7.41 (m, 2H), 7.40–7.36 (m, 4H), 5.69 (ddd, 1H, $J = 15.5, 6.4, 6.4$ Hz), 5.42 (ddddd, 1H, $J = 15.5, 9.7, 1.4, 1.4$ Hz), 3.75–3.68 (m, 2H), 3.26 (ddpd, 1H, $J = 10.6, 9.5, 3.2$ Hz), 3.15–3.09 (m, 2H), 2.39–2.29 (m, 2H), 2.08–2.01 (m, 1H), 1.76–1.57 (m, 14H), 1.39–1.14 (m, 9H), 1.12–1.02 (m, 2H), 1.05 (s, 9H), 0.86 (t, 3H, $J = 7.2$ Hz).

$^{13}$C NMR (126 MHz, CD$_2$Cl$_2$) $\delta$ 135.7, 135.6, 134.3, 133.9, 133.8, 129.79, 129.78, 127.79, 127.78, 126.10, 69.5, 63.2, 58.2, 36.0, 33.9, 32.8, 29.3, 29.0, 26.9, 26.7, 25.5, 22.5, 19.3, 14.0.

FT-IR (neat) 3071, 3048, 2931, 2856, 1590, 1471, 1453, 1428, 1389, 1323, 1257, 1221, 1188, 1164, 1138, 1110, 1048, 1028, 998, 980, 939, 895, 854, 822, 764, 738 cm$^{-1}$.

MS (ESI) m/z (M$^+$+Na) calcld for C$_{37}$H$_{57}$NNaO$_3$S$i$: 646, found: 646.

$[\alpha]^{25}_D = +1.7^\circ$ (c = 0.99, CHCl$_3$).
(S,E)-N-Benzyl-11-chloro-N-phenyl-1-(thiophen-2-yl)undec-6-ene-5-sulfonamide (Table 5, Entry 4). N-Benzyl-1-bromo-N-phenyl-5-(thiophen-2-yl)pentane-1-sulfonamide (239 mg, 0.500 mmol) and (E)-(6-chlorohex-1-en-1-yl)zirconium reagent (1.00 mmol) were used. The product was purified by column chromatography (first purification: 5% ethyl acetate/hexanes; second purification: 15% cyclopentyl methyl ether/hexanes). Viscous light-yellow oil. First run: 165 mg (64%, 80% ee). Second run: 156 mg (60%, 81% ee).

The ee was determined by HPLC on a CHIRALPAK AD-H column (10% i-PrOH/hexanes, 0.8 mL/min) with tR = 17.5 min (major), 23.8 min (minor).

1H NMR (500 MHz, CDCl3) δ 7.32–7.28 (m, 2H), 7.27–7.19 (m, 8H), 7.12 (dd, 1H, J = 5.1, 1.2 Hz), 6.91 (dd, 1H, J = 5.1, 3.4 Hz), 6.77 (dddd, 1H, J = 3.3, 1.0, 1.0, 1.0 Hz), 5.80 (dd, 1H, J = 15.3, 6.8, 6.8 Hz), 5.46 (dddd, 1H, J = 15.4, 9.7, 1.5, 1.5 Hz), 4.99 (d, 1H, J = 15.1 Hz), 4.67 (d, 1H, J = 15.1 Hz), 3.60–3.55 (m, 1H), 3.58 (t, 2H, J = 6.6 Hz), 2.87–2.76 (m, 2H), 2.26–2.13 (m, 2H), 2.04 (dddd, 1H, J = 13.6, 9.9, 6.3, 3.4 Hz), 1.86–1.72 (m, 3H), 1.72–1.56 (m, 4H), 1.50–1.41 (m, 1H), 1.32–1.22 (m, 1H).

13C NMR (126 MHz, CDCl3) δ 145.6, 139.7, 139.0, 137.3, 129.4, 129.3, 128.7, 128.6, 127.9, 127.8, 127.0, 124.5, 124.2, 123.2, 66.7, 56.7, 45.4, 32.5, 32.2, 31.6, 29.9, 29.2, 26.5, 26.2.

FT-IR (neat) 3064, 3032, 2933, 2860, 1595, 1493, 1454, 1337, 1216, 1145, 1093, 1065, 1028, 976, 916, 862, 775 cm⁻¹.

MS (ESI) m/z (M+Na) calcd for C32H34ClINaO2S2: 538, found: 538.

[α]D 25° = -23.0° (c = 1.03, CHCl3).

(S,E)-4-(((1-Phenylhept-1-en-3-yl)sulfonyl)morpholine (Table 5, Entry 5). 4-((1-Bromopentyl)sulfonyl)morpholine (150 mg, 0.500 mmol), and (E)-styrylzirconium reagent (1.00 mmol) were used. The product was purified by column chromatography (15% ethyl acetate/hexanes). White solid. First run: 110 mg (68%, 97% ee). Second run: 110 mg (68%, 95% ee).

The ee was determined by HPLC on a CHIRALPAK AS-H column (10% i-PrOH/hexanes, 1.0 mL/min) with tR = 18.7 min (minor), 30.1 min (major).

1H NMR (500 MHz, CDCl3) δ 7.42–7.39 (m, 2H), 7.38–7.34 (m, 2H), 7.32–7.29 (m, 1H), 6.62 (d, 1H, J = 15.9 Hz), 6.06 (dd, 1H, J = 15.9, 9.8 Hz), 3.70–3.62 (m, 5H), 3.38–3.30 (m, 4H), 2.17–2.10 (m, 1H), 1.87–1.79 (m, 1H), 1.45–1.22 (m, 4H), 0.90 (t, 3H, J = 7.0 Hz).
$^{13}$C NMR (126 MHz, CDCl$_3$) δ 137.0, 135.7, 129.0, 128.7, 126.7, 122.7, 67.1, 67.0, 46.8, 29.0, 28.8, 22.4, 14.0.

FT-IR (neat) 2958, 2923, 2859, 1450, 1339, 1324, 1260, 1148, 1114, 1073, 955, 743 cm$^{-1}$.

MS (ESI) m/z (M$^+$+Na) calcd for C$_{17}$H$_{25}$NNaO$_3$S: 346, found: 346.

$[\alpha]^{25}_D = -82^\circ$ (c = 0.98, CHCl$_3$).

\[
\begin{align*}
\text{Me} & \quad \text{S} \quad \text{Cy} \\
& \quad \text{O} \quad \text{O} \\
& \quad \text{CH}_2\text{Ph}
\end{align*}
\]

(S,E)-(4-Cyclohexyl-4-(methylsulfonyl)but-2-en-1-yl)benzene (Table 5, Entry 6).

(Bromo(methylsulfonyl)methyl)cyclohexane (179 mg, 0.700 mmol), (E)-(3-phenylprop-1-en-1-yl)zirconium reagent (1.40 mmol), and (R,R)-L1 (30.4 mg, 0.091 mmol) were used. The product was purified by column chromatography (15% ethyl acetate/hexanes). Light-yellow oil. First run: 109 mg (53%, 93% ee). Second run: 98 mg (48%, 93% ee).

The ee was determined by HPLC on a CHIRALPAK IB-3 column (5% i-PrOH/hexanes, 1.0 mL/min) with $t_1 = 13.0$ min (minor), 22.9 min (major).

$^1$H NMR (500 MHz, CDCl$_3$) δ 7.33–7.30 (m, 2H), 7.25–7.21 (m, 1H), 7.18–7.16 (m, 2H), 5.89 (ddd, 1H, $J = 15.2, 6.9, 6.9$ Hz), 5.71 (dddd, 1H, $J = 15.3, 10.4, 1.4, 1.4$ Hz), 3.48 (d, 2H, $J = 6.9$ Hz), 3.29 (dd, 1H, $J = 10.4, 3.8$ Hz), 2.76 (s, 3H), 2.32 (tq, 1H, $J = 11.9, 3.5$ Hz), 2.08–2.02 (m, 1H), 1.78–1.72 (m, 2H), 1.70–1.61 (m, 2H), 1.40–1.26 (m, 2H), 1.23–1.07 (m, 3H).

$^{13}$C NMR (126 MHz, CDCl$_3$) δ 139.2, 139.0, 128.8, 128.6, 126.6, 122.6, 73.2, 39.8, 39.3, 36.0, 32.2, 28.9, 26.4, 26.1, 26.0.

FT-IR (neat) 3083, 3060, 3026, 2927, 2852, 1660, 1602, 1494, 1452, 1411, 1351, 1295, 1240, 1173, 1133, 1077, 1029, 978, 894, 852, 784, 751, 700 cm$^{-1}$.

MS (ESI) m/z (M$^+$+Na) calcd for C$_{17}$H$_{25}$NNaO$_3$S: 315, found: 315.

$[\alpha]^{25}_D = +60.8^\circ$ (c = 1.00, CHCl$_3$).
V. Determination of Absolute Stereochemistry

Product from entry 7 of Table 2 (run with (S,S)-L1). (R)-5-((tert-Butyldimethylsilyl)oxy)-N,N-dimethyl-1-phenylpentane-1-sulfonamide. A crystal suitable for X-ray crystallography was grown by vapor diffusion with dichloromethane and pentane.

A suitable crystal of C_{19}H_{35}NO_{3}SSi was selected for analysis. All measurements were made on a Bruker SMART 1000 CCD with filtered Mo-Kα radiation at a temperature of 100 K. Using Olex2, the structure was solved with the ShelXS structure solution program using Direct Methods and refined with the ShelXL refinement package using Least Squares minimization. The absolute stereochemistry was determined on the basis of the absolute structure parameter.

(6) Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. J. Appl. Crystallogr. 2009, 42, 339–341.
(7) Sheldrick, G. M. Acta Cryst. 2008, A64, 112–122.
Table S–1. Crystal data and structure refinement for crystal01.

| Property                        | Value                           |
|---------------------------------|---------------------------------|
| Identification code             | crystal01                        |
| Empirical formula               | C_{19}H_{35}NO_3SSi              |
| Formula weight                  | 385.63                          |
| Temperature                     | 100 K                           |
| Wavelength                      | 0.71073 Å                       |
| Crystal system                  | Monoclinic                      |
| Space group                     | P2₁                             |
| Unit cell dimensions            | a = 5.9209(6) Å, α = 90°.       |
|                                 | b = 10.6607(12) Å, β = 99.2230(10)°. |
|                                 | c = 17.0647(19) Å, γ = 90°.     |
| Volume                          | 1063.2(2) Å³                    |
| Z                               | 2                               |
| Density (calculated)            | 1.205 Mg/m³                     |
| Absorption coefficient          | 0.226 mm⁻¹                      |
| F(000)                          | 420                             |
| Crystal size                    | 0.4 x 0.4 x 0.1 mm³             |
| Theta range for data collection | 1.209 to 29.107°.               |
| Index ranges                    | -7<=h<=8, -13<=k<=14, -22<=l<=23 |
| Reflections collected           | 16770                           |
| Independent reflections         | 5160 [R(int) = 0.0236]           |
| Completeness to theta = 25.000° | 100.0 %                         |
| Absorption correction           | Semi-empirical from equivalents |
| Max. and min. transmission      | 1.0000 and 0.9257               |
| Refinement method               | Full-matrix least-squares on F² |
| Data / restraints / parameters  | 5160 / 1 / 233                  |
| Goodness-of-fit on F²           | 1.098                           |
| Final R indices [I>2sigma(I)]   | R1 = 0.0279, wR2 = 0.0669       |
| R indices (all data)            | R1 = 0.0308, wR2 = 0.0690       |
| Absolute structure parameter    | 0.02(2)                         |
| Largest diff. peak and hole     | 0.325 and -0.163 e/Å⁻³          |
Table S–2. Atomic coordinates (× 10^4) and equivalent isotropic displacement parameters (Å^2 × 10^3) for crystal01. U(eq) is defined as one third of the trace of the orthogonalized U^ij tensor.

|     | x         | y         | z         | U(eq) |
|-----|-----------|-----------|-----------|-------|
| S(1)| -4031(1)  | 5489(1)   | 517(1)    | 14(1) |
| Si(1)| 2618(1)   | 1014(1)   | 3465(1)   | 14(1) |
| O(1)| -3746(3)  | 5244(1)   | -291(1)   | 20(1) |
| O(2)| -6212(2)  | 5232(1)   | 755(1)    | 19(1) |
| O(3)|  648(2)   | 432(2)    | 2769(1)   | 19(1) |
| N(1)| -3503(3)  | 6967(2)   | 689(1)    | 16(1) |
| C(1)| -4272(4)  | 7610(2)   | 1361(1)   | 20(1) |
| C(2)| -1459(4)  | 7506(2)   | 435(1)    | 22(1) |
| C(3)| -1857(3)  | 4582(2)   | 1132(1)   | 12(1) |
| C(4)| -2416(3)  | 3172(2)   | 1032(1)   | 15(1) |
| C(5)| -552(3)   | 2381(2)   | 1521(1)   | 16(1) |
| C(6)| -952(4)   | 967(2)    | 1416(1)   | 18(1) |
| C(7)|  820(4)   | 204(2)    | 1957(1)   | 21(1) |
| C(8)| -1531(3)  | 5030(2)   | 1983(1)   | 12(1) |
| C(9)|  508(3)   | 5621(2)   | 2311(1)   | 17(1) |
| C(10)|  842(3)  | 6027(2)   | 3095(1)   | 21(1) |
| C(11)|  -835(4) | 5849(2)   | 3558(1)   | 21(1) |
| C(12)| -2858(4) | 5251(2)   | 3243(1)   | 20(1) |
| C(13)| -3211(3) | 4849(2)   | 2456(1)   | 16(1) |
| C(14)|  1116(4) | 2120(2)   | 4054(1)   | 24(1) |
| C(15)|  4837(4) | 1893(2)   | 3029(1)   | 27(1) |
| C(16)|  3960(3) | -312(2)   | 4110(1)   | 15(1) |
| C(17)|  5647(4) | 216(2)    | 4811(1)   | 23(1) |
| C(18)|  2103(4) | -1074(2)  | 4431(1)   | 22(1) |
| C(19)|  5248(4) | -1178(2)  | 3614(1)   | 24(1) |
Table S–3. Bond lengths [Å] and angles [°] for crystal01.

| Bond         | Length (Å)  |
|--------------|-------------|
| S(1)-O(1)    | 1.4394(15)  |
| S(1)-O(2)    | 1.4405(15)  |
| S(1)-N(1)    | 1.6247(18)  |
| S(1)-C(3)    | 1.8048(19)  |
| Si(1)-O(3)   | 1.6471(14)  |
| Si(1)-C(14)  | 1.865(2)    |
| Si(1)-C(15)  | 1.863(2)    |
| Si(1)-C(16)  | 1.886(2)    |
| O(3)-C(7)    | 1.426(2)    |
| N(1)-C(1)    | 1.469(3)    |
| N(1)-C(2)    | 1.466(3)    |
| C(3)-C(4)    | 1.543(3)    |
| C(3)-C(8)    | 1.512(3)    |
| C(4)-C(5)    | 1.526(3)    |
| C(5)-C(6)    | 1.532(3)    |
| C(6)-C(7)    | 1.517(3)    |
| C(8)-C(9)    | 1.396(3)    |
| C(8)-C(13)   | 1.391(3)    |
| C(9)-C(10)   | 1.390(3)    |
| C(10)-C(11)  | 1.378(3)    |
| C(11)-C(12)  | 1.387(3)    |
| C(12)-C(13)  | 1.393(3)    |
| C(16)-C(17)  | 1.537(3)    |
| C(16)-C(18)  | 1.537(3)    |
| C(16)-C(19)  | 1.535(3)    |
| O(1)-S(1)-O(2)| 118.93(9) |
| O(1)-S(1)-N(1)| 107.45(9) |
| O(1)-S(1)-C(3)| 106.10(9) |
| O(2)-S(1)-N(1)| 106.79(9) |
| O(2)-S(1)-C(3)| 108.81(9) |
| N(1)-S(1)-C(3)| 108.40(9) |
| O(3)-Si(1)-C(14)| 106.44(9) |
| O(3)-Si(1)-C(15)| 111.35(9) |
| O(3)-Si(1)-C(16)| 108.79(9) |
C(14)-Si(1)-C(16)  110.66(10)
C(15)-Si(1)-C(14)  108.96(11)
C(15)-Si(1)-C(16)  110.57(10)
C(7)-O(3)-Si(1)  127.79(13)
C(1)-N(1)-S(1)  121.23(14)
C(2)-N(1)-S(1)  118.06(14)
C(2)-N(1)-C(1)  114.98(17)
C(4)-C(3)-S(1)  109.70(13)
C(8)-C(3)-S(1)  111.03(13)
C(8)-C(3)-C(4)  113.95(16)
C(5)-C(4)-C(3)  110.75(15)
C(4)-C(5)-C(6)  113.27(16)
C(7)-C(6)-C(5)  112.19(16)
O(3)-C(7)-C(6)  110.46(17)
C(9)-C(8)-C(3)  119.76(17)
C(13)-C(8)-C(3)  121.38(17)
C(13)-C(8)-C(9)  118.86(18)
C(10)-C(9)-C(8)  120.46(18)
C(11)-C(10)-C(9)  120.29(19)
C(10)-C(11)-C(12)  119.91(19)
C(11)-C(12)-C(13)  120.06(19)
C(8)-C(13)-C(12)  120.41(19)
C(17)-C(16)-Si(1)  109.87(14)
C(17)-C(16)-C(18)  109.15(17)
C(18)-C(16)-Si(1)  110.21(14)
C(19)-C(16)-Si(1)  109.22(14)
C(19)-C(16)-C(17)  109.34(17)
C(19)-C(16)-C(18)  109.03(17)
Table S–4. Anisotropic displacement parameters (Å$^2 \times 10^3$) for crystal01. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [ h^2 a^*^2 U_{11} + \ldots + 2 h k a^* b^* U_{12} ]$

|     | U$^{11}$ | U$^{22}$ | U$^{33}$ | U$^{23}$ | U$^{13}$ | U$^{12}$ |
|-----|---------|---------|---------|---------|---------|---------|
| S(1)| 14(1)   | 14(1)   | 12(1)   | 1(1)    | -1(1)   | 0(1)    |
| Si(1)| 14(1)  | 12(1)   | 15(1)   | 2(1)    | 1(1)    | -1(1)   |
| O(1)| 26(1)   | 21(1)   | 13(1)   | 0(1)    | -2(1)   | 0(1)    |
| O(2)| 14(1)   | 19(1)   | 23(1)   | 1(1)    | -1(1)   | -1(1)   |
| O(3)| 21(1)   | 20(1)   | 14(1)   | 2(1)    | -2(1)   | -6(1)   |
| N(1)| 20(1)   | 13(1)   | 16(1)   | 2(1)    | 4(1)    | 1(1)    |
| C(1)| 24(1)   | 14(1)   | 22(1)   | 0(1)    | 5(1)    | 3(1)    |
| C(2)| 27(1)   | 17(1)   | 24(1)   | 2(1)    | 8(1)    | -6(1)   |
| C(3)| 12(1)   | 13(1)   | 11(1)   | 1(1)    | 1(1)    | 2(1)    |
| C(4)| 18(1)   | 13(1)   | 12(1)   | -2(1)   | 1(1)    | 0(1)    |
| C(5)| 19(1)   | 14(1)   | 13(1)   | -1(1)   | -1(1)   | 2(1)    |
| C(6)| 24(1)   | 15(1)   | 14(1)   | -1(1)   | -2(1)   | 0(1)    |
| C(7)| 27(1)   | 15(1)   | 18(1)   | -1(1)   | -1(1)   | 4(1)    |
| C(8)| 15(1)   | 10(1)   | 12(1)   | 1(1)    | 1(1)    | 1(1)    |
| C(9)| 14(1)   | 20(1)   | 18(1)   | 1(1)    | 3(1)    | 0(1)    |
| C(10)| 18(1)  | 20(1)   | 22(1)   | -5(1)   | -5(1)   | -1(1)   |
| C(11)| 30(1)  | 19(1)   | 14(1)   | -3(1)   | 0(1)    | 5(1)    |
| C(12)| 24(1)  | 21(1)   | 16(1)   | 0(1)    | 8(1)    | 2(1)    |
| C(13)| 18(1)  | 14(1)   | 15(1)   | 1(1)    | 2(1)    | 0(1)    |
| C(14)| 23(1)  | 20(1)   | 29(1)   | -5(1)   | 0(1)    | 4(1)    |
| C(15)| 23(1)  | 28(1)   | 29(1)   | 12(1)   | 2(1)    | -8(1)   |
| C(16)| 16(1)  | 14(1)   | 15(1)   | 2(1)    | 1(1)    | 1(1)    |
| C(17)| 23(1)  | 25(1)   | 20(1)   | 5(1)    | -4(1)   | -2(1)   |
| C(18)| 24(1)  | 17(1)   | 24(1)   | 5(1)    | 4(1)    | -2(1)   |
| C(19)| 24(1)  | 21(1)   | 29(1)   | 1(1)    | 5(1)    | 6(1)    |
Table S–5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for crystal01.

|      | x   | y   | z   | U(eq) |
|------|-----|-----|-----|-------|
| H(1A)| -5587 | 7185 | 1497 | 30    |
| H(1B)| -4669 | 8461 | 1216 | 30    |
| H(1C)| -3064 | 7603 | 1809 | 30    |
| H(2A)| -208  | 7483 | 869  | 33    |
| H(2B)| -1761 | 8359 | 270  | 33    |
| H(2C)| -1070 | 7027 | -1   | 33    |
| H(3) | -413  | 4726 | 934  | 15    |
| H(4A)| -3873 | 3002 | 1201 | 18    |
| H(4B)| -2546 | 2944 | 476  | 18    |
| H(5A)| -473  | 2592 | 2077 | 19    |
| H(5B)| 911   | 2591 | 1368 | 19    |
| H(6A)| -2463 | 763  | 1529 | 22    |
| H(6B)| -902  | 741  | 869  | 22    |
| H(7A)| 586   | -681 | 1840 | 25    |
| H(7B)| 2340  | 428  | 1861 | 25    |
| H(9) | 1649  | 5743 | 2003 | 20    |
| H(10)| 2205  | 6421 | 3308 | 25    |
| H(11)| -611  | 6130 | 4081 | 26    |
| H(12)| -3980 | 5118 | 3558 | 24    |
| H(13)| -4578 | 4458 | 2246 | 19    |
| H(14A)| 483 | 2801 | 3721 | 36    |
| H(14B)| 2183 | 2444 | 4490 | 36    |
| H(14C)| -93 | 1686 | 4254 | 36    |
| H(15A)| 5788 | 1311 | 2802 | 40    |
| H(15B)| 5761 | 2371 | 3437 | 40    |
| H(15C)| 4107 | 2449 | 2624 | 40    |
| H(17A)| 6824 | 681  | 4613 | 35    |
| H(17B)| 6325 | -463 | 5136 | 35    |
| H(17C)| 4846 | 759  | 5121 | 35    |
| H(18A)| 1374 | -556 | 4777 | 32    |
| H(18B)| 2791 | -1786 | 4721 | 32    |
|     |     |     |     |     |
|-----|-----|-----|-----|-----|
| H(18C) | 986 | -1356 | 3996 | 32 |
| H(19A) | 4214 | -1474 | 3160 | 37 |
| H(19B) | 5860 | -1880 | 3931 | 37 |
| H(19C) | 6475 | -722 | 3439 | 37 |

S–38
Product from entry 2 of Table 3: (S)-(Cyclohexyl(methylsulfonyl)methyl)benzene (from a reaction using \((R,R)\)-L1). A crystal suitable for X-ray crystallography was grown by vapor diffusion with dichloromethane and pentane.

A suitable crystal of \(\text{C}_{14}\text{H}_{20}\text{O}_{2}\text{S}\) was selected for analysis. All measurements were made on a Bruker APEX-II CCD with filtered Mo-K\(\alpha\) radiation at a temperature of 100 K. Using Olex2,\(^6\) the structure was solved with the ShelXS\(^7\) structure solution program using Direct Methods and refined with the ShelXL\(^7\) refinement package using Least Squares minimization. The absolute stereochemistry was determined on the basis of the absolute structure parameter.
Table S–6. Crystal data and structure refinement for crystal03.

| Property                              | Value                                      |
|---------------------------------------|--------------------------------------------|
| Identification code                   | crystal03                                  |
| Empirical formula                     | C_{14}H_{20}O_{2}S                         |
| Formula weight                        | 252.36                                     |
| Temperature                           | 100.15 K                                   |
| Wavelength                            | 0.71073 Å                                  |
| Crystal system                        | Orthorhombic                               |
| Space group                           | P2\_2\_2\_1                               |
| Unit cell dimensions a = 6.2283(3) Å  | α = 90°                                   |
|                                      | b = 13.7866(7) Å                          | β = 90°                                   |
|                                      | c = 15.3937(8) Å                          | γ = 90°                                   |
| Volume                                | 1321.81(12) Å\(^3\)                      |
| Z                                     | 4                                          |
| Density (calculated)                  | 1.268 Mg/m\(^3\)                          |
| Absorption coefficient                | 0.233 mm\(^4\)                            |
| F(000)                                | 544                                        |
| Crystal size                          | 0.62 x 0.16 x 0.09 mm\(^3\)               |
| Theta range for data collection       | 1.983 to 33.731°                           |
| Index ranges                          | -9<=h<=9, -21<=k<=20, -23<=l<=23           |
| Reflections collected                 | 39471                                      |
| Independent reflections               | 4910 [R(int) = 0.0621]                     |
| Completeness to theta = 25.000°       | 100.0 %                                    |
| Absorption correction                 | Semi-empirical from equivalents            |
| Max. and min. transmission            | 1.0000 and 0.8575                          |
| Refinement method                     | Full-matrix least-squares on \(F^2\)      |
| Data / restraints / parameters        | 4910 / 0 / 155                             |
| Goodness-of-fit on \(F^2\)            | 1.141                                      |
| Final R indices \([I>2\sigma(I)]\)    | R1 = 0.0540, wR2 = 0.1115                  |
| R indices (all data)                  | R1 = 0.0741, wR2 = 0.1179                  |
| Absolute structure parameter          | 0.01(3)                                    |
| Largest diff. peak and hole           | 0.692 and -0.385 e/Å\(^3\)                |
Table S–7. Atomic coordinates ( x $10^4$) and equivalent isotropic displacement parameters (Å$^2$ x $10^3$) for crystal03. U(eq) is defined as one third of the trace of the orthogonalized $U_{ij}$ tensor.

|     | x    | y    | z    | U(eq) |
|-----|------|------|------|-------|
| S(1)| 7502(1) | 3757(1) | 4892(1) | 17(1) |
| O(1)| 6971(3) | 3206(1) | 5662(1) | 21(1) |
| O(2)| 9662(3) | 4136(1) | 4836(1) | 21(1) |
| C(1)| 5566(4) | 4731(2) | 4817(2) | 14(1) |
| C(2)| 5963(4) | 5465(2) | 5570(2) | 14(1) |
| C(3)| 7759(4) | 6208(2) | 5418(2) | 17(1) |
| C(4)| 8064(4) | 6849(2) | 6224(2) | 20(1) |
| C(5)| 5993(4) | 7371(2) | 6464(2) | 21(1) |
| C(6)| 4161(4) | 6651(2) | 6587(2) | 23(1) |
| C(7)| 3868(4) | 5994(2) | 5790(2) | 19(1) |
| C(8)| 5392(4) | 5139(2) | 3904(2) | 15(1) |
| C(9)| 7131(4) | 5552(2) | 3461(2) | 17(1) |
| C(10)| 6869(4) | 5905(2) | 2620(2) | 18(1) |
| C(11)| 4895(4) | 5840(2) | 2207(2) | 18(1) |
| C(12)| 3168(4) | 5434(2) | 2644(2) | 19(1) |
| C(13)| 3419(4) | 5081(2) | 3483(2) | 17(1) |
| C(14)| 7027(5) | 3018(2) | 3977(2) | 23(1) |
Table S–8. Bond lengths [Å] and angles [°] for crystal03.

| Bond                  | Length (Å) |
|-----------------------|------------|
| S(1)-O(1)             | 1.4458(19) |
| S(1)-O(2)             | 1.4461(19) |
| S(1)-C(1)             | 1.809(2)   |
| S(1)-C(14)            | 1.763(3)   |
| C(1)-H(1)             | 1.0000     |
| C(1)-C(2)             | 1.557(3)   |
| C(1)-C(8)             | 1.518(3)   |
| C(2)-H(2)             | 1.0000     |
| C(2)-C(3)             | 1.535(3)   |
| C(2)-C(7)             | 1.533(3)   |
| C(3)-H(3A)            | 0.9900     |
| C(3)-H(3B)            | 0.9900     |
| C(3)-C(4)             | 1.536(3)   |
| C(4)-H(4A)            | 0.9900     |
| C(4)-H(4B)            | 0.9900     |
| C(4)-C(5)             | 1.523(4)   |
| C(5)-H(5A)            | 0.9900     |
| C(5)-H(5B)            | 0.9900     |
| C(5)-C(6)             | 1.524(4)   |
| C(6)-H(6A)            | 0.9900     |
| C(6)-H(6B)            | 0.9900     |
| C(6)-C(7)             | 1.536(4)   |
| C(7)-H(7A)            | 0.9900     |
| C(7)-H(7B)            | 0.9900     |
| C(8)-C(9)             | 1.401(3)   |
| C(8)-C(13)            | 1.391(4)   |
| C(9)-H(9)             | 0.9500     |
| C(9)-C(10)            | 1.393(3)   |
| C(10)-H(10)           | 0.9500     |
| C(10)-C(11)           | 1.387(4)   |
| C(11)-H(11)           | 0.9500     |
| C(11)-C(12)           | 1.386(4)   |
| C(12)-H(12)           | 0.9500     |
| C(12)-C(13)           | 1.390(4)   |
| C(13)-H(13)           | 0.9500     |
| Bond                  | Distance (Å) |
|----------------------|--------------|
| C(14)-H(14A)        | 0.9800       |
| C(14)-H(14B)        | 0.9800       |
| C(14)-H(14C)        | 0.9800       |
| O(1)-S(1)-O(2)      | 116.89(12)   |
| O(1)-S(1)-C(1)      | 106.82(11)   |
| O(1)-S(1)-C(14)     | 108.23(11)   |
| O(2)-S(1)-C(1)      | 110.34(10)   |
| O(2)-S(1)-C(14)     | 108.51(13)   |
| C(14)-S(1)-C(1)     | 105.43(13)   |
| S(1)-C(1)-H(1)      | 105.6        |
| C(2)-C(1)-S(1)      | 109.22(16)   |
| C(2)-C(1)-H(1)      | 105.6        |
| C(8)-C(1)-S(1)      | 112.41(17)   |
| C(8)-C(1)-H(1)      | 105.6        |
| C(8)-C(1)-C(2)      | 117.36(18)   |
| C(1)-C(2)-H(2)      | 107.1        |
| C(3)-C(2)-C(1)      | 115.9(2)     |
| C(3)-C(2)-H(2)      | 107.1        |
| C(7)-C(2)-C(1)      | 109.8(2)     |
| C(7)-C(2)-H(2)      | 107.1        |
| C(7)-C(2)-C(3)      | 109.60(18)   |
| C(2)-C(3)-H(3A)     | 109.5        |
| C(2)-C(3)-H(3B)     | 109.5        |
| C(2)-C(3)-C(4)      | 110.6(2)     |
| H(3A)-C(3)-H(3B)    | 108.1        |
| C(4)-C(3)-H(3A)     | 109.5        |
| C(4)-C(3)-H(3B)     | 109.5        |
| C(3)-C(4)-H(4A)     | 109.4        |
| C(3)-C(4)-H(4B)     | 109.4        |
| H(4A)-C(4)-H(4B)    | 108.0        |
| C(5)-C(4)-C(3)      | 111.3(2)     |
| C(5)-C(4)-H(4A)     | 109.4        |
| C(5)-C(4)-H(4B)     | 109.4        |
| C(4)-C(5)-H(5A)     | 109.5        |
| C(4)-C(5)-H(5B)     | 109.5        |
| C(4)-C(5)-C(6)      | 110.9(2)     |

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H(5A)-C(5)-H(5B) 108.1
C(6)-C(5)-H(5A) 109.5
C(6)-C(5)-H(5B) 109.5
C(5)-C(6)-H(6A) 109.2
C(5)-C(6)-H(6B) 109.2
C(5)-C(6)-C(7) 111.9(2)
H(6A)-C(6)-H(6B) 107.9
C(7)-C(6)-H(6A) 109.2
C(7)-C(6)-H(6B) 109.2
C(2)-C(7)-C(6) 110.9(2)
C(2)-C(7)-H(7A) 109.5
C(2)-C(7)-H(7B) 109.5
C(6)-C(7)-H(7A) 109.5
C(6)-C(7)-H(7B) 109.5
H(7A)-C(7)-H(7B) 108.1
C(9)-C(8)-C(1) 123.1(2)
C(13)-C(8)-C(1) 118.2(2)
C(13)-C(8)-C(9) 118.7(2)
C(8)-C(9)-H(9) 119.9
C(10)-C(9)-C(8) 120.2(2)
C(10)-C(9)-H(9) 119.9
C(9)-C(10)-H(10) 119.7
C(11)-C(10)-C(9) 120.5(2)
C(11)-C(10)-H(10) 119.7
C(10)-C(11)-H(11) 120.3
C(12)-C(11)-C(10) 119.4(2)
C(12)-C(11)-H(11) 120.3
C(11)-C(12)-H(12) 119.8
C(11)-C(12)-C(13) 120.4(2)
C(13)-C(12)-H(12) 119.8
C(8)-C(13)-H(13) 119.6
C(12)-C(13)-C(8) 120.8(2)
C(12)-C(13)-H(13) 119.6
S(1)-C(14)-H(14A) 109.5
S(1)-C(14)-H(14B) 109.5
S(1)-C(14)-H(14C) 109.5
H(14A)-C(14)-H(14B) 109.5
H(14A)-C(14)-H(14C)  109.5
H(14B)-C(14)-H(14C)  109.5
Table S-9. Anisotropic displacement parameters (Å$^2 \times 10^3$) for crystal03. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [ h^2 a^{*2} U^{11} + ... + 2hka^*b^*U^{12} ]$

|     | U$^{11}$ | U$^{22}$ | U$^{33}$ | U$^{23}$ | U$^{13}$ | U$^{12}$ |
|-----|----------|----------|----------|----------|----------|----------|
| S(1) | 19(1)    | 13(1)    | 18(1)    | 1(1)     | -1(1)    | -1(1)    |
| O(1) | 26(1)    | 17(1)    | 20(1)    | 2(1)     | -1(1)    | -2(1)    |
| O(2) | 17(1)    | 16(1)    | 29(1)    | 2(1)     | 0(1)     | 0(1)     |
| C(1) | 11(1)    | 15(1)    | 17(1)    | 1(1)     | -1(1)    | -3(1)    |
| C(2) | 14(1)    | 11(1)    | 18(1)    | 2(1)     | -1(1)    | -2(1)    |
| C(3) | 11(1)    | 17(1)    | 24(1)    | -2(1)    | 0(1)     | -3(1)    |
| C(4) | 15(1)    | 16(1)    | 28(1)    | -2(1)    | -3(1)    | -2(1)    |
| C(5) | 18(1)    | 15(1)    | 29(1)    | -4(1)    | -1(1)    | 2(1)     |
| C(6) | 17(1)    | 22(1)    | 31(2)    | -6(1)    | 4(1)     | 0(1)     |
| C(7) | 12(1)    | 18(1)    | 26(1)    | -2(1)    | 0(1)     | 0(1)     |
| C(8) | 15(1)    | 12(1)    | 18(1)    | 1(1)     | 0(1)     | 0(1)     |
| C(9) | 14(1)    | 17(1)    | 20(1)    | 0(1)     | -1(1)    | -3(1)    |
| C(10)| 19(1)    | 14(1)    | 20(1)    | 1(1)     | 4(1)     | -1(1)    |
| C(11)| 22(1)    | 14(1)    | 18(1)    | 0(1)     | -1(1)    | 3(1)     |
| C(12)| 18(1)    | 18(1)    | 21(1)    | -2(1)    | -3(1)    | 2(1)     |
| C(13)| 12(1)    | 17(1)    | 21(1)    | 0(1)     | 1(1)     | -2(1)    |
| C(14)| 32(2)    | 14(1)    | 22(1)    | -3(1)    | -2(1)    | -1(1)    |
Table S-10. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for crystal03.

|     | x  | y  | z  | U(eq) |
|-----|----|----|----|-------|
| H(1)| 4140 | 4428 | 4942 | 17    |
| H(2)| 6373 | 5077 | 6093 | 17    |
| H(3A)| 7392 | 6620 | 4912 | 21    |
| H(3B)| 9117 | 5865 | 5286 | 21    |
| H(4A)| 8533 | 6442 | 6719 | 23    |
| H(4B)| 9203 | 7333 | 6109 | 23    |
| H(5A)| 5613 | 7837 | 6000 | 25    |
| H(5B)| 6210 | 7741 | 7009 | 25    |
| H(6A)| 2812 | 7011 | 6695 | 28    |
| H(6B)| 4457 | 6244 | 7103 | 28    |
| H(7A)| 2725 | 5513 | 5908 | 22    |
| H(7B)| 3415 | 6392 | 5286 | 22    |
| H(9)| 8494 | 5592 | 3736 | 20    |
| H(10)| 8051 | 6192 | 2327 | 21    |
| H(11)| 4728 | 6071 | 1629 | 22    |
| H(12)| 1806 | 5398 | 2368 | 23    |
| H(13)| 2229 | 4797 | 3773 | 20    |
| H(14A)| 7233 | 3400 | 3447 | 34    |
| H(14B)| 5551 | 2773 | 3996 | 34    |
| H(14C)| 8032 | 2471 | 3980 | 34    |
Product from entry 5 of Table 5 (run with (3R,8S)–L6). (S,E)-4-((1-Phenylhept-1-en-3-yl)sulfonyl)morpholine. A crystal suitable for X-ray crystallography was grown by vapor diffusion with Et$_2$O and pentane.

![Chemical structure]

A suitable crystal of C$_{17}$H$_{25}$NO$_3$S was selected for analysis. All measurements were made on a Bruker APEX-II CCD with filtered Mo-Kα radiation at a temperature of 100 K. Using Olex2,$^6$ the structure was solved with the ShelXS$^7$ structure solution program using Direct Methods and refined with the ShelXL$^7$ refinement package using Least Squares minimization. The absolute stereochemistry was determined on the basis of the absolute structure parameter.
Table S–11. Crystal data and structure refinement for crystal02.

| Property                        | Value                        |
|---------------------------------|------------------------------|
| Identification code             | crystal02                    |
| Empirical formula               | C₁₇H₂₅NO₃S                  |
| Formula weight                  | 323.44                       |
| Temperature                     | 100 K                        |
| Wavelength                      | 0.71073 Å                    |
| Crystal system                  | Monoclinic                   |
| Space group                     | P2₁                           |
| Unit cell dimensions            | a = 12.8350(6) Å, α = 90°.   |
|                                 | b = 5.6272(3) Å, β = 113.133(2)°. |
|                                 | c = 12.9187(6) Å, γ = 90°.   |
| Volume                          | 858.03(7) Å³                 |
| Z                               | 2                            |
| Density (calculated)            | 1.252 Mg/m³                  |
| Absorption coefficient          | 0.201 mm⁻¹                   |
| F(000)                          | 348                          |
| Crystal size                    | 0.5 x 0.12 x 0.12 mm³        |
| Theta range for data collection | 1.714 to 31.552°.            |
| Index ranges                    | -18<=h<=18, -8<=k<=8, -19<=l<=19 |
| Reflections collected           | 57330                        |
| Independent reflections         | 5731 [R(int) = 0.0344]        |
| Completeness to theta = 25.242°| 100.0 %                      |
| Absorption correction           | Semi-empirical from equivalents |
| Max. and min. transmission      | 1.0000 and 0.8839            |
| Refinement method               | Full-matrix least-squares on F² |
| Data / restraints / parameters  | 5731 / 1 / 200               |
| Goodness-of-fit on F²           | 1.073                        |
| Final R indices [I>2sigma(I)]   | R1 = 0.0258, wR2 = 0.0683    |
| R indices (all data)            | R1 = 0.0273, wR2 = 0.0695    |
| Absolute structure parameter   | 0.019(11)                    |
| Largest diff. peak and hole     | 0.444 and -0.190 e/Å⁻³       |
Table S–12. Atomic coordinates ( x 10^4) and equivalent isotropic displacement parameters (Å^2 x 10^3) for crystal02. U(eq) is defined as one third of the trace of the orthogonalized U_ij tensor.

|     | x       | y       | z       | U(eq) |
|-----|---------|---------|---------|-------|
| S(1)| 4726(1) | 7787(1) | 3347(1) | 12(1) |
| O(1)| 4294(1) | 10123(2)| 2942(1) | 19(1) |
| O(2)| 5535(1) | 7480(2) | 4482(1) | 18(1) |
| O(3)| 6358(1) | 4985(2) | 1135(1) | 21(1) |
| N(1)| 5344(1) | 6838(2) | 2534(1) | 14(1) |
| C(1)| 3537(1) | 5903(2) | 3176(1) | 11(1) |
| C(2)| 3067(1) | 6643(2) | 4055(1) | 13(1) |
| C(3)| 2154(1) | 4910(2) | 4061(1) | 14(1) |
| C(4)| 1833(1) | 5268(3) | 5067(1) | 18(1) |
| C(5)| 944(1)  | 3484(3) | 5081(1) | 25(1) |
| C(6)| 2671(1) | 6069(2) | 1991(1) | 13(1) |
| C(7)| 2415(1) | 4264(2) | 1261(1) | 13(1) |
| C(8)| 1574(1) | 4329(2) | 93(1)   | 12(1) |
| C(9)| 797(1)  | 6196(2) | -325(1) | 16(1) |
| C(10)| 21(1)  | 6194(3) | -1441(1)| 19(1) |
| C(11)| 9(1)   | 4336(3) | -2156(1)| 20(1) |
| C(12)| 764(1) | 2453(3) | -1748(1)| 20(1) |
| C(13)| 1539(1)| 2453(2) | -630(1) | 16(1) |
| C(14)| 4862(1)| 7376(2) | 1320(1) | 18(1) |
| C(15)| 5820(1)| 7255(3) | 909(1)  | 21(1) |
| C(16)| 6847(1)| 4566(3) | 2317(1) | 21(1) |
| C(17)| 5947(1)| 4555(3) | 2806(1) | 19(1) |
Table S–13. Bond lengths [Å] and angles [°] for crystal02.

| Bond                  | Length [Å]   |
|-----------------------|--------------|
| S(1)-O(1)             | 1.4424(10)   |
| S(1)-O(2)             | 1.4360(9)    |
| S(1)-N(1)             | 1.6356(11)   |
| S(1)-C(1)             | 1.7985(12)   |
| O(3)-C(15)            | 1.4267(18)   |
| O(3)-C(16)            | 1.4241(17)   |
| N(1)-C(14)            | 1.4733(16)   |
| N(1)-C(17)            | 1.4694(17)   |
| C(1)-C(2)             | 1.5379(17)   |
| C(1)-C(6)             | 1.5001(15)   |
| C(2)-C(3)             | 1.5265(17)   |
| C(3)-C(4)             | 1.5241(18)   |
| C(4)-C(5)             | 1.525(2)     |
| C(6)-C(7)             | 1.3364(17)   |
| C(7)-C(8)             | 1.4711(16)   |
| C(8)-C(9)             | 1.4021(17)   |
| C(8)-C(13)            | 1.3993(17)   |
| C(9)-C(10)            | 1.3937(16)   |
| C(10)-C(11)           | 1.391(2)     |
| C(11)-C(12)           | 1.393(2)     |
| C(12)-C(13)           | 1.3954(17)   |
| C(14)-C(15)           | 1.5194(19)   |
| C(16)-C(17)           | 1.5189(19)   |
| O(1)-S(1)-N(1)        | 106.16(6)    |
| O(1)-S(1)-C(1)        | 107.85(6)    |
| O(2)-S(1)-O(1)        | 120.05(6)    |
| O(2)-S(1)-N(1)        | 106.10(6)    |
| O(2)-S(1)-C(1)        | 107.05(6)    |
| N(1)-S(1)-C(1)        | 109.34(6)    |
| C(16)-O(3)-C(15)      | 109.98(11)   |
| C(14)-N(1)-S(1)       | 120.64(9)    |
| C(17)-N(1)-S(1)       | 118.32(9)    |
| C(17)-N(1)-C(14)      | 113.32(11)   |
| C(2)-C(1)-S(1)        | 108.03(8)    |
| Bond                  | Angle (°)   |
|-----------------------|-------------|
| C(6)-C(1)-S(1)        | 109.94(8)   |
| C(6)-C(1)-C(2)        | 112.83(10)  |
| C(3)-C(2)-C(1)        | 110.77(10)  |
| C(4)-C(3)-C(2)        | 112.63(10)  |
| C(3)-C(4)-C(5)        | 112.19(12)  |
| C(7)-C(6)-C(1)        | 123.45(11)  |
| C(6)-C(7)-C(8)        | 125.69(11)  |
| C(9)-C(8)-C(7)        | 122.55(11)  |
| C(13)-C(8)-C(7)       | 118.95(11)  |
| C(13)-C(8)-C(9)       | 118.50(11)  |
| C(10)-C(9)-C(8)       | 120.60(12)  |
| C(11)-C(10)-C(9)      | 120.30(13)  |
| C(10)-C(11)-C(12)     | 119.73(12)  |
| C(11)-C(12)-C(13)     | 119.96(12)  |
| C(12)-C(13)-C(8)      | 120.90(12)  |
| N(1)-C(14)-C(15)      | 107.67(11)  |
| O(3)-C(15)-C(14)      | 111.21(11)  |
| O(3)-C(16)-C(17)      | 111.08(11)  |
| N(1)-C(17)-C(16)      | 108.16(11)  |
Table S–14. Anisotropic displacement parameters (Å² x 10^3) for crystal02. The anisotropic displacement factor exponent takes the form: -2\pi^2 [ h^2 a^*^2 U_{11} + ... + 2 h k a^* b^* U_{12} ]

|     | U_{11} | U_{22} | U_{33} | U_{23} | U_{13} | U_{12} |
|-----|--------|--------|--------|--------|--------|--------|
| S(1)| 12(1)  | 11(1)  | 11(1)  | -1(1)  | 4(1)   | -3(1)  |
| O(1)| 24(1)  | 11(1)  | 23(1)  | 0(1)   | 11(1)  | -2(1)  |
| O(2)| 15(1)  | 26(1)  | 12(1)  | -3(1)  | 2(1)   | -7(1)  |
| O(3)| 22(1)  | 22(1)  | 21(1)  | 0(1)   | 12(1)  | 1(1)   |
| N(1)| 16(1)  | 16(1)  | 13(1)  | 3(1)   | 7(1)   | 2(1)   |
| C(1)| 10(1)  | 10(1)  | 11(1)  | 0(1)   | 3(1)   | -1(1)  |
| C(2)| 12(1)  | 12(1)  | 13(1)  | -1(1)  | 5(1)   | 0(1)   |
| C(3)| 11(1)  | 16(1)  | 14(1)  | 1(1)   | 4(1)   | 0(1)   |
| C(4)| 18(1)  | 20(1)  | 19(1)  | -2(1)  | 10(1)  | -1(1)  |
| C(5)| 22(1)  | 29(1)  | 28(1)  | 2(1)   | 15(1)  | -4(1)  |
| C(6)| 11(1)  | 13(1)  | 12(1)  | 1(1)   | 2(1)   | 0(1)   |
| C(7)| 11(1)  | 14(1)  | 12(1)  | 0(1)   | 2(1)   | 0(1)   |
| C(8)| 11(1)  | 14(1)  | 11(1)  | -2(1)  | 3(1)   | -2(1)  |
| C(9)| 15(1)  | 16(1)  | 14(1)  | -1(1)  | 3(1)   | 1(1)   |
| C(10)| 15(1)| 21(1)  | 16(1)  | 2(1)   | 2(1)   | 2(1)   |
| C(11)| 16(1)| 29(1)  | 12(1)  | -2(1)  | 2(1)   | -4(1)  |
| C(12)| 18(1)| 26(1)  | 15(1)  | -7(1)  | 6(1)   | -3(1)  |
| C(13)| 13(1)| 18(1)  | 17(1)  | -4(1)  | 5(1)   | 0(1)   |
| C(14)| 18(1)| 23(1)  | 13(1)  | 4(1)   | 7(1)   | 3(1)   |
| C(15)| 24(1)| 23(1)  | 19(1)  | 4(1)   | 12(1)  | 1(1)   |
| C(16)| 18(1)| 24(1)  | 23(1)  | 4(1)   | 10(1)  | 4(1)   |
| C(17)| 21(1)| 16(1)  | 22(1)  | 7(1)   | 12(1)  | 4(1)   |
Table S–15. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^2 x 10^3) for crystal02.

|     | x    | y    | z    | U(eq) |
|-----|------|------|------|-------|
| H(1) | 3809 | 4223 | 3327 | 13    |
| H(2A)| 3691 | 6680 | 4811 | 15    |
| H(2B)| 2742 | 8262 | 3881 | 15    |
| H(3A)| 1471 | 5115 | 3359 | 16    |
| H(3B)| 2431 | 3264 | 4072 | 16    |
| H(4A)| 1536 | 6898 | 5045 | 22    |
| H(4B)| 2520 | 5104 | 5770 | 22    |
| H(5A)| 764  | 3776 | 5741 | 37    |
| H(5B)| 1241 | 1868 | 5118 | 37    |
| H(5C)| 257  | 3664 | 4395 | 37    |
| H(6) | 2285 | 7535 | 1747 | 16    |
| H(7) | 2810 | 2813 | 1519 | 15    |
| H(9) | 800  | 7474 | 156  | 19    |
| H(10)| -503 | 7467 | -1715| 23    |
| H(11)| -513 | 4352 | -2920| 24    |
| H(12)| 752  | 1170 | -2231| 24    |
| H(13)| 2051 | 1161 | -356 | 19    |
| H(14A)| 4520 | 8982 | 1187 | 22    |
| H(14B)| 4266 | 6206 | 911  | 22    |
| H(15A)| 5514 | 7563 | 89   | 25    |
| H(15B)| 6385 | 8503 | 1288 | 25    |
| H(16A)| 7412 | 5819 | 2689 | 25    |
| H(16B)| 7245 | 3017 | 2467 | 25    |
| H(17A)| 5409 | 3231 | 2480 | 23    |
| H(17B)| 6304 | 4338 | 3632 | 23    |
Injection Date  : 12/18/2012 2:33:11 PM          Seq. Line :   1
Sample Name     : JC9121A                         Location : Vial 4
Acq. Operator   : CE                                   Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\OD-01-40.M
Last changed    : 12/18/2012 2:48:17 PM by CE
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\OD-02-20.M
Last changed    : 7/30/2014 4:56:50 PM by MK
(modified after loading)

--- Area Percent Report ---

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100
Signal 2: DAD1 B, Sig=254,10 Ref=360,100
Signal 3: DAD1 C, Sig=210,10 Ref=360,100
Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
1  10.864 BB    0.2815 8588.73828  473.84717  98.0169
2  13.437 PV    0.2756  173.77281    7.93276   1.9831

Totals :                  8762.51109  481.77993

Results obtained with enhanced integrator!

Signal 6: DAD1 F, Sig=240,10 Ref=360,100
Signal 7: DAD1 G, Sig=270,10 Ref=360,100

--- End of Report ---
Injection Date  : 12/18/2012 3:04:38 PM          Seq. Line :   2
Sample Name     : JC9121B                         Location : Vial 5
Acq. Operator   : CE                                   Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\OD-01-30.M
Last changed    : 4/7/2011 2:40:51 AM by CC
Analysis Method : C:\HPCHEM\1\METHODS\AD005-40.M
Last changed    : 8/2/2014 8:33:12 PM by MK
(modified after loading)

Area: 169.81142    9.50396   1.8500
Area: 9009.37891  431.37000  98.1500

Results obtained with enhanced integrator!

Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
  1  10.689 FM 0.2978  169.81142    9.50396   1.8500
  2  13.020 MM 0.3481 9009.37891  431.37000  98.1500

Totals :                  9179.19032  440.87395

Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

**End of Report**
Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

| # | RetTime | Width | Area   | Height | Area | %     |
|---|---------|-------|--------|--------|------|-------|
| 1 | 12.026  | BP    | 0.3146 | 113.62238 | 5.33121 | 2.4632 |
| 2 | 13.125  | BB    | 0.3752 | 4499.24658 | 185.38203 | 97.5368 |

Totals: 4612.86896 | 190.71324 |

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

| # | RetTime | Width | Area   | Height | Area | %     |
|---|---------|-------|--------|--------|------|-------|
| 1 | 12.023  | MM    | 0.3532 | 59.19486 | 2.79296 | 2.2676 |
| 2 | 13.125  | MM    | 0.4060 | 2551.31982 | 104.72879 | 97.7324 |

Totals: 2610.51468 | 107.52175 |

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

| # | RetTime | Width | Area   | Height | Area | %     |
|---|---------|-------|--------|--------|------|-------|
| 1 | 12.025  | MM    | 0.3401 | 560.64459 | 27.47764 | 2.3671 |
| 2 | 13.125  | MM    | 0.4189 | 2.31246e4 | 919.98969 | 97.6329 |

Totals: 2.36852e4 | 947.46732 |

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

| # | RetTime | Width | Area   | Height | Area | %     |
|---|---------|-------|--------|--------|------|-------|
| 1 | 12.030  | BP    | 0.3344 | 358.89542 | 16.20137 | 2.7223 |
| 2 | 13.125  | BB    | 0.3746 | 1.28247e4 | 529.39978 | 97.2777 |

Totals: 1.31836e4 | 545.60115 |

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

---

*** End of Report ***

---

Table 2 (continued)
**Injection Date**: 1/5/2013 12:33:05 AM  
**Seq. Line**: 6  
**Sample Name**: JC9149A  
**Location**: Vial 6  
**Acq. Operator**: CE  
**Acq. Instrument**: Instrument 1  
**Inj Volume**: 15 µl  
**Actual Inj Volume**: 5 µl  
**Acq. Method**: C:\HPCHEM\1\METHODS\AD-02-40.M  
**Last changed**: 1/4/2013 10:05:27 PM by CE  
**Analysis Method**: C:\HPCHEM\1\METHODS\OD-02-20.M  
**Last changed**: 7/30/2014 4:58:11 PM by MK

### Area Percent Report

**Sorted By**: Signal  
**Multiplier**: 1.0000  
**Dilution**: 1.0000  
**Use Multiplier & Dilution Factor with ISTDs**

| Signal | Description          | Peak RetTime | Width | Area (mAU*s) | Height (mAU) | Area % |
|--------|----------------------|--------------|-------|--------------|--------------|--------|
| 1      | DAD1 A, Sig=250,10 Ref=360,100 | 25.848 MM | 0.7041 | 1.20400e4 | 285.00970 | 96.7508 |
| 2      | DAD1 B, Sig=254,10 Ref=360,100 | 28.891 MM | 0.7018 | 404.33865 | 9.60306 | 3.2492 |
| Totals |                      |              |       | 1.24443e4 | 294.61277 |

Results obtained with enhanced integrator!

| Signal | Description          | Peak RetTime | Width | Area (mAU) | Height |
|--------|----------------------|--------------|-------|------------|--------|
| 4      | DAD1 D, Sig=230,10 Ref=360,100 | 25.848 MM | 0.7041 | 1.20400e4 | 285.00970 |
| 5      | DAD1 E, Sig=280,10 Ref=360,100 | 28.891 MM | 0.7018 | 404.33865 | 9.60306 |

**Table 2**: entry 3  
**Table 1**: entry 4

---

**Area Percent Report**

---

**Data File**: C:\HPCHEM\1\DATA\201301~1\JC9149A2.D  
**Sample Name**: JC9149A  
**Instrument 1**: 7/30/2014 4:58:16 PM MK
## Area Percent Report

### Sorted By: Signal

**Multiplier:** 1.0000  
**Dilution:** 1.0000  

**Use Multiplier & Dilution Factor with ISTDs**

#### Signal 1: DAD1 A, Sig=250,10 Ref=360,100

| Peak RetTime Type | Width   | Area       | Height   | Area %  |
|-------------------|---------|------------|----------|---------|
| #1                | [min]   | [min]     | [mAU*s] | [mAU]   | %       |
|                  | 25.969  | 0.6423     | 997.43585 | 25.88220 | 3.3740  |
|                  | 28.316  | 0.8177     | 2.85648e4 | 582.20422 | 96.6260 |

**Totals:** 2.95622e4 608.08643

Results obtained with enhanced integrator!

#### Signal 2: DAD1 B, Sig=254,10 Ref=360,100

#### Signal 3: DAD1 C, Sig=210,10 Ref=360,100

#### Signal 4: DAD1 D, Sig=230,10 Ref=360,100

#### Signal 5: DAD1 E, Sig=280,10 Ref=360,100

---

### Diagrams

- Graph A: DAD1 A, Sig=250,10 Ref=360,100
- Graph B: DAD1 B, Sig=254,10 Ref=360,100
- Graph C: DAD1 C, Sig=210,10 Ref=360,100
- Graph D: DAD1 D, Sig=230,10 Ref=360,100
- Graph E: DAD1 E, Sig=280,10 Ref=360,100
Area Percent Report

Signal 1: DAD1 A, Sig=250,10 Ref=360,100
Signal 2: DAD1 B, Sig=254,10 Ref=360,100
Signal 3: DAD1 C, Sig=210,10 Ref=360,100

| Peak RetTime Type | Width | Area      | Height     | Area       |
|------------------|-------|-----------|------------|------------|
| #    | [min] |   [min]   | [mAU*s]    | [mAU]      | %          |
| 1    | 13.813 | 0.3644    | 309.33765  | 14.14928   | 1.9693     |
| 2    | 20.195 | 0.5342    | 1.53989e4  | 480.47076  | 98.0307    |

Totals: 1.57082e4  494.62004

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

--- End of Report ---
| Peak RetTime Type | Width  | Area          | Height   | Area % |
|------------------|--------|---------------|----------|--------|
| MM               | 0.3734 | 1.43263e4     | 639.37732| 98.1776|
| MM               | 0.4779 | 265.93488     | 9.27401  | 1.8224 |

Totals: 1.45922e4 648.65133

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

---

**Area Percent Report**

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100
Signal 2: DAD1 B, Sig=254,10 Ref=360,100
Signal 3: DAD1 C, Sig=210,10 Ref=360,100

---

*** End of Report ***
Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100
Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  13.872 MM    0.4517 2.92907e4  1080.68958  98.9997

Signal 2: DAD1 B, Sig=254,10 Ref=360,100
Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  16.710 MM    0.4686  295.95520   10.52571   1.0003

Signal 3: DAD1 C, Sig=210,10 Ref=360,100
Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  13.873 MM    0.4361 5596.80615  213.91138  99.0331
2  16.712 MM    0.4778   54.64594    1.90619   0.9669

Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  13.873 MM    0.4361 5596.80615  213.91138  99.0331
2  16.712 MM    0.4778   54.64594    1.90619   0.9669

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Totals :                  2.95866e4  1091.21529

Results obtained with enhanced integrator!

Signal 6: DAD1 E, Sig=280,10 Ref=360,100
Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  13.873 MM    0.4361 5596.80615  213.91138  99.0331
2  16.712 MM    0.4778   54.64594    1.90619   0.9669

Totals :                  5651.45210  215.81756

Results obtained with enhanced integrator!

*** End of Report ***
### Area Percent Report

```
| Signal   | Ret Time [min] | Width [min] | Area [mAU*s] | Height [mAU] | %     |
|----------|----------------|-------------|--------------|--------------|-------|
| DAD1 A   | 13.310         | 0.3540      | 408.69366    | 19.24129     | 2.3091 |
| DAD1 B   | 21.041         | 0.6492      | 1.72903e4    | 443.91245    | 97.6909 |
| DAD1 C   | 13.307         | 0.3703      | 81.74083     | 3.67898      | 2.4967 |
| DAD1 D   | 21.041         | 0.6541      | 3192.27197   | 81.33752     | 97.5033 |
```

```
Totals: Area [mAU*s] = 1.76990e4, Height [mAU] = 463.15373
```

Results obtained with enhanced integrator!

### Signal 4: DAD1 D, Sig=230,10 Ref=360,100

```
| Signal   | Ret Time [min] | Width [min] | Area [mAU*s] | Height [mAU] | %     |
|----------|----------------|-------------|--------------|--------------|-------|
| DAD1 D   | 13.307         | 0.3703      | 81.74083     | 3.67898      | 2.4967 |
| DAD1 D   | 21.041         | 0.6541      | 3192.27197   | 81.33752     | 97.5033 |
```

```
Totals: Area [mAU*s] = 3274.01280, Height [mAU] = 85.01650
```

Results obtained with enhanced integrator!

### Signal 5: DAD1 E, Sig=280,10 Ref=360,100

---

**End of Report**
Injection Date  : 2/22/2013 11:28:27 AM          Seq. Line :   2
Sample Name     : JC9221A                         Location : Vial 9
Acq. Operator   : CE                                   Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\OD-05-40.M
Last changed    : 4/7/2011 5:39:35 PM by CC
Analysis Method : C:\HPCHEM\1\METHODS\OD-02-20.M
Last changed    : 7/30/2014 5:09:46 PM by MK
(modified after loading)

| Peak RetTime Type  | Width     | Area      | Height     | Area     |
|--------------------|-----------|-----------|------------|----------|
|                    |           |           |            |          |
|                    | #         | [min]     | [min]      | [mAU*s]  | [mAU]    | %         |
|--------------------|-----------|-----------|------------|----------|
| 1                  | MM        | 18.257    | 0.5628     | 1.29286e4| 382.84482| 95.1621   |
| 2                  | MM        | 22.785    | 0.6684     | 657.27887| 16.39051 | 4.8379    |
| Totals             |           |           |            | 1.35859e4| 399.23533|
| Results obtained with enhanced integrator!
| 3                  | BB        | 18.259    | 0.5223     | 1.15049e4| 342.18851| 95.1596   |
| 4                  | BB        | 22.773    | 0.5728     | 585.20709| 14.37923 | 4.8404    |
| Totals             |           |           |            | 1.20901e4| 356.56774|
| Results obtained with enhanced integrator!

**Area Percent Report**

**Sorted By**             :      Signal
**Multiplier**            :      1.0000
**Dilution**              :      1.0000

Signal 1: DAD1 A, Sig=250,10 Ref=360,100
Signal 2: DAD1 B, Sig=254,10 Ref=360,100
Signal 3: DAD1 C, Sig=210,10 Ref=360,100
Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

---

**Area Percent Report**

| Peak RetTime Type  | Width     | Area      | Height     | Area     |
|--------------------|-----------|-----------|------------|----------|
|                    |           |           |            |          |
|                    | #         | [min]     | [min]      | [mAU*s]  | [mAU]    | %         |
|--------------------|-----------|-----------|------------|----------|
| 1                  | MM        | 18.257    | 0.5628     | 1.29286e4| 382.84482| 95.1621   |
| 2                  | MM        | 22.785    | 0.6684     | 657.27887| 16.39051 | 4.8379    |
| Totals             |           |           |            | 1.35859e4| 399.23533|
| Results obtained with enhanced integrator!
| 3                  | BB        | 18.259    | 0.5223     | 1.15049e4| 342.18851| 95.1596   |
| 4                  | BB        | 22.773    | 0.5728     | 585.20709| 14.37923 | 4.8404    |
| Totals             |           |           |            | 1.20901e4| 356.56774|
| Results obtained with enhanced integrator!

---

Table 2: entry 8

---

Data File C:\HPCHEM\1\DATA\201302~1\JC9221A1.D                                    Sample Name: JC9221A
Instrument 1 7/30/2014 5:09:47 PM MK

---

Table 1: entry 10

---

S–104

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S–104
Injection Date: 2/22/2013 12:09:44 PM
Seq. Line: 3
Sample Name: JC9221B
Location: Vial 10
Acq. Operator: CE
Inj: 1
Acq. Instrument: Instrument 1
Inj Volume: 15 µl
Different Inj Volume from Sequence!
Actual Inj Volume: 5 µl
Acq. Method: C:\HPCHEM\1\METHODS\OD-05-40.M
Last changed: 4/7/2011 5:39:35 PM by CC
Analysis Method: C:\HPCHEM\1\METHODS\AD005-40.M
Last changed: 8/2/2014 9:04:09 PM by MK
(modified after loading)

Area: 369.912 18.990
Area: 8082.46 23.052

Area: 309.103 18.962
Area: 7213.13 23.055

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Peak RetTime Type Width Area Height Area
# [min] [min] [mAU*s] [mAU] [%]
--|--|--|--|--|--|--|
1 18.962 MM 0.5785 309.10349 8.90468 4.1092
2 23.055 MM 0.6991 7213.12598 171.96364 95.8908
Totals: 7522.22946 180.86832

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100
Peak RetTime Type Width Area Height Area
# [min] [min] [mAU*s] [mAU] [%]
--|--|--|--|--|--|--|
1 18.990 MM 0.5818 369.91183 10.59696 4.3764
2 23.052 MM 0.6975 8082.46289 193.11874 95.6236
Totals: 8452.37473 203.71571

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100
Signal 3: DAD1 C, Sig=210,10 Ref=360,100
Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Peak RetTime Type Width Area Height Area
# [min] [min] [mAU*s] [mAU] [%]
--|--|--|--|--|--|--|
1 18.990 MM 0.5818 369.91183 10.59696 4.3764
2 23.052 MM 0.6975 8082.46289 193.11874 95.6236
Totals: 8452.37473 203.71571

Results obtained with enhanced integrator!

Peak RetTime Type Width Area Height Area
# [min] [min] [mAU*s] [mAU] [%]
--|--|--|--|--|--|--|
1 18.962 MM 0.5785 309.10349 8.90468 4.1092
2 23.055 MM 0.6991 7213.12598 171.96364 95.8908
Totals: 7522.22946 180.86832

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Table S-105

\[ \text{MeN-S} \cdot \text{O}\]
\[ \text{Ph} \]

Table 2: S-8 L1

Page 1 of 2
### Area Percent Report

**Sorted By:** Signal  
**Multiplier:** 1.0000  
**Dilution:** 1.0000  

Use Multiplier & Dilution Factor with ISTDs

| Signal   | Type  | Width | Area      | Height     | Area % |
|----------|-------|-------|-----------|------------|--------|
| DAD1 A   | MM    | 0.374 | 11488.9   | 512.01752  | 100.00 |
| DAD1 B   | MM    | 0.374 | 11488.9   | 512.01752  | 100.00 |
| DAD1 C   | MM    | 0.374 | 11488.9   | 512.01752  | 100.00 |
| DAD1 D   | MM    | 0.374 | 11488.9   | 512.01752  | 100.00 |
| DAD1 E   | MM    | 0.374 | 11488.9   | 512.01752  | 100.00 |

**Results obtained with enhanced integrator!**

**Signal 4:** DAD1 D, Sig=230, Ref=360,100  
**Signal 5:** DAD1 E, Sig=280, Ref=360,100

---

**End of Report**

---

**Data File:** C:\HPCHEM\1\DATA\GROUP\JC12043A.D  
**Sample Name:** JC12043A  
**Instrument 1:** 7/30/2014 6:54:57 PM MK

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**Diagram:**

- Chart 1: Graph of data with peaks labeled.
- Chart 2: Molecular structure of MeCN.
- Chart 3: Graph showing area and width measurements.
- Chart 4: Graph displaying peak retention times.
- Chart 5: Graph showing integration results.
Injection Date  : 6/16/2014 8:19:00 PM           Seq. Line :   5
Sample Name     : JC12043B                        Location : Vial 12
Acq. Operator   : MK                                   Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 6 µl
Acq. Method     : C:\HPCHEM\1\METHODS\OD-01-30.M
Last changed    : 6/16/2014 7:51:29 PM by MK
(modified after loading)
Analysis Method : C:\HPCHEM\1\METHODS\AD005-40.M
Last changed    : 8/1/2014 11:32:53 PM by MK
(modified after loading)

| Peak RetTime | Width  | Area       | Height  | Area Percent |
|--------------|--------|------------|---------|--------------|
| #             | [min]  | [min]      | [mAU*s] | [mAU]        | %            |
| 1             | 14.572 | 0.3958     | 7769.50049 | 327.13113   | 100.0000     |

Totals :                  7769.50049  327.13113

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

**Area Percent Report**

| Sorted By             | Multiplier            | Dilution              |
|-----------------------|-----------------------|-----------------------|
|                       | 1.0000                | 1.0000                |

User Multiplier & Dilution Factor with ISTDs

---

**Table 2**

With (S,S)-1-L

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**Graphical Data**

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Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100
Signal 2: DAD1 B, Sig=254,10 Ref=360,100
Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  17.558 MM    0.4595 5773.34912  209.42706  97.0125
2  20.783 MM    0.5738  177.79001    5.16444   2.9875

Totals :                  5951.13913  214.59150

Results obtained with enhanced integrator!
Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

*** End of Report ***
Injection Date  : 7/20/2013 9:37:03 AM           Seq. Line :  34
Sample Name     : JC10185                         Location : Vial 82
Acq. Operator   : CE                                   Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\AD-04-30.M
Last changed    : 11/29/2010 7:04:08 PM by JTM
Analysis Method : C:\HPCHEM\1\METHODS\OD-02-20.M
Last changed    : 7/30/2014 6:59:32 PM by MK
(modified after loading)

Area: 22.9571
16.698

Area: 9479.26
25.782

Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100
Signal 2: DAD1 B, Sig=254,10 Ref=360,100
Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  16.698 MM    0.2409   22.95708    1.58850   0.2416
2  25.782 MM    0.5562 9479.26074  284.07181  99.7584

Totals :                  9502.21782  285.66031

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

*** End of Report ***
### Area Percent Report

**Sorted By**: Signal 1: DAD1 A, Sig=250,10 Ref=360,100  
**Multiplier**: 1.0000  
**Dilution**: 1.0000

| Peak RetTime Type  Width     Area      Height     Area |
|----------------------|------------------|------------------|------------------|
| #   | [min] | [min] | [mAU*s] | [mAU] | %    |
| ---- |-------|-------|---------|--------|-----|
| 1    | 33.929 | MF    | 1.3461  | 2160.27637 | 26.74717 | 4.5480 |
| 2    | 41.679 | MF    | 1.8592  | 4.53393e4  | 406.43207 | 95.4520 |

**Totals**: 4.74996e4  
**Area Percent**: 433.17924

Results obtained with enhanced integrator!

**Signal 4**: DAD1 D, Sig=230,10 Ref=360,100  
**Signal 5**: DAD1 E, Sig=280,10 Ref=360,100

---

**End of Report**
Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100
Signal 2: DAD1 B, Sig=254,10 Ref=360,100
Signal 3: DAD1 C, Sig=210,10 Ref=360,100
Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1   8.444 MM    0.1553 6512.97803  698.96423  99.7435
2   9.932 MM    0.1343   16.74774    2.07831   0.2565
Totals :                  6529.72577  701.04255

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1   8.444 FM    0.1553 2352.96094  252.45752  99.5957
2   9.930 MM    0.1630    9.55209 9.76556e-1   0.4043
Totals :                  2362.51303  253.43408

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100
### Table 3

| Signal | RetTime Type | Width | Area      | Height | Area % |
|--------|--------------|-------|-----------|--------|--------|
| **DAD1 A** | 8.381 MM | 0.1398 | 35.99236 | 4.28965 | 0.6915 |
| **DAD1 B** | 9.743 MM | 0.1683 | 5168.82764 | 511.87228 | 99.3085 |
| **DAD1 C** | 8.373 VB | 0.1312 | 22.56680 | 2.60208 | 1.2052 |
| **DAD1 D** | 9.743 MM | 0.1686 | 1849.83777 | 182.89543 | 98.7948 |
| **DAD1 E** | 9.743 MM | 0.1699 | 1849.83777 | 182.89543 | 98.7948 |

### Table 4

**Peak RetTime Type** | **Width** | **Area** | **Height** | **Area %**
--- | --- | --- | --- | ---
1. | 8.381 MM | 0.1398 | 35.99236 | 4.28965 | 0.6915 |
2. | 9.743 MM | 0.1683 | 5168.82764 | 511.87228 | 99.3085 |
1. | 8.373 VB | 0.1312 | 22.56680 | 2.60208 | 1.2052 |
2. | 9.743 MM | 0.1686 | 1849.83777 | 182.89543 | 98.7948 |

**Area Percent Summary**

- **Signal 1**: 0.6915%
- **Signal 2**: 99.3085%
- **Signal 3**: 1.2052%
- **Signal 4**: 98.7948%

**Results obtained with enhanced integrator!**
| Signal 1: DAD1 A, Sig=250,10 Ref=360,100 | Area: 12157.6 mAU | RetTime: 13.582 min |
|----------------------------------------|------------------|-------------------|
| Signal 2: DAD1 B, Sig=254,10 Ref=360,100 | Area: 955.983 mAU | RetTime: 18.672 min |
| Signal 3: DAD1 C, Sig=210,10 Ref=360,100 | Area: 3889.759 mAU | RetTime: 13.582 min |
| Signal 4: DAD1 D, Sig=230,10 Ref=360,100 | Area: 297.191 mAU | RetTime: 18.670 min |
| Signal 5: DAD1 E, Sig=280,10 Ref=360,100 | Area: 3889.759 mAU | RetTime: 13.582 min |

Results obtained with enhanced integrator!
### Area Percent Report

| Signal                  | Peak RetTime [min] | Width [min] | Area [mAU*s] | Height [mAU] | %       |
|-------------------------|-------------------|-------------|--------------|--------------|---------|
| DAD1 A, Sig=250,10 Ref=360,100 | 14.425            | 0.4183      | 901.16510    | 35.90579     | 8.4785  |
| DAD1 A, Sig=254,10 Ref=360,100 | 19.379            | 0.5770      | 9727.60840   | 280.98239    | 91.5215 |
| Totals                  |                   |             | 1.06288e4    | 316.88818    |         |

Results obtained with enhanced integrator!

| DAD1 B, Sig=210,10 Ref=360,100 | 14.429            | 0.4146      | 279.89063    | 11.25042     | 8.3390  |
| DAD1 B, Sig=230,10 Ref=360,100 | 19.381            | 0.5735      | 3076.50317   | 89.40532     | 91.6610 |
| Totals                  |                   |             | 3356.39380   | 100.65574    |         |

Results obtained with enhanced integrator!

### S-117

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**Area Process Report**

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Table 3: Entry 5

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Data File C:\HPCHEM\1\DATA\201212~1\JC9119B.D
Sample Name: JC9119B
Instrument 1 8/2/2014 10:22:01 PM MK

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Page 2 of 2
Injection Date: 2/16/2013 5:08:58 PM  
Seq. Line: 2  
Sample Name: JC9225  
Location: Vial 7  
Acq. Operator: CE  
Inj: 1  
Acq. Instrument: Instrument 1  
Inj Volume: 15 µl  
Different Inj Volume from Sequence!  
Actual Inj Volume: 5 µl  
Acq. Method: C:\HPCHEM\1\METHODS\OD-02-30.M  
Last changed: 2/16/2013 4:45:43 PM by CE  
Analysis Method: C:\HPCHEM\1\METHODS\OD-02-20.M  
Last changed: 7/30/2014 7:16:00 PM by MK

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100
Signal 2: DAD1 B, Sig=254,10 Ref=360,100
Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1   8.120 MM    0.2662 1.62037e4  1014.59381  97.9659
2  10.117 MM    0.2798  336.43698   20.03921   2.0341

Totals: 1.65402e4 1034.63302

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1   8.119 MM    0.2655 1.16168e4   729.36182  97.8639
2  10.121 MM    0.2835  253.56413   14.90512   2.1361

Totals: 1.18703e4 744.26694

Results obtained with enhanced integrator!

*** End of Report ***
### Injection Date

Injection Date: 2/16/2013 5:40:14 PM  
Seq. Line: 3

### Sample Name

Sample Name: JC9229  
Location: Vial 8

### Acq. Operator

Acq. Operator: CE  
Inj: 1

### Acq. Instrument

Acq. Instrument: Instrument 1  
Inj Volume: 15 µl

Different Inj Volume from Sequence!  
Actual Inj Volume: 5 µl

### Acq. Method

Acq. Method: C:\HPCHEM\1\METHODS\OD-02-30.M  
Last changed: 2/16/2013 4:45:43 PM by CE

### Analysis Method

Analysis Method: C:\HPCHEM\1\METHODS\AD005-40.M  
Last changed: 8/1/2014 11:49:11 PM by MK (modified after loading)

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### Area Percent Report

#### Sorted By: Signal

Multiplier: 1.0000  
Dilution: 1.0000

Use Multiplier & Dilution Factor with ISTDs

**Signal 1: DAD1 A, Sig=250,10 Ref=360,100**

**Signal 2: DAD1 B, Sig=254,10 Ref=360,100**

**Signal 3: DAD1 C, Sig=210,10 Ref=360,100**

| Peak RetTime | Type Width | Area mAU*s | Height mAU | Area % |
|--------------|------------|------------|------------|--------|
| 8.212        | MM 0.2747  | 512.46289  | 31.09460   | 2.5090 |
| 10.047       | MM 0.3127  | 1.99125e4  | 1061.17505 | 97.4910 |

**Totals:** 2.04249e4 1092.26965

Results obtained with enhanced integrator!

**Signal 4: DAD1 D, Sig=230,10 Ref=360,100**

| Peak RetTime | Type Width | Area mAU*s | Height mAU | Area % |
|--------------|------------|------------|------------|--------|
| 8.212        | MM 0.2489  | 330.94400  | 22.15661   | 2.2587 |
| 10.047       | MM 0.3138  | 1.43211e4  | 760.68982  | 97.7413 |

**Totals:** 1.46520e4 782.84643

Results obtained with enhanced integrator!

**Signal 5: DAD1 E, Sig=280,10 Ref=360,100**

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*** End of Report ***
### Area Percent Report

**Sorted By** | **Signal**  
---|---
Multiplier | 1.0000  
Dilution | 1.0000

**Use Multiplier & Dilution Factor with ISTDs**

**Signal 1: DAD1 A, Sig=250,10 Ref=360,100**

| Peak RetTime Type | Width | Area      | Height     | Area %  |
|-------------------|-------|-----------|------------|--------|
| #     | [min] |       |           |        |
| 1   | 8.786 | FM   | 0.2972 | 1.46781e4 | 98.8336 |

**Signal 2: DAD1 B, Sig=254,10 Ref=360,100**

| Peak RetTime Type | Width | Area      | Height     | Area %  |
|-------------------|-------|-----------|------------|--------|
| #     | [min] |       |           |        |
| 2   | 12.039 | MM  | 0.3704 | 173.21939 | 1.1664 |

**Totals:**

- Area: 1.48513e4
- Height: 830.90137

*Results obtained with enhanced integrator!*

**Signal 3: DAD1 C, Sig=210,10 Ref=360,100**

| Peak RetTime Type | Width | Area      | Height     | Area %  |
|-------------------|-------|-----------|------------|--------|
| #     | [min] |       |           |        |
| 1   | 8.787 | FM   | 0.2959 | 8493.53320 | 98.9365 |
| 2   | 12.038 | MM  | 0.3593 | 91.29572   | 1.0635 |

**Totals:**

- Area: 8584.82892
- Height: 482.68535

*Results obtained with enhanced integrator!*

**Signal 4: DAD1 D, Sig=230,10 Ref=360,100**

| Peak RetTime Type | Width | Area      | Height     | Area %  |
|-------------------|-------|-----------|------------|--------|
| #     | [min] |       |           |        |
| 1   | 8.787 | FM   | 0.2959 | 478.45062  | 98.9365 |
| 2   | 12.038 | MM  | 0.3593 | 4.23473    | 1.0635 |

**Totals:**

- Area: 482.68535

**Signal 5: DAD1 E, Sig=280,10 Ref=360,100**

| Peak RetTime Type | Width | Area      | Height     | Area %  |
|-------------------|-------|-----------|------------|--------|
| #     | [min] |       |           |        |

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**Area Percent Report**

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**S–120**

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**Table 4, entry 2**

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**with (R,α-L)-1**

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**O–O**

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**with (R,α-L)-1**

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**O–O**

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**with (R,α-L)-1**

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**O–O**

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**with (R,α-L)-1**

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**O–O**

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**with (R,α-L)-1**

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**O–O**

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**with (R,α-L)-1**

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**O–O**

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**with (R,α-L)-1**

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**O–O**

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**with (R,α-L)-1**

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**O–O**

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**with (R,α-L)-1**

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**O–O**

---

**with (R,α-L)-1**

---

**O–O**

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**with (R,α-L)-1**

---

**O–O**

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**with (R,α-L)-1**

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**O–O**
... End of Report ...
**Area Percent Report**

| Signal 1: DAD1 A, Sig=250,10 Ref=360,100 | Peak RetTime | Type  | Width | Area      | Height     | Area %  |
|----------------------------------------|--------------|-------|-------|-----------|------------|---------|
| #                                       | [min]        |       |       | [mAU*s]   | [mAU]      |        |
| 1                                      | 8.670        | MM    | 0.2815| 1.3982e4  | 827.70404  | 98.6305 |
| 2                                      | 11.410       | MM    | 0.3566| 194.15e3  | 9.07509    | 1.3695  |
| Totals                                 |              |       |       | 1.4176e4  | 836.77913  |         |

Results obtained with enhanced integrator!

| Signal 2: DAD1 B, Sig=254,10 Ref=360,100 | Peak RetTime | Type  | Width | Area      | Height     | Area %  |
|----------------------------------------|--------------|-------|-------|-----------|------------|---------|
| #                                       | [min]        |       |       | [mAU*s]   | [mAU]      |        |
| 1                                      | 8.670        | MM    | 0.2817| 8988.77e3 | 531.88464  | 98.6412 |
| 2                                      | 11.407       | MM    | 0.3539| 123.82e2  | 5.83146    | 1.3588  |
| Totals                                 |              |       |       | 9112.59e3 | 537.71610  |         |

Results obtained with enhanced integrator!

| Signal 3: DAD1 C, Sig=210,10 Ref=360,100 | Peak RetTime | Type  | Width | Area      | Height     | Area %  |
|----------------------------------------|--------------|-------|-------|-----------|------------|---------|
| #                                       | [min]        |       |       | [mAU*s]   | [mAU]      |        |
| 1                                      | 8.669        | MM    | 0.2922| 7836.81e3 | 447.05707  | 98.6123 |
| 2                                      | 11.389       | MM    | 0.3057| 110.28e2  | 6.01225    | 1.3877  |
| Totals                                 |              |       |       | 7947.09e3 | 453.06932  |         |

Results obtained with enhanced integrator!

| Signal 4: DAD1 D, Sig=230,10 Ref=360,100 | Peak RetTime | Type  | Width | Area      | Height     | Area %  |
|----------------------------------------|--------------|-------|-------|-----------|------------|---------|
| #                                       | [min]        |       |       | [mAU*s]   | [mAU]      |        |
| 1                                      | 8.671        | MM    | 0.2825| 1.6267e4  | 959.65015  | 98.6183 |
| 2                                      | 11.422       | MM    | 0.3601| 227.92e2  | 10.54932   | 1.3817  |
| Totals                                 |              |       |       | 1.6495e4  | 970.19947  |         |

Results obtained with enhanced integrator!

| Signal 5: DAD1 E, Sig=280,10 Ref=360,100 | Peak RetTime | Type  | Width | Area      | Height     | Area %  |
|----------------------------------------|--------------|-------|-------|-----------|------------|---------|
| #                                       | [min]        |       |       | [mAU*s]   | [mAU]      |        |
| 1                                      | 8.671        | MM    | 0.2779| 1236.76e3 | 74.16629   | 98.7732 |
| 2                                      | 11.416       | MM    | 0.3130| 15.36e1   | 8.18057e-1 | 1.2268  |
| Totals                                 |              |       |       | 1252.13e3 | 74.98435   |         |

Results obtained with enhanced integrator!
Table 4: Entry 3

| Substance | Rf Value |
|-----------|----------|
| CoCl₂ | 0.58 |
| Co(CN)₅N³⁻ | 0.32 |

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

| Peak RetTime | Width | Area   | Height | Area % |
|--------------|-------|--------|--------|--------|
| 8.758 MM     | 0.2769| 235.331| 14.16449| 1.4574 |
| 11.300 MM    | 0.3687| 1.59122e4| 719.25024| 98.5426 |

Totals: 1.61475e4, 733.41473

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

| Peak RetTime | Width | Area   | Height | Area % |
|--------------|-------|--------|--------|--------|
| 8.754 MM     | 0.2731| 148.941| 9.09059| 1.4356 |
| 11.300 MM    | 0.3685| 1.02257e4| 462.43088| 98.5644 |

Totals: 1.03746e4, 471.52147

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

| Peak RetTime | Width | Area   | Height | Area % |
|--------------|-------|--------|--------|--------|
| 8.777 MM     | 0.2656| 146.912| 9.21852| 1.5848 |
| 11.300 MM    | 0.3831| 9123.17285| 396.85388| 98.4152 |

Totals: 9270.08516, 406.07240

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

| Peak RetTime | Width | Area   | Height | Area % |
|--------------|-------|--------|--------|--------|
| 8.761 MM     | 0.2672| 267.744| 16.69801| 1.4230 |
| 11.299 MM    | 0.3680| 1.85479e4| 840.07916| 98.5770 |

Totals: 1.88156e4, 856.77717

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

| Peak RetTime | Width | Area   | Height | Area % |
|--------------|-------|--------|--------|--------|
| 8.734 MM     | 0.2625| 19.5616| 1.24219| 1.3830 |
| 11.298 MM    | 0.3636| 1394.85559| 63.93124| 98.6170 |

Totals: 1414.41723, 65.17344

Results obtained with enhanced integrator!

*** End of Report ***
### Area Percent Report

| Signal 1: DAD1 A, Sig=250,10 Ref=360,100 | Peak RetTime Type | Width | Area      | Height | Area   | %       |
|----------------------------------------|------------------|-------|-----------|--------|--------|---------|
|                                          | [min]            | [min] | [mAU*s]   | [mAU]  |        |         |
| 1                                      | 10.467           | 0.3598| 2.52725e4| 1170.62231| 97.4488|         |
| 2                                      | 12.915           | 0.3802| 661.61938| 29.00374 | 2.5512 |         |
| **Totals**                             |                  |       | 2.59342e4| 1199.62605 |       |         |

Results obtained with enhanced integrator!

| Signal 2: DAD1 B, Sig=254,10 Ref=360,100 | Peak RetTime Type | Width | Area      | Height | Area   | %       |
|----------------------------------------|------------------|-------|-----------|--------|--------|---------|
|                                          | [min]            | [min] | [mAU*s]   | [mAU]  |        |         |
| 1                                      | 10.467           | 0.3264| 1.13284e4| 578.50757| 97.7955|         |
| 2                                      | 12.912           | 0.3912| 255.36906| 10.87842 | 2.2045 |         |
| **Totals**                             |                  |       | 1.15838e4| 589.38598 |       |         |

Results obtained with enhanced integrator!

| Signal 3: DAD1 C, Sig=210,10 Ref=360,100 | Peak RetTime Type | Width | Area      | Height | Area   | %       |
|----------------------------------------|------------------|-------|-----------|--------|--------|---------|
|                                          | [min]            | [min] | [mAU*s]   | [mAU]  |        |         |
| 1                                      | 10.467           | 0.3209| 5006.08350| 260.03439| 97.9882|         |
| 2                                      | 12.916           | 0.3720| 102.78178| 4.60473 | 2.0118 |         |
| **Totals**                             |                  |       | 5108.86527| 264.63913 |       |         |

Results obtained with enhanced integrator!

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**Area:** 25272.5 10.467
**Area:** 661.619 12.915
**Area:** 11328.4 10.467
**Area:** 255.369 12.912
**Area:** 5006.08 10.467
**Area:** 102.782 12.916
S-125

Table 4. Entry 4

Table with (S,S)-1L

S–125

Sample Name: JC9159B

Injection Date: 1/17/2013 6:11:02 PM

Sample Name: JC9159B

Inj: 1

Acq. Operator: CE

Acq. Method: C:\HPCHEM\1\METHODS\OD-03-30.M

Acq. Instrument: Instrument 1

Last changed: 12/31/2012 2:58:25 PM by CE

Analysis Method: C:\HPCHEM\1\METHODS\AD005-40.M

Last changed: 8/1/2014 11:54:28 PM by MK

(modified after loading)

Area: 703.807

11.314

Area: 30989.3

13.703

Area: 250.681

11.310

Area: 13574.8

13.703

Area: 109.32

11.311

Area: 5959.29

13.703

Data File C:\HPCHEM\1\DATA\201301~1\JC9159B1.D

Sample Name: JC9159B
### Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

| Signal 1: DAD1 A, Sig=250,10 Ref=360,100 |  |
|------------------------------------------|--|
| Peak RetTime | Type | Width | Area | Height | Area % |
| 16.642 MM | MM | 0.4739 | 1.61671e4 | 568.63507 | 98.0867 |
| Totals: | | | 1.64825e4 | 579.69842 |

Results obtained with enhanced integrator!

| Signal 2: DAD1 B, Sig=254,10 Ref=360,100 |  |
|------------------------------------------|--|
| Peak RetTime | Type | Width | Area | Height | Area % |
| 19.290 MM | MM | 0.4751 | 315.36053 | 11.06335 | 1.9133 |

| Signal 3: DAD1 C, Sig=210,10 Ref=360,100 |  |
|------------------------------------------|--|
| Peak RetTime | Type | Width | Area | Height | Area % |
| Totals: | | | 8328.55867 | 299.39160 |

Results obtained with enhanced integrator!

| Signal 4: DAD1 D, Sig=230,10 Ref=360,100 |  |
|------------------------------------------|--|
| Peak RetTime | Type | Width | Area | Height | Area % |
| 16.641 MM | MM | 0.4629 | 8169.60059 | 294.14471 | 98.0914 |
| Totals: | | | 8328.55867 | 299.39160 |

Results obtained with enhanced integrator!

| Signal 5: DAD1 E, Sig=280,10 Ref=360,100 |  |
|------------------------------------------|--|
| Peak RetTime | Type | Width | Area | Height | Area % |
| Totals: | | | 8328.55867 | 299.39160 |
### Injection Details

- **Injection Date:** 8/6/2013 12:36:17 AM
- **Seq. Line:** 3
  - **Sample Name:** JC10223B
- **Location:** Vial 95
- **Acq. Operator:** MK
- **Acq. Instrument:** Instrument 1
  - **Inj Volume:** 15 µl
    - *Actual Inj Volume:* 5 µl
- **Acq. Method:** C:\HPCHEM\1\METHODS\AS-05-40.M
- **Last changed:** 4/7/2011 5:40:42 PM by CC
- **Analysis Method:** C:\HPCHEM\1\METHODS\AD005-40.M
- **Last changed:** 8/1/2014 11:56:57 PM by MK

### Area Percent Report

**Sorted By:** Signal

**Multiplier:** 1.0000

**Dilution:** 1.0000

**Use Multiplier & Dilution Factor with ISTDs**

**Signal 1:** DAD1 A, Sig=250,10 Ref=360,100
- **Peak RetTime:** 16.717 [min]
- **Type:** MM
- **Width:** 0.4488 [min]
- **Area:** 433.83279 [mAU*s]
- **Height:** 16.11137 [mAU]
- **Area %:** 2.1630

**Signal 2:** DAD1 B, Sig=254,10 Ref=360,100
- **Peak RetTime:** 19.053 [min]
- **Type:** MM
- **Width:** 0.5754 [min]
- **Area:** 1.96231e4 [mAU*s]
- **Height:** 568.36749 [mAU]
- **Area %:** 97.8370

**Totals:**
- **Area:** 2.00569e4 [mAU*s]
- **Height:** 584.47886 [mAU]

**Results obtained with enhanced integrator!**

**Signal 3:** DAD1 C, Sig=210,10 Ref=360,100
- **Peak RetTime:** 16.722 [min]
- **Type:** MM
- **Width:** 0.4358 [min]
- **Area:** 196.73267 [mAU*s]
- **Height:** 7.52359 [mAU]
- **Area %:** 1.9765

**Signal 4:** DAD1 D, Sig=230,10 Ref=360,100
- **Peak RetTime:** 16.722 [min]
- **Type:** MM
- **Width:** 0.4358 [min]
- **Area:** 196.73267 [mAU*s]
- **Height:** 7.52359 [mAU]
- **Area %:** 1.9765

**Signal 5:** DAD1 E, Sig=280,10 Ref=360,100
- **Peak RetTime:** 16.730 [min]
- **Type:** BP
- **Width:** 0.3569 [min]
- **Area:** 92.86021 [mAU*s]
- **Height:** 3.75030 [mAU]
- **Area %:** 1.8070

**Signal 6:** DAD1 F, Sig=300,10 Ref=360,100
- **Peak RetTime:** 16.730 [min]
- **Type:** BB
- **Width:** 0.3569 [min]
- **Area:** 92.86021 [mAU*s]
- **Height:** 3.75030 [mAU]
- **Area %:** 1.8070

**Totals:**
- **Area:** 5138.85484 [mAU*s]
- **Height:** 154.39329 [mAU]

**Results obtained with enhanced integrator!**

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### Page 2 of 2
### Area Percent Report

| Signal | Multiplier | Dilution | Use Multiplier & Dilution Factor with ISTDs | Area Percent Report |
|--------|------------|----------|---------------------------------------------|---------------------|
| DAD1 A, Sig=250,10 Ref=360,100 | 1.0000 | 1.0000 | | |
| DAD1 B, Sig=254,10 Ref=360,100 | 1.0000 | 1.0000 | | |
| DAD1 C, Sig=210,10 Ref=360,100 | 1.0000 | 1.0000 | | |
| DAD1 D, Sig=230,10 Ref=360,100 | 1.0000 | 1.0000 | | |
| DAD1 E, Sig=280,10 Ref=360,100 | 1.0000 | 1.0000 | | |

| Peak RetTime Type | Width | Area      | Height | Area Percentage |
|-------------------|-------|-----------|--------|-----------------|
| #     | [min] |   [min]   | [mAU*s] | [mAU]           | %               |
| 1     | 9.763 | 0.3255    | 29017.8 | 1485.83618      | 98.2944         |
| 2     | 11.939| 0.2984    | 503.50601| 28.12491       | 1.7056          |
| Totals|       |           | 29521.3 | 1513.96109      |                 |
| 1     | 9.764 | 0.2880    | 9872.96777| 571.38434     | 98.4903         |
| 2     | 11.937| 0.2972    | 151.33591| 8.48584       | 1.5097          |
| Totals|       |           | 10024.3 | 579.87018       |                 |

Results obtained with enhanced integrator!

### Table 4; entry G

| Compound | RetTime | Area | Height | Area Percentage |
|----------|---------|------|--------|-----------------|
| 1        |         |      |        |                 |
| 2        |         |      |        |                 |
| 3        |         |      |        |                 |

Results obtained with enhanced integrator!
Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100
Signal 2: DAD1 B, Sig=254,16 Ref=360,100
Signal 3: DAD1 C, Sig=210,8 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  16.923 MM    0.4228  278.14233  10.96450    1.4127
2  22.703 MM    0.5880 1.94105e4  550.20209  98.5873
Totals :                  1.96886e4   561.16659

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100
Signal 5: DAD1 E, Sig=280,16 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  16.924 MM    0.4149   81.13730    3.25970    1.3945
2  22.703 MM    0.5891 5737.36523  162.31212  98.6055
Totals :                  5818.50253  165.57181

Results obtained with enhanced integrator!

*** End of Report ***
Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=254,4 Ref=360,100
Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  16.838 MM    0.4381 1.56666e4   596.02325  98.6193

Totals :                  1.58860e4   602.64157
Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,16 Ref=360,100
Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  16.838 MM    0.4396 4621.76465  175.22726  98.5882
2  22.878 MM    0.5719   66.18513    1.92889   1.4118

Totals :                  4687.94978  177.15616
Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,16 Ref=360,100

*** End of Report ***
Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

| # | Ret Time | Width | Area   | Height | %     |
|---|----------|-------|--------|--------|-------|
| 1 | 11.658   | 0.400 | 4706.69 | 195.89 | 94.11 |
| 2 | 15.406   | 0.537 | 294.53  | 9.14   | 5.89  |

Totals: 5001.22 Area, 205.04 Height, 94.14 %

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

| # | Ret Time | Width | Area   | Height | %     |
|---|----------|-------|--------|--------|-------|
| 1 | 11.659   | 0.401 | 4211.18 | 175.21 | 94.15 |
| 2 | 15.406   | 0.536 | 261.79  | 8.13   | 5.85  |

Totals: 4472.96 Area, 183.35 Height, 94.15 %

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

| # | Ret Time | Width | Area   | Height | %     |
|---|----------|-------|--------|--------|-------|
| 1 | 11.659   | 0.401 | 5285.32 | 219.49 | 94.06 |
| 2 | 15.420   | 0.379 | 333.85  | 10.61  | 5.94  |

Totals: 5619.17 Area, 230.10 Height, 94.06 %

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

| # | Ret Time | Width | Area   | Height | %     |
|---|----------|-------|--------|--------|-------|
| 1 | 11.658   | 0.370 | 1.321e4 | 550.22 | 94.13 |
| 2 | 15.406   | 0.498 | 824.43  | 25.86  | 5.87  |

Totals: 1.4036e4 Area, 576.08 Height, 94.13 %

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100

| # | Ret Time | Width | Area   | Height | %     |
|---|----------|-------|--------|--------|-------|
| 1 | 11.659   | 0.400 | 1143.70 | 47.65  | 94.19 |
| 2 | 15.379   | 0.531 | 70.54   | 2.21   | 5.81  |

Totals: 1214.24 Area, 49.87 Height, 94.19 %

Results obtained with enhanced integrator!

=====================================================================
### Area Percent Report

| Signal | Peak RetTime | Width | Area   | Height | Percentage |
|--------|--------------|-------|--------|--------|------------|
| **Signal 1: DAD1 A, Sig=250,10 Ref=360,100** | 11.708 MM | 0.4004 | 459.712 | 19.13779 | 4.8354 |
|  | 15.305 MM | 0.5404 | 9047.599 | 279.01901 | 95.1646 |
| **Totals** | | | 9507.31055 | 298.15680 | |
| **Results obtained with enhanced integrator!** | | | | | |
| **Signal 2: DAD1 B, Sig=254,10 Ref=360,100** | 11.706 MM | 0.4003 | 410.98477 | 17.11033 | 4.8296 |
|  | 15.305 MM | 0.5407 | 8098.67578 | 249.64102 | 95.1704 |
| **Totals** | | | 8509.66055 | 266.75135 | |
| **Results obtained with enhanced integrator!** | | | | | |
| **Signal 3: DAD1 C, Sig=210,10 Ref=360,100** | 11.708 MM | 0.4001 | 523.60632 | 21.81178 | 4.9454 |
|  | 15.306 MM | 0.5401 | 10064.1 | 310.58426 | 95.0546 |
| **Totals** | | | 1.05877e4 | 332.39604 | |
| **Results obtained with enhanced integrator!** | | | | | |
| **Signal 4: DAD1 D, Sig=230,10 Ref=360,100** | 11.708 MM | 0.4006 | 1306.40112 | 54.35100 | 4.8897 |
|  | 15.305 MM | 0.5438 | 2.54111e4 | 778.85779 | 95.1103 |
| **Totals** | | | 2.67175e4 | 833.20879 | |
| **Results obtained with enhanced integrator!** | | | | | |
| **Signal 5: DAD1 E, Sig=280,10 Ref=360,100** | 11.710 MM | 0.3975 | 110.80473 | 4.64648 | 4.8097 |
|  | 15.305 MM | 0.5379 | 2192.98193 | 67.95478 | 95.1903 |
| **Totals** | | | 2303.78667 | 72.60126 | |
| **Results obtained with enhanced integrator!** | | | | | |

### Table 4. any 8

| Compound | Mass Formula | Molar Mass | Retention Time (min) | Area     |
|----------|--------------|------------|----------------------|----------|
| Boon     | (S,S)-L    | 202.1806   | 4.8097               | 4.8097   |
| Met-S     | (S,S)-L    | 264.1806   | 9.6194               | 9.6194   |

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**Data File:** C:\HPCHEM\1\DATA\201306~1\JCX133B1.D
**Sample Name:** JC10133B
**Instrument 1** 8/2/2014 12:08:33 AM MK

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**Table 4. any 8**

| Compound | Mass Formula | Molar Mass | Retention Time (min) | Area     |
|----------|--------------|------------|----------------------|----------|
| Boon     | (S,S)-L    | 202.1806   | 4.8097               | 4.8097   |
| Met-S     | (S,S)-L    | 264.1806   | 9.6194               | 9.6194   |

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**Data File:** C:\HPCHEM\1\DATA\201306~1\JCX133B1.D
**Sample Name:** JC10133B
**Instrument 1** 8/2/2014 12:08:33 AM MK

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**Table 4. any 8**

| Compound | Mass Formula | Molar Mass | Retention Time (min) | Area     |
|----------|--------------|------------|----------------------|----------|
| Boon     | (S,S)-L    | 202.1806   | 4.8097               | 4.8097   |
| Met-S     | (S,S)-L    | 264.1806   | 9.6194               | 9.6194   |

---

**Data File:** C:\HPCHEM\1\DATA\201306~1\JCX133B1.D
**Sample Name:** JC10133B
**Instrument 1** 8/2/2014 12:08:33 AM MK

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**Table 4. any 8**

| Compound | Mass Formula | Molar Mass | Retention Time (min) | Area     |
|----------|--------------|------------|----------------------|----------|
| Boon     | (S,S)-L    | 202.1806   | 4.8097               | 4.8097   |
| Met-S     | (S,S)-L    | 264.1806   | 9.6194               | 9.6194   |

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**Data File:** C:\HPCHEM\1\DATA\201306~1\JCX133B1.D
**Sample Name:** JC10133B
**Instrument 1** 8/2/2014 12:08:33 AM MK

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**Table 4. any 8**

| Compound | Mass Formula | Molar Mass | Retention Time (min) | Area     |
|----------|--------------|------------|----------------------|----------|
| Boon     | (S,S)-L    | 202.1806   | 4.8097               | 4.8097   |
| Met-S     | (S,S)-L    | 264.1806   | 9.6194               | 9.6194   |

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**Data File:** C:\HPCHEM\1\DATA\201306~1\JCX133B1.D
**Sample Name:** JC10133B
**Instrument 1** 8/2/2014 12:08:33 AM MK

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**Table 4. any 8**

| Compound | Mass Formula | Molar Mass | Retention Time (min) | Area     |
|----------|--------------|------------|----------------------|----------|
| Boon     | (S,S)-L    | 202.1806   | 4.8097               | 4.8097   |
| Met-S     | (S,S)-L    | 264.1806   | 9.6194               | 9.6194   |

---

**Data File:** C:\HPCHEM\1\DATA\201306~1\JCX133B1.D
**Sample Name:** JC10133B
**Instrument 1** 8/2/2014 12:08:33 AM MK

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**Table 4. any 8**

| Compound | Mass Formula | Molar Mass | Retention Time (min) | Area     |
|----------|--------------|------------|----------------------|----------|
| Boon     | (S,S)-L    | 202.1806   | 4.8097               | 4.8097   |
| Met-S     | (S,S)-L    | 264.1806   | 9.6194               | 9.6194   |
**Area Percent Report**

**Sorted By**             :      Signal

**Multiplier**            :      1.0000

**Dilution**              :      1.0000

Use Multiplier & Dilution Factor with ISTDs

**Signal 1:** DAD1 A, Sig=250,10 Ref=360,100

**Peak RetTime**

| #   | [min] | Width | Area      | Height     | %        |
|-----|-------|-------|-----------|------------|----------|
| 1   | 17.631| 0.4328| 541.48834 | 20.85328   | 2.1201   |
| 2   | 18.885| 0.5805| 2.49992e4 | 717.76105  | 97.8799  |

** Totals :**                  2.55407e4   738.61432

Results obtained with enhanced integrator!

**Signal 2:** DAD1 B, Sig=254,10 Ref=360,100

**Peak RetTime**

| #   | [min] | Width | Area      | Height     | %        |
|-----|-------|-------|-----------|------------|----------|
| 1   | 17.636| 0.4283| 264.78415 | 10.30480   | 1.8809   |
| 2   | 18.885| 0.5576| 1.38128e4 | 412.84863  | 98.1191  |

** Totals :**                  1.40776e4   423.15343

Results obtained with enhanced integrator!

**Signal 3:** DAD1 C, Sig=210,10 Ref=360,100

**Peak RetTime**

| #   | [min] | Width | Area      | Height     | %        |
|-----|-------|-------|-----------|------------|----------|
| 1   | 17.638| 0.4283| 144.39316 | 5.61829    | 1.8169   |
| 2   | 18.884| 0.5489| 7802.81396| 236.90977  | 98.1831  |

** Totals :**                  7947.20712  242.52807

Results obtained with enhanced integrator!

**Signal 4:** DAD1 D, Sig=230,10 Ref=360,100

**Peak RetTime**

| #   | [min] | Width | Area      | Height     | %        |
|-----|-------|-------|-----------|------------|----------|
| 1   | 17.636| 0.4283| 264.78415 | 10.30480   | 1.8809   |
| 2   | 18.885| 0.5576| 1.38128e4 | 412.84863  | 98.1191  |

** Totals :**                  1.40776e4   423.15343

Results obtained with enhanced integrator!

**Signal 5:** DAD1 E, Sig=280,10 Ref=360,100

**Peak RetTime**

| #   | [min] | Width | Area      | Height     | %        |
|-----|-------|-------|-----------|------------|----------|
| 1   | 17.638| 0.4283| 144.39316 | 5.61829    | 1.8169   |
| 2   | 18.884| 0.5489| 7802.81396| 236.90977  | 98.1831  |

** Totals :**                  7947.20712  242.52807

Results obtained with enhanced integrator!

**Area Percent Report**

**Note:** Results obtained with enhanced integrator!
Injection Date : 8/1/2013 11:34:20 AM           Seq. Line :   3
Sample Name     : JC10219B                        Location : Vial 80
Acq. Operator   : CE                                   Inj :   1
Acq. Instrument : Instrument 1                  Inj Volume : 15 µl
Different Inj Volume from Sequence !     Actual Inj Volume : 5 µl
Acq. Method     : C:\HPCHEM\1\METHODS\OD-05-40.M
Last changed    : 4/7/2011 5:39:35 PM by CC
Analysis Method : C:\HPCHEM\1\METHODS\AD005-40.M
Last changed    : 8/2/2014 12:10:26 AM by MK
(modified after loading)

Area: 24169.5 17.216  
Area: 539.224 18.891  

Area: 13169.5 17.217  
Area: 251.401 18.908  

Area: 7430.03 17.217  
Area: 136.019 18.904  

Data File C:\HPCHEM\1\DATA\201308~1\JC10219B.D                                   Sample Name: JC10219B
Instrument 1 8/2/2014 12:11:18 AM MK
Area Percent Report

Signal 1: DAD1 A, Sig=250,10 Ref=360,100
Signal 2: DAD1 B, Sig=254,10 Ref=360,100
Signal 3: DAD1 C, Sig=210,10 Ref=360,100
Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  13.947 FM    0.4147 1.41912e4   570.28912  98.1507
2  17.393 MM    0.4723  267.37726    9.43601   1.8493

Totals :                  1.44586e4   579.72513

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100
Area Percent Report

Sorted By             :      Signal
Multiplier            :      1.0000
Dilution              :      1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100
Signal 2: DAD1 B, Sig=254,10 Ref=360,100
Signal 3: DAD1 C, Sig=210,10 Ref=360,100
Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  18.563 MF    0.6055  800.43811   22.03275   5.2285
2  20.658 FM    1.4329 1.45087e4   168.75829  94.7715

Totals :                  1.53092e4   190.79103

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

*** End of Report ***
**Injection Date**: 9/4/2013 1:38:06 PM  
**Seq. Line**: 11  
**Sample Name**: JC10275  
**Location**: Vial 9  
**Acq. Operator**: MK  
**Acq. Instrument**: Instrument 1  
**Inj Volume**: 15 µl  

**Different Inj Volume from Sequence!**  
**Actual Inj Volume**: 5 µl

**Acq. Method**: C:\HPCHEM\1\METHODS\JC-AD01A.M  
**Last changed**: 11/14/2012 10:39:31 PM by CE

**Analysis Method**: C:\HPCHEM\1\METHODS\AD005-40.M  
**Last changed**: 8/3/2014 12:34:42 AM by MK

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**Area Percent Report**

**Sorted By**: Signal  
**Multiplier**: 1.0000  
**Dilution**: 1.0000

**Signal 1**: DAD1 A, Sig=250,10 Ref=360,100  
**Signal 2**: DAD1 B, Sig=254,10 Ref=360,100  
**Signal 3**: DAD1 C, Sig=210,10 Ref=360,100  

**Peak RetTime Type**  
| # | RetTime | Width | Area       | Height | Area Percent |
|---|---------|-------|------------|--------|-------------|
| 1 | 16.630  | 0.3948| 3.00347e4  | 1267.95288 | 97.5011     |
| 2 | 17.710  | 0.3597| 769.77063  | 35.66695  | 2.4989      |

**Totals**: 3.08045e4 1303.61983

Results obtained with enhanced integrator!

**Signal 4**: DAD1 D, Sig=230,10 Ref=360,100  
**Signal 5**: DAD1 E, Sig=280,10 Ref=360,100

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***End of Report***
Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100
Signal 2: DAD1 B, Sig=254,10 Ref=360,100
Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type Width Area Height Area %
---|-------|----|-------|----------|----------|--------|
1  16.800 MM  0.3820 1447.42444 63.15571  3.0557
2  17.912 MM  0.5129 4.59204e4 1492.23425 96.9443

Totals: 4.73678e4 1555.38996

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

*** End of Report ***
Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100
Signal 2: DAD1 B, Sig=254,10 Ref=360,100
Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  13.897 MM    0.3634 9419.56641  432.00473  97.1339
2  16.371 MM    0.4602  277.94183   10.06597   2.8661

Totals: 9697.50824 442.07070

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100
Signal 5: DAD1 E, Sig=280,10 Ref=360,100

... End of Report...
**Injection Date**: 9/16/2013 1:15:30 PM  
**Seq. Line**: 8  
**Sample Name**: JC10279A  
**Location**: Vial 94  
**Acq. Operator**: MK  
**Acq. Instrument**: Instrument 1  
**Inj Volume**: 15 µl  
**Actual Inj Volume**: 1 µl  
**Acq. Method**: C:\HPCHEM\1\METHODS\AD-10-40.M  
**Last changed**: 7/12/2012 11:12:25 AM by CE  
**Analysis Method**: C:\HPCHEM\1\METHODS\AD005-40.M  
**Last changed**: 8/2/2014 12:24:51 AM by MK  

**Area Percent Report**

**Sorted By**: Signal  
**Multiplier**: 1.0000  
**Dilution**: 1.0000  

**Signal 1**: DAD1 A, Sig=250,10 Ref=360,100  
---|-------|----|-------|----------|----------|--------|
1  17.565 MM    0.4749  209.48149    7.35103   9.7971
2  23.873 MM    0.6716 1928.72815   47.86718  90.2029

**Totals**: 2138.20964   55.21821

**Results obtained with enhanced integrator!**

**Signal 2**: DAD1 B, Sig=254,10 Ref=360,100  
---|-------|----|-------|----------|----------|--------|
1  17.558 MM    0.4747  105.07636    3.68929   9.8035
2  23.875 MM    0.6715  966.74506   23.99374  90.1965

**Totals**: 1071.82142   27.68303

**Results obtained with enhanced integrator!**

**Signal 3**: DAD1 C, Sig=210,10 Ref=360,100  
---|-------|----|-------|----------|----------|--------|
1  17.567 MM    0.4652  922.10120   33.03711   9.4862
2  23.873 MM    0.6781 8798.38086  216.24049  90.5138

**Totals**: 9720.48206  249.27760

**Results obtained with enhanced integrator!**

**Signal 4**: DAD1 D, Sig=230,10 Ref=360,100  
---|-------|----|-------|----------|----------|--------|
1  17.571 MM    0.4736  593.79047   20.89495   9.7026
2  23.875 MM    0.6721 5526.11816  137.03973  90.2974

**Totals**: 6119.90863  157.93469

**Results obtained with enhanced integrator!**

**Signal 5**: DAD1 E, Sig=280,10 Ref=360,100  
---|-------|----|-------|----------|----------|--------|

**End of Report***

**Data File**: C:\HPCHEM\1\DATA\201309~1\JC10279A.D  
**Sample Name**: JC10279A  
**Instrument 1**: 8/2/2014 12:26:11 AM MK
Injection Date: 9/17/2013 5:04:56 AM  
Seq. Line:  24  
Sample Name: JC10281B  
Location: Vial 97  
Acq. Operator: MK  
Inj: 1  
Acq. Instrument: Instrument 1  
Inj Volume: 15 µl  
Different Inj Volume from Sequence!  
Actual Inj Volume: 6 µl  
Acq. Method: C:\HPCHEM\1\METHODS\AS-10-40.M  
Last changed: 9/3/2013 3:07:57 PM by MK  
Analysis Method: C:\HPCHEM\1\METHODS\OD-02-20.M  
Last changed: 7/30/2014 10:40:14 PM by MK  
(modified after loading)  

Area: 118.485  
18.705  
Area: 9716.74  
30.059  
Area: 127.457  
18.704  
Area: 10351.7  
30.058  
Area: 138.82  
18.710  
Area: 9845.16  
30.044  
Area: 42.5912  
18.681  
Area: 3120.39  
30.058  
Area: 25.9779  
18.705  
Area: 1970.17  
30.062  

Results obtained with enhanced integrator!  

Area Percent Report  

Sorted By: Signal  
Multiplier: 1.0000  
Dilution: 1.0000  
Use Multiplier & Dilution Factor with ISTDs  

Signal 1: DAD1 A, Sig=250,10 Ref=360,100  
Peak RetTime Type Width Area Height Area %  
# [min] [min] [mAU*s] [mAU]  
1 18.705 MM 0.6218 118.48459 3.17571 1.2047  
2 30.059 MM 1.1259 9716.74219 143.83633 98.7953  
Totals: 9835.22678 147.01204  

Signal 2: DAD1 B, Sig=254,10 Ref=360,100  
Peak RetTime Type Width Area Height Area %  
# [min] [min] [mAU*s] [mAU]  
1 18.704 MM 0.6262 127.45731 3.39228 1.2163  
2 30.058 MM 1.1261 1.03517e4 153.21146 98.7837  
Totals: 1.04792e4 156.60374  

Signal 3: DAD1 C, Sig=210,10 Ref=360,100  
Peak RetTime Type Width Area Height Area %  
# [min] [min] [mAU*s] [mAU]  
1 18.710 MM 0.6299 138.81970 3.67318 1.3904  
2 30.044 MM 1.1245 9845.16113 145.92497 98.6096  
Totals: 9983.98083 149.59815  

Signal 4: DAD1 D, Sig=230,10 Ref=360,100  
Peak RetTime Type Width Area Height Area %  
# [min] [min] [mAU*s] [mAU]  
1 18.681 MM 0.6396 42.59124 1.10979 1.3466  
2 30.058 MM 1.1261 3120.39331 46.18145 98.6534  
Totals: 3162.98455 47.29125  

Signal 5: DAD1 E, Sig=280,10 Ref=360,100  
Peak RetTime Type Width Area Height Area %  
# [min] [min] [mAU*s] [mAU]  
1 18.705 MM 0.6513 25.97787 6.64763e-1 1.3014  
2 30.062 MM 1.1250 1970.16589 29.18823 98.6986  
Totals: 1996.14376 29.85299  

Results obtained with enhanced integrator!  

*** End of Report ***
Injection Date: 9/17/2013 4:23:37 AM
Seq. Line: 23
Sample Name: JC10281A
Location: Vial 96
Acq. Operator: MK
Inj: 1
Acq. Instrument: Instrument 1
Inj Volume: 15 µl
Different Inj Volume from Sequence!
Actual Inj Volume: 3 µl
Acq. Method: C:\HPCHEM\1\METHODS\AS-10-40.M
Last changed: 9/3/2013 3:07:57 PM by MK
Analysis Method: C:\HPCHEM\1\METHODS\AD005-40.M
Last changed: 8/2/2014 12:27:30 AM by MK
(modified after loading)

Area Percent Report

Sorted By: Signal
Multiplier: 1.0000
Dilution: 1.0000
Use Multiplier & Dilution Factor with ISTDs

Signal 1: DAD1 A, Sig=250,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  18.481 MM    0.6395 1.06802e4   278.33743  97.4903
2  30.127 MM    1.1077  274.94009    4.13675   2.5097
Totals :                  1.09552e4   282.47418

Results obtained with enhanced integrator!

Signal 2: DAD1 B, Sig=254,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  18.481 MM    0.6401 1.13830e4   296.37387  97.5313
2  30.114 MM    1.0968  288.12051    4.37829   2.4687
Totals :                  1.16711e4   300.75216

Results obtained with enhanced integrator!

Signal 3: DAD1 C, Sig=210,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  18.479 MM    0.6415 1.07601e4   279.53766  97.2401
2  30.154 MM    1.0830  305.39581    4.69971   2.7599
Totals :                  1.10655e4   284.23737

Results obtained with enhanced integrator!

Signal 4: DAD1 D, Sig=230,10 Ref=360,100

Peak RetTime Type  Width     Area      Height     Area
#   [min]        [min]   [mAU*s]     [mAU]        %
----|-------|----|-------|----------|----------|--------|
1  18.482 MM    0.6382 3414.37500   89.16418  97.5752
2  30.009 MM    1.0444   84.84958    1.35400   2.4248
Totals :                  3499.22458   90.51818

Results obtained with enhanced integrator!

Signal 5: DAD1 E, Sig=280,10 Ref=360,100


--- End of Report ---
### Data File C:\HPCHEM\1\DATA\201402~1\JC11123.D

Sample Name: JC11123

### Instrument 1 7/30/2014 10:42:07 PM MK

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**Area Percent Report**

| Signal 1: DAD1 A, Sig=250,10 Ref=360,100 |
|------------------------------------------|
| Peak RetTime Type | Width | Area     | Height | Area |
| #   | [min] | [min]   | [mAU*s] | [mAU] | [%] |
| 1  | 13.030 | MF | 0.2116 | 1760.29651 | 138.68036 | 3.9208 |
| 2  | 22.909 | MM | 0.6103 | 4.31360e4 | 1177.93591 | 96.0792 |
| **Totals:** | | | 4.48962e4 | 1316.61627 |

Results obtained with enhanced integrator!

| Signal 4: DAD1 D, Sig=230,10 Ref=360,100 |
|------------------------------------------|
| Peak RetTime Type | Width | Area     | Height | Area |
| #   | [min] | [min]   | [mAU*s] | [mAU] | [%] |
| 1  | 13.030 | MF | 0.2092 | 107.29430 | 8.54649 | 2.9057 |
| 2  | 22.909 | MM | 0.4974 | 3585.25415 | 120.13020 | 97.0943 |
| **Totals:** | | | 3692.54845 | 128.67670 |

Results obtained with enhanced integrator!

| Signal 5: DAD1 E, Sig=280,10 Ref=360,100 |
|------------------------------------------|
| Peak RetTime Type | Width | Area     | Height | Area |
| #   | [min] | [min]   | [mAU*s] | [mAU] | [%] |
| 1  | 13.030 | MF | 0.25 | 107.29430 | 8.54649 | 2.9057 |
| 2  | 22.909 | MM | 0.4974 | 3585.25415 | 120.13020 | 97.0943 |
| **Totals:** | | | 3692.54845 | 128.67670 |

Results obtained with enhanced integrator!

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### Area Percent Report

| Signal 2: DAD1 B, Sig=254,10 Ref=360,100 |
|------------------------------------------|
| Peak RetTime Type | Width | Area     | Height | Area |
| #   | [min] | [min]   | [mAU*s] | [mAU] | [%] |
| 1  | 13.030 | MF | 0.2116 | 1760.29651 | 138.68036 | 3.9208 |
| 2  | 22.909 | MM | 0.6103 | 4.31360e4 | 1177.93591 | 96.0792 |
| **Totals:** | | | 4.48962e4 | 1316.61627 |

Results obtained with enhanced integrator!

| Signal 3: DAD1 C, Sig=210,10 Ref=360,100 |
|------------------------------------------|
| Peak RetTime Type | Width | Area     | Height | Area |
| #   | [min] | [min]   | [mAU*s] | [mAU] | [%] |
| 1  | 13.030 | MF | 0.2116 | 1760.29651 | 138.68036 | 3.9208 |
| 2  | 22.909 | MM | 0.6103 | 4.31360e4 | 1177.93591 | 96.0792 |
| **Totals:** | | | 4.48962e4 | 1316.61627 |

Results obtained with enhanced integrator!
### Area Percent Report

**Sorted By:** Signal  
**Multiplier:** 1.0000  
**Dilution:** 1.0000  

*Use Multiplier & Dilution Factor with ISTDs*

#### Signal 1: DAD1 A, Sig=250,10 Ref=360,100

| Peak RetTime | Type | Width | Area [mAU*s] | Height [mAU] | % |
|--------------|------|-------|--------------|--------------|---|
| 12.829       | MF   | 0.3098| 2.41232e4    | 1297.61914   | 96.1899 |

**Totals:** 2.50787e4 1340.11574

Results obtained with enhanced integrator!

#### Signal 2: DAD1 B, Sig=254,10 Ref=360,100

| Peak RetTime | Type | Width | Area [mAU*s] | Height [mAU] | % |
|--------------|------|-------|--------------|--------------|---|
| 23.784       | FM   | 0.3747| 955.53381    | 42.49660     | 3.8101 |

**Totals:** 1898.61647 122.66490

Results obtained with enhanced integrator!

#### Signal 3: DAD1 C, Sig=210,10 Ref=360,100

| Peak RetTime | Type | Width | Area [mAU*s] | Height [mAU] | % |
|--------------|------|-------|--------------|--------------|---|
| 12.830       | MF   | 0.2556| 1843.23779   | 120.16679    | 97.0832 |
| 23.795       | FM   | 0.3695| 55.37868     | 2.49811      | 2.9168 |

**Totals:** 1898.61647 122.66490

Results obtained with enhanced integrator!

#### Signal 4: DAD1 D, Sig=230,10 Ref=360,100

| Peak RetTime | Type | Width | Area [mAU*s] | Height [mAU] | % |
|--------------|------|-------|--------------|--------------|---|
| 12.829       | MF   | 0.2556| 1843.23779   | 120.16679    | 97.0832 |
| 23.795       | FM   | 0.3695| 55.37868     | 2.49811      | 2.9168 |

**Totals:** 1898.61647 122.66490

Results obtained with enhanced integrator!

#### Signal 5: DAD1 E, Sig=280,10 Ref=360,100

| Peak RetTime | Type | Width | Area [mAU*s] | Height [mAU] | % |
|--------------|------|-------|--------------|--------------|---|
| 12.830       | MF   | 0.2556| 1843.23779   | 120.16679    | 97.0832 |
| 23.795       | FM   | 0.3695| 55.37868     | 2.49811      | 2.9168 |

**Totals:** 1898.61647 122.66490

Results obtained with enhanced integrator!