

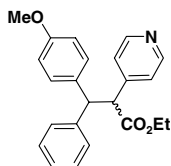
SUPPORTING INFORMATION

Nucleophilic Displacement at Benzhydryl Centers: Asymmetric Synthesis of 1,1-Diarylalkyl Derivatives

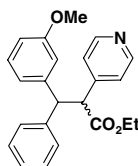
Yuri Bolshan,^{†§} Cheng-yi Chen,[‡] Jennifer R. Chilenski,[‡] Francis Gosselin,[†] David J. Mathre,[‡] Paul D. O'Shea,^{*†} Amélie Roy,[†] and Richard D. Tillyer[‡]

[†]Department of Process Research, Merck Frosst Centre for Therapeutic Research, P.O. Box 1005, Pointe Claire-Dorval, Québec, H9R 4P8, Canada and [‡]Department of Process Research, Merck Research laboratories, P.O. Box 2000, Rahway NJ 07065, USA.

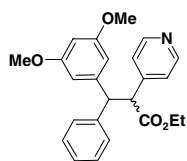
paul.oshea@merck.com



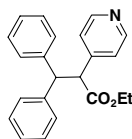
Ethyl 3-(4-methoxy-phenyl)-3-phenyl-2-pyridin-4-yl-propionate (5a): ¹H NMR (500 MHz, CDCl₃) δ 8.42 (m, 2H), 7.39 (d, 1H, *J* = 7.5 Hz), 7.32 (d, 1H, *J* = 8.6 Hz), 7.29 (t, 1H, *J* = 7.6 Hz), 7.23 (m, 2H), 7.10-6.99 (m, 3H), 6.95 (d, 1H, *J* = 7.6 Hz), 6.83 (d, 1H, *J* = 8.6 Hz), 6.62 (d, 1H, *J* = 8.6 Hz), 4.62 (d, 0.5H, *J* = 5.7 Hz), 4.59 (d, 0.5H, *J* = 5.6 Hz), 4.38 (d, 1H, *J* = 12.2 Hz), 3.99-3.89 (m, 2H), 3.74 (s, 1.5H), 3.64 (s, 1.5H), 1.01 (t, 1.5H, *J* = 7.1 Hz), 0.97 (t, 1.5H, *J* = 7.1 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 171.5, 158.4, 158.0, 149.8, 149.7, 146.2, 146.1, 142.3, 141.0, 134.1, 132.8, 129.8, 129.2, 129.0, 128.7, 128.6, 128.4, 127.9, 127.5, 126.8, 126.5, 123.8, 123.7, 114.0, 113.8, 61.1, 61.0, 56.6, 55.1, 55.0, 53.8, 53.7, 13.9 (br); LRMS calcd for C₂₃H₂₃NO₃: 361.17; Found: 361.9 [M+1].



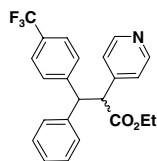
Ethyl 3-(3-methoxy-phenyl)-3-phenyl-2-pyridin-4-yl-propionate (5b): ¹H NMR (500 MHz, CDCl₃) δ 8.42 (app d, 1H, *J* = 5.9 Hz), 8.40 (app d, 1H, *J* = 5.9 Hz), 7.41 (d, 1H, *J* = 7.1 Hz), 7.29-7.25 (m, 2H), 7.22-7.15 (m, 2H), 7.05 (app d, 2H, *J* = 4.3 Hz), 7.02-6.96 (m, 2H), 6.72 (dd, 0.5H, *J*₁ = 2.0 Hz, *J*₂ = 8.1 Hz), 6.67 (d, 0.5H, *J* = 7.7 Hz), 6.62 (m, 0.5H), 6.53 (dd, 0.5H, *J*₁ = 2.1 Hz, *J*₂ = 8.2 Hz), 4.63 (dd, 1H, *J*₁ = 2.4 Hz, *J*₂ = 12.3 Hz), 4.43 (dd, 1H, *J*₁ = 5.9 Hz, *J*₂ = 12.3 Hz), 3.99-3.85 (m, 2H), 3.72 (s, 1.5), 3.59 (s, 1.5H), 0.99 (t, 1.5H, *J* = 7.1 Hz), 0.94 (t, 1.5H, *J* = 7.1 Hz); ¹³C NMR (125 MHz, CDCl₃) δ 171.2, 171.1, 159.5, 159.2, 149.6, 149.5, 145.82, 145.80, 143.4, 142.1, 141.7, 140.4, 129.4, 129.2, 128.4, 128.2, 127.9, 127.5, 126.7, 126.4, 123.6, 120.2, 119.7, 114.2, 113.6, 111.8, 111.2, 61.0, 60.9, 56.1, 54.9, 54.7, 54.4, 54.3, 13.6 (br); LRMS calcd for C₂₃H₂₃NO₃: 361.17; Found: 361.9 [M+1].



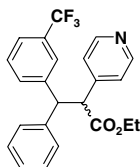
Ethyl 3-(3,5-dimethoxyphenyl)-3-phenyl-2-pyridin-4-yl-propionate (5d): ^1H NMR (500 MHz, CDCl_3) δ 8.44 (dd, 1H, $J_1 = 1.4$ Hz, $J_2 = 4.6$ Hz), 8.40 (dd, 1H, $J_1 = 1.4$ Hz, $J_2 = 4.6$ Hz), 7.41 (d, 1H, $J = 7.3$ Hz), 7.29 (m, 2H), 7.18 (m, 1.5H), 7.07-7.00 (m, 2.5H), 6.57 (d, 1H, $J = 2.1$ Hz), 6.30 (m, 0.5H), 6.22 (d, 1H, $J = 2.1$ Hz), 6.13 (m, 0.5H), 4.58 (dd, 1H, $J_1 = 4.1$ Hz, $J_2 = 12.3$ Hz), 4.39 (dd, 1H, $J_1 = 9.7$ Hz, $J_2 = 12.2$ Hz), 4.04-3.88 (m, 2H), 3.74 (s, 3H), 3.60 (s, 3H), 1.04 (t, 1.5H, $J = 7.1$ Hz), 0.96 (t, 1.5H, $J = 7.1$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 171.4, 171.3, 160.8, 160.5, 149.8, 149.7, 146.0, 145.9, 144.3, 142.9, 141.7, 140.5, 128.6, 128.4, 128.0, 127.6, 126.9, 126.6, 123.74, 123.73, 106.6, 105.9, 98.4, 97.9, 61.2, 61.0, 56.23, 56.21, 55.2, 55.0, 54.7, 54.6, 13.8 (br); LRMS calcd for $\text{C}_{24}\text{H}_{25}\text{NO}_4$: 391.18; Found: 392.1 [M+1].



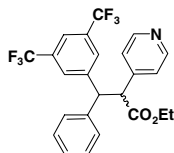
Ethyl 3,3-diphenyl-2-pyridin-4-yl-propionate (5e): ^1H NMR (500 MHz, CDCl_3) δ 8.41 (app d, 2H, $J = 5.9$ Hz), 7.41 (d, 2H, $J = 7.6$ Hz), 7.29 (t, 2H, $J = 7.5$ Hz), 7.23 (m, 2H), 7.19 (app t, 1H, $J = 7.4$ Hz), 7.09-7.05 (m, 4H), 7.03-6.98 (m, 1H), 4.65 (d, 1H, $J = 12.3$ Hz), 4.43 (d, 1H, $J = 12.3$ Hz), 3.99-3.88 (m, 2H), 0.97 (t, 3H, $J = 7.1$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 171.4, 149.7, 145.9, 141.9, 140.6, 128.6, 128.4, 128.0, 127.6, 126.8, 126.5, 123.7, 61.1, 56.4, 54.4, 13.7; LRMS calcd for $\text{C}_{22}\text{H}_{21}\text{NO}_2$: 331.16; Found: 332.0 [M+1].



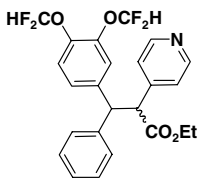
Ethyl 3-phenyl-2-pyridin-4-yl-3-(4-trifluoromethyl-phenyl)-propionate (5f): ^1H NMR (500 MHz, CDCl_3) δ 8.44 (m, 2H), 7.56 (m, 2H), 7.41-7.31 (m, 3H), 7.24 (m, 2.5H), 7.18 (d, 1H, $J = 8.2$ Hz), 7.12 (m, 1H), 7.05 (m, 1.5H), 4.73 (app t, 1H), 4.43 (d, 1H, $J = 12.3$ Hz), 4.02-3.90 (m, 2H), 1.00 (m, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 171.0, 170.9, 149.9, 149.7, 146.1, 145.4, 145.3, 144.8, 140.9, 139.6, 128.7, 128.5, 128.3, 127.9, 127.5, 127.1, 126.8, 125.4 (q, $J = 4$ Hz), 125.3 (q, $J = 4$ Hz), 123.9 (q, $J = 272$ Hz), 123.7 (q, $J = 272$ Hz), 123.5, 123.4, 61.2, 61.1, 55.92, 55.88, 54.2, 13.6 (br); LRMS calcd for $\text{C}_{23}\text{H}_{20}\text{F}_3\text{NO}_2$: 399.14; Found: 400.0 [M+1].



Ethyl 3-phenyl-2-pyridin-4-yl-3-(3-trifluoromethyl-phenyl)-propionate (5g): ^1H NMR (500 MHz, CDCl_3) δ 8.44 (m, 2H), 7.75 (s, 0.5H), 7.60 (d, 0.5H, $J = 7.8$ Hz), 7.44 (m, 1.5H), 7.37 (m, 1H), 7.31-7.26 (m, 4H), 7.21-7.14 (m, 1H), 7.07 (m, 2H), 7.01 (m, 0.5H), 4.79 (d, 0.5H, $J = 15.4$ Hz), 4.77 (d, 0.5H, $J = 15.4$ Hz), 4.51 (d, 1H, $J = 12.2$ Hz), 4.00-3.86 (m, 2H), 0.95 (m, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 171.2, 171.0, 149.9, 149.8, 145.6, 143.1, 141.9, 141.0, 139.8, 131.5, 131.4, 130.8 (q, $J = 32$ Hz), 130.6 (q, $J = 32$ Hz), 129.1, 128.9, 128.8, 128.6, 128.0, 127.6, 127.2, 126.9, 124.8 (q, $J = 4$ Hz), 124.1 (q, $J = 4$ Hz), 123.7 (q, $J = 4$ Hz), 124.0 (q, $J = 272$ Hz), 123.8 (q, $J = 273$ Hz), 123.8-123.6 (m), 61.3, 61.2, 56.2, 56.1, 54.37, 54.36, 13.6 (br); LRMS calcd for $\text{C}_{23}\text{H}_{20}\text{F}_3\text{NO}_2$: 399.14; Found: 400.0 [M+1].

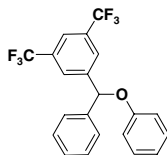


Ethyl 3-(3,5-bis-trifluoromethyl-phenyl)-3-phenyl-2-pyridin-4-yl-propionate (5i): ^1H NMR (500 MHz, CDCl_3) δ 8.48 (m, 2H), 7.95 (s, 1H), 7.78 (s, 0.5H), 7.60 (s, 1.5H), 7.46 (d, 1H, $J = 7.3$ Hz), 7.35-7.23 (m, 4H), 7.14-7.04 (m, 2H), 4.94 (d, 0.5H, $J = 12.3$ Hz), 4.87 (d, 0.5 H, $J = 12.2$ Hz), 4.57 (m, 1H), 4.06-3.91 (m, 2H), 1.00 (m, 3H); ^{13}C NMR (125 MHz, CDCl_3) δ 171.0, 170.6, 150.2, 149.9, 145.0, 144.9, 143.6, 140.0, 138.8, 131.7 (app quint, $J = 33$ Hz), 129.0, 128.8, 128.3 (br), 128.0, 127.8 (br), 127.62, 127.59, 127.3, 123.6, 123.4, 123.1 (q, $J = 273$ Hz), 122.9 (q, $J = 273$ Hz), 120.9 (m), 120.6 (m), 61.5, 61.4, 55.9, 55.8, 54.2, 13.5 (br); LRMS calcd for $\text{C}_{24}\text{H}_{19}\text{F}_6\text{NO}_2$: 467.13; Found: 468.1 [M+1].

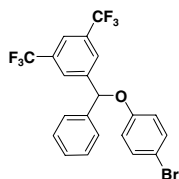


Ethyl 3-(3,4-bis-difluoromethoxy-phenyl)-3-phenyl-2-pyridin-4-yl-propionate (5k): ^1H NMR (500 MHz, CDCl_3) δ 8.45 (d, 1H, $J = 5.9$ Hz), 8.41 (d, 1H, $J = 5.9$ Hz), 7.39 (d, 1H, $J = 7.5$ Hz), 7.36 (s, 0.5H), 7.33-7.27 (m, 1.5H), 7.25-7.18 (m, 3H), 7.11-6.94 (m, 4H), 6.52 (t, 0.5H, $J = 73.6$ Hz), 6.48 (t, 0.5H, $J = 73.5$ Hz), 6.36 (t, 0.5H, $J = 73.5$ Hz), 6.31 (t, 0.5H, $J = 73.6$ Hz), 4.67 (m, 1H), 4.39 (d, 1H, $J = 12.3$ Hz), 4.02-3.90 (m, 2H), 1.01 (t, 1.5H, $J = 7.1$ Hz), 0.96 (t, 1.5H, $J = 7.1$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 171.1, 170.8, 149.9, 149.7, 145.4, 145.3, 142.0 (m), 141.7 (m), 141.2, 141.0 (m), 140.8, 140.7 (m), 139.8, 139.6, 128.7, 128.6, 127.8, 127.4, 127.2, 126.9, 126.2, 125.8, 123.5, 122.20, 122.18, 121.9, 121.6, 115.70 (t, $J = 262$ Hz), 115.68 (t, $J = 262$ Hz), 115.6 (t, $J =$

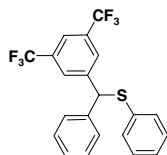
262 Hz), 115.5 (t, $J = 262$ Hz), 61.3, 61.2, 56.1, 56.0, 53.8, 53.6, 13.5, 13.4; LRMS calcd for $C_{24}H_{21}F_4NO_4$: 463.14; Found: 464.1 [M+1].



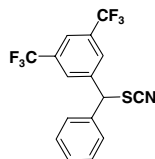
1-(Phenoxy-phenyl-methyl)-3,5-bis-trifluoromethyl-benzene (7a): 1H NMR (500 MHz, $CDCl_3$) δ 7.90 (s, 2H), 7.81 (s, 1H), 7.42-7.37 (m, 4H), 7.34-7.31 (m, 1H), 7.28-7.23 (m, 2H), 6.97-6.94 (m, 3H), 6.28 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 157.3, 144.2, 139.5, 131.8 (q, $J = 33$ Hz), 129.5, 129.1, 128.5, 126.8 (m), 126.7, 123.0 (q, $J = 273$ Hz), 121.8, 121.7 (m), 116.1, 80.6; LRMS calcd for $C_{21}H_{14}F_6O$: 396.09; Found: 395.0 [M-1].



1-[(4-Bromo-phenoxy)-phenyl-methyl]-3,5-bis-trifluoromethyl-benzene (7b): 1H NMR (500 MHz, $CDCl_3$) δ 7.87 (s, 2H), 7.81 (s, 1H), 7.38 (m, 4H), 7.33 (m, 3H), 6.84 (m, 2H), 6.23 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 156.3, 143.7, 139.0, 132.5, 132.0 (q, $J = 34$ Hz), 129.2, 128.8, 126.7 (m), 124.2, 123.1 (q, $J = 273$ Hz), 121.9 (app quint), 118.0, 114.2, 81.0; LRMS calcd for $C_{21}H_{13}BrF_6O$: 474.01; Found: 473.0 [M-1].

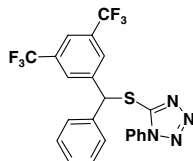


1-(Phenyl-phenylsulfanyl-methyl)-3,5-bis-trifluoromethyl-benzene (7c): 1H NMR (500 MHz, $CDCl_3$) δ 7.79 (s, 2H), 7.72 (s, 1H), 7.40-7.34 (m, 4H), 7.31-7.27 (m, 1H), 7.26-7.23 (m, 2H), 7.22-7.19 (m, 3H), 5.57 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 143.9, 139.0, 134.1, 132.1, 131.7 (q, $J = 33$ Hz), 129.1, 129.0, 128.6 (m), 128.2, 128.0, 127.8, 123.2 (q, $J = 273$ Hz), 121.2 (app quint), 57.4; LRMS calcd for $C_{21}H_{14}F_6S$: 412.07; Found: 411.1 [M-1].

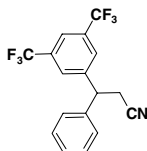


1-(Phenyl-thiocyanato-methyl)-3,5-bis-trifluoromethyl-benzene (7d): 1H NMR (500 MHz, $CDCl_3$) δ 7.91 (s, 2H), 7.89 (s, 1H), 7.46-7.39 (m, 3H), 7.37-7.34 (m, 2H), 5.91 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 140.4, 135.5, 132.6 (q, $J = 34$ Hz), 129.6, 128.4 (m),

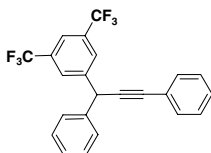
128.0, 122.8 (q, $J = 273$ Hz), 122.9 (app quint), 56.1; LRMS calcd for $C_{16}H_9F_6NS$: 361.04; Found: 359.9 [M-1].



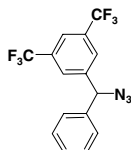
5-[(3,5-Bis-trifluoromethyl-phenyl)-phenyl-methylsulfanyl]-1-phenyl-1H-tetrazole (7e): 1H NMR (500 MHz, $CDCl_3$) δ 8.01 (s, 2H), 7.84 (s, 1H), 7.62-7.58 (m, 3H), 7.55-7.53 (m, 2H), 7.43-7.36 (s, 5H), 6.57 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 152.2, 142.2, 137.2, 133.2, 132.0 (q, $J = 33$ Hz), 130.4, 129.8, 129.2, 128.8, 128.4, 128.3, 128.1, 123.9, 123.0 (q, $J = 273$ Hz), 122.0 (m), 55.4; LRMS calcd for $C_{22}H_{14}F_6N_4S$: 480.08; Found: 451.0 [M-N₂-1].



3-(3,5-Bis-trifluoromethyl-phenyl)-3-phenyl-propionitrile (7f): 1H NMR (500 MHz, $CDCl_3$) δ 7.87 (s, 2H), 7.83 (s, 1H), 7.43-7.36 (m, 5H), 3.50 (s, 1H), 3.33 (d, 1H, $J = 16.7$ Hz), 3.23 (d, 1H, $J = 16.7$ Hz); ^{13}C NMR (125 MHz, $CDCl_3$) δ 146.7, 142.2, 131.9 (q, $J = 33$ Hz), 129.2, 129.1, 126.1 (br), 125.6, 121.4 (q, $J = 273$ Hz), 122.1 (m), 116.4, 75.9, 32.3; LRMS calcd for $C_{17}H_{11}F_6N$: 343.08; Found: 344.0 [M+1].

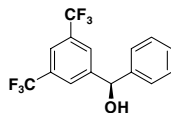


1-(1,3-Diphenyl-prop-2-ynyl)-3,5-bis-trifluoromethyl-benzene (7g): 1H NMR (500 MHz, $CDCl_3$) δ 8.15 (s, 2H), 7.79 (s, 1H), 7.67 (app d, 2H, $J = 7.8$ Hz), 7.50 (app d, 2H, $J = 7.2$ Hz), 7.38-7.29 (m, 6H), 3.20 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 147.8, 143.6, 131.8, 131.60 (q, $J = 33$ Hz), 129.2, 128.8, 128.6, 128.5, 126.30, 126.27, 126.0, 123.3 (q, $J = 273$ Hz), 121.7 (quint, $J = 4$ Hz), 121.6, 90.0, 88.6, 74.2; LRMS calcd for $C_{23}H_{14}F_6$: 404.10; Found: 403.1 [M-1].

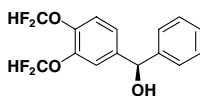


1-(Azido-phenyl-methyl)-3,5-bis-trifluoromethyl-benzene (7h): 1H NMR (500 MHz, $CDCl_3$) δ 7.82 (s, 1H), 7.80 (s, 2H), 7.44-7.36 (m, 3H), 7.29 (m, 2H), 5.81 (s, 1H); ^{13}C NMR (125 MHz, $CDCl_3$) δ 142.5, 137.9, 132.0 (q, $J = 34$ Hz), 129.3, 129.1, 127.6, 127.2

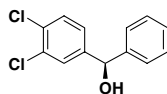
(m), 123.1 (q, $J = 273$ Hz), 121.9 (app quint), 67.5; LRMS calcd for $C_{15}H_9F_6N_3$: 345.07; Found: 317.9 [M-N₂].



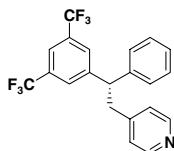
(R)-(3,5-Bis-trifluoromethyl-phenyl)-phenyl-methanol (8a): ¹H NMR (500 MHz, CDCl₃) δ 7.87 (s, 2H), 7.78 (s, 1H), 7.41-7.33 (m, 5H), 5.94 (d, 1H, $J = 3.1$ Hz), 2.37 (d, 1H, $J = 3.3$ Hz); ¹³C NMR (125 MHz, CDCl₃) δ 146.0, 142.3, 131.6 (q, $J = 33$ Hz), 129.1, 128.6, 126.7, 126.4, 123.3 (q, $J = 273$ Hz), 121.4 (quint, $J = 4$ Hz), 75.3; LRMS calcd for $C_{15}H_{10}F_6O$: 320.06; Found: 303.2 [M-OH]; IR (neat, cm⁻¹) 3331, 1455, 1378, 1339, 1278, 1166, 1119, 1042, 899, 849, 730, 699; $[\alpha]_D^{20} -32.5$ (MeOH, $c = 9.5$).



(R)-(3,4-Bis-difluoromethoxy-phenyl)-phenyl-methanol (8b): ¹H NMR (500 MHz, CDCl₃) δ 7.34-7.25 (m, 6H), 7.40-7.29 (m, 6H), 7.17-7.12 (m, 2H), 6.48 (t, 1H, $J = 73.6$ Hz), 6.46 (t, 1H, $J = 73.6$ Hz), 5.69 (s, 1H), 2.70 (s, 1H); ¹³C NMR (125 MHz, CDCl₃) δ 142.9, 142.7, 142.2 (t, $J = 3$ Hz), 142.3 (t, $J = 3$ Hz), 128.7, 128.1, 126.5, 124.4, 122.2, 120.1, 117.9, 115.8 (t, $J = 262$ Hz), 115.8 (t, $J = 262$ Hz); LRMS calcd for $C_{15}H_{12}F_4O_3$: 316.07; Found: 299.1 [M-OH]; IR (neat, cm⁻¹) 3339, 3069, 3034, 2992, 1509, 1392, 1270, 1208, 1119, 1096, 1042, 741; $[\alpha]_D^{20} -22.8$ (MeOH, $c = 13.9$).

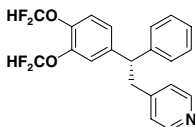


(R)-(3,4-Dichloro-phenyl)-phenyl-methanol (8c): ¹H NMR (500 MHz, CDCl₃) δ 7.51 (d, 1H, $J_1 = 1.9$ Hz), 7.40-7.29 (m, 6H), 7.20 (ddm, 1H, $J_1 = 2.1$ Hz, $J_2 = 8.3$ Hz), 5.79 (d, 1H, $J = 3.3$ Hz), 2.26 (d, 1H, $J = 3.4$ Hz); ¹³C NMR (125 MHz, CDCl₃) δ 143.8, 142.8, 132.5, 131.3, 130.3, 128.7, 128.3, 128.1, 126.5, 125.7, 75.5; LRMS calcd for $C_{13}H_{10}Cl_2O$: 252.01; Found: 235.0 [M-OH]; IR (neat, cm⁻¹) 3323, 3065, 3030, 2880, 1563, 1494, 1471, 1390, 1185, 1131, 1027, 887.8, 806.7, 699; $[\alpha]_D^{20} -33.3$ (MeOH, $c = 11.5$).

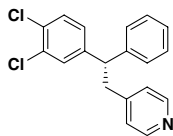


(2S)-4-[2-(3,5-Bis-trifluoromethyl-phenyl)-2-phenyl-ethyl]-pyridine (10a): ¹H NMR (500 MHz, CDCl₃) δ 8.42 (dd, 2H, $J_1 = 1.6$ Hz, $J_2 = 4.4$ Hz), 7.72 (s, 1H), 7.63 (s, 2H), 7.31 (app t, 2H, $J = 7.1$ Hz), 7.25 (m, 1H), 7.16 (m, 2H), 6.91 (m, 2H), 4.36 (t, 1H, $J = 8.0$ Hz), 3.42 (dd, 1H, $J_1 = 8.4$ Hz, $J_2 = 13.5$ Hz), 3.34 (dd, 1H, $J_1 = 7.5$ Hz, $J_2 = 13.8$

Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 149.7, 147.8, 146.2, 141.2, 131.7 (q, $J = 33$ Hz), 128.9, 127.9 (br s), 127.7, 127.3, 124.2, 123.1 (q, $J = 273$ Hz), 120.6 (quint, $J = 4$ Hz), 51.7, 40.9; LRMS calcd for $\text{C}_{21}\text{H}_{15}\text{F}_6\text{N}$: 395.11; Found: 396.0 $[\text{M}+1]$; IR (neat, cm^{-1}) 3069, 3030, 1602, 1374, 1278, 1170, 1127, 896, 703; $[\alpha]_{\text{D}}^{20} -5.6$ (MeOH, $c = 9.9$).



(2S)-4-[2-(3,4-Bis-difluoromethoxy-phenyl)-2-phenyl-ethyl]-pyridine (10b): ^1H NMR (500 MHz, CDCl_3) δ 8.38 (dd, 2H, $J_1 = 1.5$ Hz, $J_2 = 4.5$ Hz), 7.26 (t, 2H, $J = 7.4$ Hz), 7.20-7.16 (m, 3H), 7.12- 7.08 (m, 2H), 7.03 (dd, 1H, $J_1 = 2.1$ Hz, $J_2 = 8.4$ Hz) 6.91 (d, 2H, $J = 6.0$ Hz), 6.45 (t, 1H, $J = 73.6$ Hz), 6.43 (t, 1H, $J = 73.7$ Hz), 4.22 (t, 1H, $J = 8.0$ Hz), 3.33 (dd, 1H, $J_1 = 13.7$ Hz, $J_2 = 7.9$ Hz), 3.27 (dd, 1H, $J_1 = 13.7$ Hz, $J_2 = 8.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 149.4, 148.3, 142.6, 142.2, 141.9, 140.6, 128.6, 127.6, 126.9, 125.9, 124.2, 122.1, 121.8, 115.69 (t, $J = 262$ Hz), 115.67 (t, $J = 262$ Hz); LRMS calcd for $\text{C}_{21}\text{H}_{17}\text{F}_4\text{NO}_2$: 391.12; Found: 392.0 $[\text{M}+1]$; IR (neat, cm^{-1}) 3065, 3030, 1602, 1509, 1382, 1270, 1119, 1100, 1042, 730, 703; $[\alpha]_{\text{D}}^{20} -7.0$ (MeOH, $c = 8.7$).



(2S)-4-[2-(3,4-Dichloro-phenyl)-2-phenyl-ethyl]-pyridine (10c): ^1H NMR (500 MHz, CDCl_3) δ 8.41 (dd, 2H, $J_1 = 1.5$ Hz, $J_2 = 4.5$ Hz), 7.33-7.27 (m, 4H), 7.24-7.20 (tm, 1H, $J_1 = 7.3$ Hz), 7.15 (m, 2H), 7.01 (dd, 1H, $J_1 = 2.1$ Hz, $J_2 = 8.3$ Hz), 6.92 (m, 2H), 4.19 (t, 1H, $J = 7.9$ Hz), 3.34 (dd, 1H, $J_1 = 13.7$ Hz, $J_2 = 7.9$ Hz), 3.29 (dd, 1H, $J_1 = 13.7$ Hz, $J_2 = 8.0$ Hz); ^{13}C NMR (125 MHz, CDCl_3) δ 149.4, 148.1, 143.7, 142.0, 132.2, 130.2, 129.5, 128.5, 127.5, 127.1, 126.8, 124.1, 50.9, 40.7; LRMS calcd for $\text{C}_{19}\text{H}_{15}\text{Cl}_2\text{N}$: 327.06; Found: 328.0 $[\text{M}+1]$; IR (neat, cm^{-1}) 3065, 3030, 2945, 1602, 1471, 1135, 1031, 803; $[\alpha]_{\text{D}}^{20} -18.9$ (MeOH, $c = 8.9$).

X-Ray Crystal Structure of Compound **8c**.

Crystals of **8c** were grown from cyclohexane. A single crystal was selected for single crystal x-ray data collection on a Bruker Smart Apex system. The crystal was a thin rod with dimensions of 0.38 mm x 0.10 mm x 0.02 mm. The unit cell was collected on 30 second scan rate and indexing gave the cell setting to be orthorhombic. A full quadrant of data at a 30 second scan rate was collected. The structure was solved and refined in the orthorhombic P212121 space group using Bruker Saint+, Shelxl-97, and Bruker ShelXTL software. See the following Tables for tabulated information pertaining to the final specifications of the solved structure. An ORTEP plot (at 50% probability level) and a view showing the hydrogen bonding network in the crystal shown in the following figures.

Table 1. Crystal data and structure refinement for Compound **8c**

Identification code	jrc0573m	
Empirical formula	C13 H10 Cl2 O	
Formula weight	253.11	
Temperature	298(2) K	
wavelength	0.71073 Å	
Crystal system, space group	Orthorhombic,	P2(1)2(1)2(1)
unit cell dimensions	a = 4.841(5) Å	alpha = 90 deg.
	b = 11.380(11) Å	beta = 90 deg.
	c = 21.65(2) Å	gamma = 90 deg.
Volume	1193(2) Å ³	
Z, calculated density	4,	1.409 Mg/m ³
Absorption coefficient	0.518 mm ⁻¹	
F(000)	520	
Crystal size	0.38 x 0.10 x 0.02 mm	
Theta range for data collection	1.88 to 23.25 deg.	
Limiting indices	-5<=h<=5	
	-12<=k<=12	
	-24<=l<=24	
Reflections collected / unique	9703 / 1723	[R(int) = 0.1184]
Completeness to theta = 23.25	100.0 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	1723 / 0 / 145	
Goodness-of-fit on F ²	1.107	
Final R indices [I>2sigma(I)]	R1 = 0.0662,	WR2 = 0.1317
R indices (all data)	R1 = 0.0864,	WR2 = 0.1402
Absolute structure parameter	0.11(15)	
Largest diff. peak and hole	0.295 and -0.211 e.Å ⁻³	

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³) for Compound **8c**. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
C(1)	4545(9)	7561(4)	5924(2)	37(1)
C(2)	2349(10)	6836(5)	6236(2)	41(1)
C(3)	1321(13)	5837(5)	5967(3)	59(2)
C(4)	-618(13)	5140(5)	6260(4)	75(2)
C(5)	-1542(16)	5421(6)	6831(4)	75(2)
C(6)	-534(14)	6393(6)	7110(3)	71(2)
C(7)	1425(12)	7118(5)	6822(3)	54(2)
C(8)	4279(10)	8879(4)	6054(2)	38(1)
C(9)	2574(11)	9575(5)	5708(2)	44(1)
C(10)	2382(10)	10766(5)	5814(3)	51(2)
C(11)	3891(14)	11251(5)	6267(3)	56(2)
C(12)	5598(13)	10596(6)	6618(3)	66(2)
C(13)	5822(11)	9402(5)	6517(2)	53(2)
Cl(1)	191(3)	11583(1)	5359(1)	79(1)

c1(2)	3722(5)	12752(1)	6411(1)	95(1)
o(1)	4394(6)	7344(3)	5286(1)	47(1)

Table 3. Bond lengths [Å] and angles [deg] for Compound 8c.

c(1)-o(1)	1.405(5)
c(1)-c(2)	1.506(7)
c(1)-c(8)	1.532(7)
c(1)-H(1A)	0.9800
c(2)-c(3)	1.371(7)
c(2)-c(7)	1.383(7)
c(3)-c(4)	1.382(8)
c(3)-H(3)	0.9300
c(4)-c(5)	1.352(9)
c(4)-H(4)	0.9300
c(5)-c(6)	1.352(9)
c(5)-H(5)	0.9300
c(6)-c(7)	1.403(8)
c(6)-H(6)	0.9300
c(7)-H(7)	0.9300
c(8)-c(9)	1.367(7)
c(8)-c(13)	1.384(7)
c(9)-c(10)	1.378(8)
c(9)-H(9)	0.9300
c(10)-c(11)	1.341(8)
c(10)-c1(1)	1.722(6)
c(11)-c(12)	1.347(8)
c(11)-c1(2)	1.738(6)
c(12)-c(13)	1.381(8)
c(12)-H(12)	0.9300
c(13)-H(13)	0.9300
o(1)-H(1)	0.8200
o(1)-c(1)-c(2)	108.0(4)
o(1)-c(1)-c(8)	110.4(4)
c(2)-c(1)-c(8)	113.2(4)
o(1)-c(1)-H(1A)	108.4
c(2)-c(1)-H(1A)	108.4
c(8)-c(1)-H(1A)	108.4
c(3)-c(2)-c(7)	117.7(5)
c(3)-c(2)-c(1)	121.3(5)
c(7)-c(2)-c(1)	120.9(5)
c(2)-c(3)-c(4)	121.8(6)
c(2)-c(3)-H(3)	119.1
c(4)-c(3)-H(3)	119.1
c(5)-c(4)-c(3)	120.5(7)
c(5)-c(4)-H(4)	119.7
c(3)-c(4)-H(4)	119.7
c(6)-c(5)-c(4)	118.9(7)
c(6)-c(5)-H(5)	120.6
c(4)-c(5)-H(5)	120.6
c(5)-c(6)-c(7)	121.7(6)
c(5)-c(6)-H(6)	119.1
c(7)-c(6)-H(6)	119.1
c(2)-c(7)-c(6)	119.3(6)
c(2)-c(7)-H(7)	120.3
c(6)-c(7)-H(7)	120.3
c(9)-c(8)-c(13)	118.3(5)
c(9)-c(8)-c(1)	121.1(5)
c(13)-c(8)-c(1)	120.6(5)
c(8)-c(9)-c(10)	121.2(5)
c(8)-c(9)-H(9)	119.4
c(10)-c(9)-H(9)	119.4
c(11)-c(10)-c(9)	119.4(5)
c(11)-c(10)-c1(1)	122.2(5)
c(9)-c(10)-c1(1)	118.5(5)
c(10)-c(11)-c(12)	121.2(6)
c(10)-c(11)-c1(2)	120.6(5)
c(12)-c(11)-c1(2)	118.2(5)
c(11)-c(12)-c(13)	120.2(6)

C(11)-C(12)-H(12)	119.9
C(13)-C(12)-H(12)	119.9
C(12)-C(13)-C(8)	119.7(6)
C(12)-C(13)-H(13)	120.2
C(8)-C(13)-H(13)	120.2
C(1)-O(1)-H(1)	109.5

Symmetry transformations used to generate equivalent atoms:

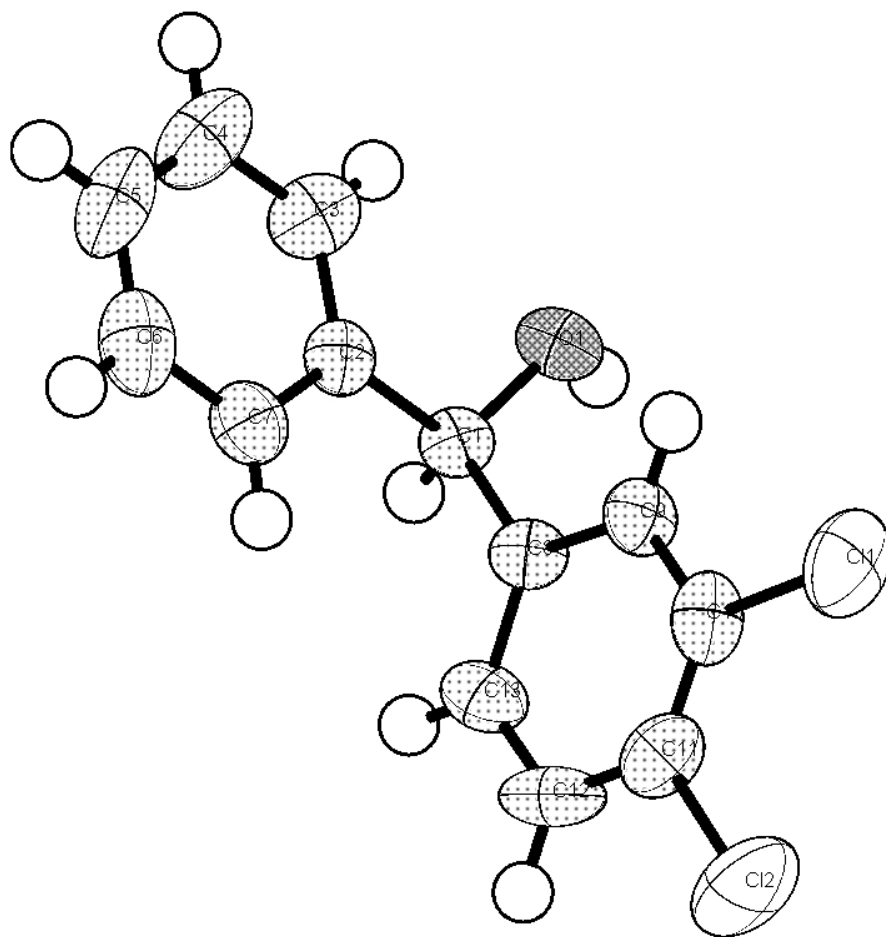
Table 4. Anisotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for compound 8c. The anisotropic displacement factor exponent takes the form: $-2 \pi^2 [h^2 a^{*2} u_{11} + \dots + 2 h k a^* b^* u_{12}]$

	U11	U22	U33	U23	U13	U12
C(1)	20(2)	42(3)	48(3)	-1(2)	0(2)	7(2)
C(2)	28(3)	48(4)	47(3)	6(3)	-2(3)	6(3)
C(3)	45(3)	53(4)	79(4)	-3(3)	2(3)	6(3)
C(4)	55(4)	51(4)	119(6)	13(4)	11(4)	2(3)
C(5)	64(4)	60(5)	102(6)	31(4)	14(4)	3(4)
C(6)	56(4)	91(5)	64(4)	24(4)	7(4)	8(4)
C(7)	51(3)	60(4)	52(4)	4(3)	-5(3)	6(3)
C(8)	19(3)	47(3)	48(3)	-5(3)	4(3)	5(2)
C(9)	41(3)	43(3)	50(3)	3(3)	-6(3)	11(3)
C(10)	33(3)	59(4)	60(4)	16(3)	5(3)	14(3)
C(11)	56(4)	40(3)	71(4)	-1(3)	7(4)	5(3)
C(12)	57(4)	70(4)	71(4)	-31(3)	-4(4)	-18(4)
C(13)	38(3)	68(4)	53(3)	-5(3)	-11(3)	-2(3)
C1(1)	73(1)	60(1)	105(1)	25(1)	-5(1)	15(1)
C1(2)	122(2)	44(1)	118(2)	-10(1)	15(1)	-6(1)
O(1)	31(2)	63(2)	46(2)	-12(2)	2(2)	5(2)

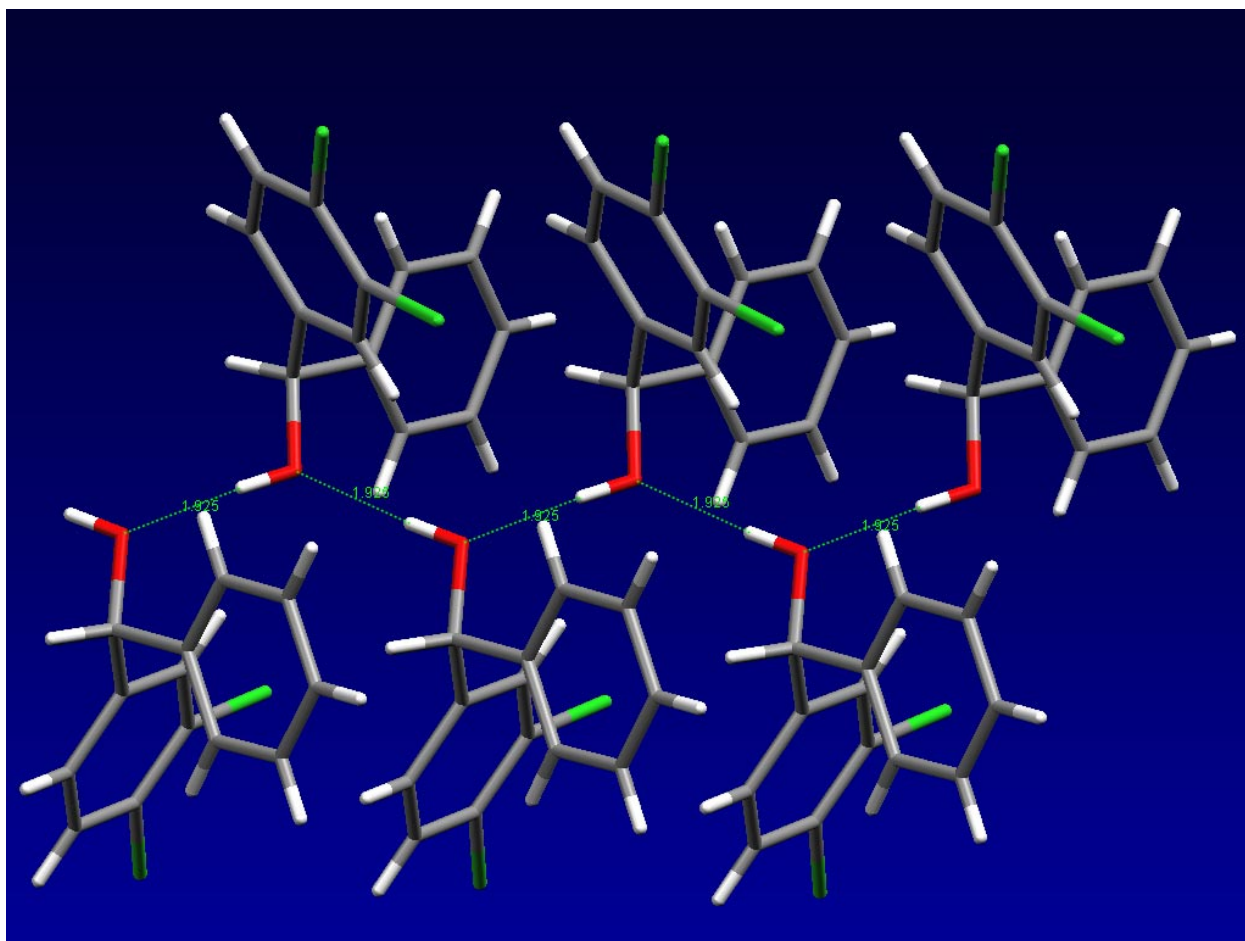
Table 5. Hydrogen coordinates ($\times 10^4$) and isotropic displacement parameters ($\text{\AA}^2 \times 10^3$) for compound 8c.

	x	y	z	U(eq)
H(1A)	6354	7296	6072	44
H(3)	1945	5622	5577	71
H(4)	-1292	4473	6063	90
H(5)	-2849	4954	7027	90
H(6)	-1156	6586	7504	85
H(7)	2095	7781	7024	65
H(9)	1526	9238	5394	53
H(12)	6629	10949	6929	79
H(13)	7003	8951	6759	64
H(1)	5927	7428	5132	70

ORTEP Plot of Compound 8c (50% probability level)



Hydrogen bonding network of Compound 8c.



View generated using Mercury (Mercury 1.1.2; Copyright CDCC 2001-2002).

X-Ray Crystal Structure of Compound 10c.

Crystals of **10c** were grown from ethanol/heptane. A single crystal was selected for single crystal x-ray data collection on a Bruker Smart Apex system. The crystal was a colorless plate with dimensions of 0.50 mm x 0.30 mm x 0.10 mm. The unit cell was collected on 10 second scan rate and indexing gave the cell setting to be monoclinic. A full quadrant of data at a 10 second scan rate was collected. The structure was solved and refined in the monoclinic C2 space group using Bruker Saint, Shelxl-97, and Bruker ShelXTL software. See the following Tables for tabulated information pertaining to the final specifications of the solved structure. An ORTEP plot (at 50% probability level) and a view showing the hydrogen bonding network in the crystal shown in the following figures.

Table 1. Crystal data and structure refinement for Compound 10c.

Identification code	Compound 10c	
Empirical formula	C ₁₉ H ₁₇ Cl ₂ N O ₄ S	
Formula weight	426.30	
Temperature	298(2) K	
wavelength	0.71073 Å	
Crystal system, space group	Monoclinic,	C2
unit cell dimensions	a = 16.453(4) Å	alpha = 90 deg.
	b = 8.310(2) Å	beta = 105.806(4)
	c = 29.604(7) Å	gamma = 90 deg.
Volume	3894.4(16) Å ³	
Z, Calculated density	8,	1.454 Mg/m ³
Absorption coefficient	0.466 mm ⁻¹	
F(000)	1760	
Crystal size	0.50 x 0.30 x 0.10 mm	
Theta range for data collection	1.43 to 24.71 deg.	
Limiting indices	-19<=h<=19	
	-9<=k<=9	
	-34<=l<=34	
Reflections collected / unique	18248 / 6631	[R(int) = 0.0624]
Completeness to theta = 24.71	99.9 %	
Absorption correction	None	
Refinement method	Full-matrix least-squares on F ²	
Data / restraints / parameters	6631 / 1 / 487	
Goodness-of-fit on F ²	1.023	
Final R indices [I>2sigma(I)]	R1 = 0.0490,	WR2 = 0.1229
R indices (all data)	R1 = 0.0543,	WR2 = 0.1270
Absolute structure parameter	0.10(5)	
Largest diff. peak and hole	0.309 and -0.408 e.Å ⁻³	

Table 2. Atomic coordinates (x 10⁴) and equivalent isotropic displacement parameters (Å² x 10³) for Compound 10c. U(eq) is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U(eq)
s(1)	839(1)	5022(1)	2106(1)	61(1)
s(21)	9077(1)	3803(1)	2807(1)	61(1)
o(1)	737(2)	5791(4)	2537(1)	82(1)
o(21)	9141(2)	3118(4)	2360(1)	91(1)
o(22)	8538(2)	5184(3)	2743(1)	72(1)
o(2)	1380(1)	3666(3)	2219(1)	69(1)
o(23)	8827(2)	2596(4)	3082(1)	107(1)
o(3)	-31(2)	4479(4)	1839(1)	88(1)
o(24)	9963(2)	4280(4)	3064(1)	84(1)
o(4)	1117(2)	6154(4)	1822(1)	97(1)
cl(22)	3066(1)	7530(2)	4603(1)	108(1)
cl(21)	4279(1)	9597(2)	5401(1)	144(1)

C(29)	6236(2)	3661(4)	6195(1)	52(1)
C(22)	4841(2)	6759(4)	5804(1)	55(1)
N(21)	2877(2)	4598(4)	6941(1)	59(1)
C(36)	4293(2)	4970(4)	6956(1)	57(1)
C(32)	7719(3)	2428(6)	6061(2)	79(1)
C(25)	3742(2)	5025(5)	5126(1)	72(1)
C(39)	3484(2)	2891(5)	6500(1)	59(1)
C(34)	7010(2)	4152(4)	6471(1)	67(1)
C(33)	7746(2)	3517(6)	6402(2)	84(1)
C(35)	4256(2)	3662(4)	6657(1)	50(1)
C(37)	3589(2)	5415(5)	7092(1)	60(1)
C(30)	6230(2)	2520(5)	5859(1)	72(1)
C(23)	4305(2)	7516(5)	5428(1)	64(1)
C(21)	4844(2)	5128(4)	5853(1)	45(1)
C(38)	2815(2)	3369(5)	6645(1)	65(1)
C(28)	5011(2)	3081(4)	6514(1)	57(1)
C(27)	5434(2)	4358(4)	6283(1)	50(1)
C(24)	3765(2)	6617(5)	5085(1)	64(1)
C(31)	6966(3)	1921(6)	5790(2)	83(1)
C(26)	4282(2)	4264(4)	5508(1)	66(1)
Cl(1)	4491(1)	-194(1)	511(1)	96(1)
C(15)	4313(2)	4836(4)	1710(1)	42(1)
C(7)	5234(2)	5619(3)	1162(1)	42(1)
Cl(2)	3196(1)	1308(2)	-375(1)	104(1)
N(1)	2792(2)	4366(4)	1897(1)	58(1)
C(9)	6149(2)	5810(4)	1148(1)	45(1)
C(4)	3794(2)	2511(5)	67(1)	55(1)
C(19)	4190(2)	3636(4)	2012(1)	54(1)
C(8)	5184(2)	5089(4)	1654(1)	49(1)
C(12)	7799(2)	6210(5)	1088(1)	69(1)
C(16)	3622(2)	5775(4)	1499(1)	53(1)
C(3)	4368(2)	1862(4)	446(1)	55(1)
C(10)	6792(2)	4856(5)	1394(1)	61(1)
C(17)	2869(2)	5512(5)	1597(1)	60(1)
C(1)	4742(2)	4499(4)	777(1)	41(1)
C(13)	7165(3)	7203(5)	834(2)	72(1)
C(14)	6347(2)	6999(4)	868(1)	60(1)
C(5)	3705(2)	4156(5)	34(1)	68(1)
C(2)	4849(2)	2857(4)	806(1)	49(1)
C(6)	4162(2)	5135(4)	385(1)	59(1)
C(11)	7609(2)	5055(6)	1368(1)	71(1)
C(18)	3433(2)	3418(5)	2098(1)	60(1)

Table 3. Bond lengths [Å] and angles [deg] for Compound 10c.

S(1)-O(4)	1.418(3)
S(1)-O(2)	1.419(3)
S(1)-O(1)	1.477(3)
S(1)-O(3)	1.504(3)
S(21)-O(23)	1.422(3)
S(21)-O(22)	1.432(3)
S(21)-O(21)	1.472(3)
S(21)-O(24)	1.502(3)
O(3)-H(3)	0.8200
O(24)-H(24)	0.8200
Cl(22)-C(24)	1.743(4)
Cl(21)-C(23)	1.731(4)
C(29)-C(30)	1.373(5)
C(29)-C(34)	1.374(5)
C(29)-C(27)	1.528(4)
C(22)-C(21)	1.363(5)
C(22)-C(23)	1.371(5)
C(22)-H(22)	0.9300
N(21)-C(37)	1.323(4)
N(21)-C(38)	1.331(5)
N(21)-H(21)	0.8600
C(36)-C(37)	1.375(5)
C(36)-C(35)	1.392(5)

C(36)-H(36)	0.9300
C(32)-C(31)	1.346(6)
C(32)-C(33)	1.348(7)
C(32)-H(32)	0.9300
C(25)-C(24)	1.330(6)
C(25)-C(26)	1.385(6)
C(25)-H(25)	0.9300
C(39)-C(38)	1.346(5)
C(39)-C(35)	1.384(4)
C(39)-H(39)	0.9300
C(34)-C(33)	1.387(6)
C(34)-H(34)	0.9300
C(33)-H(33)	0.9300
C(35)-C(28)	1.498(4)
C(37)-H(37)	0.9300
C(30)-C(31)	1.374(5)
C(30)-H(30)	0.9300
C(23)-C(24)	1.374(5)
C(21)-C(26)	1.378(5)
C(21)-C(27)	1.516(4)
C(38)-H(38)	0.9300
C(28)-C(27)	1.529(4)
C(28)-H(28A)	0.9700
C(28)-H(28B)	0.9700
C(27)-H(27)	0.9800
C(31)-H(31)	0.9300
C(26)-H(26)	0.9300
C1(1)-C(3)	1.724(4)
C(15)-C(16)	1.380(4)
C(15)-C(19)	1.391(4)
C(15)-C(8)	1.501(4)
C(7)-C(1)	1.522(4)
C(7)-C(9)	1.526(4)
C(7)-C(8)	1.546(4)
C(7)-H(7)	0.9800
C1(2)-C(4)	1.726(3)
N(1)-C(18)	1.321(4)
N(1)-C(17)	1.332(5)
N(1)-H(1)	0.8600
C(9)-C(10)	1.364(5)
C(9)-C(14)	1.383(5)
C(4)-C(3)	1.367(5)
C(4)-C(5)	1.375(6)
C(19)-C(18)	1.349(5)
C(19)-H(19)	0.9300
C(8)-H(8A)	0.9700
C(8)-H(8B)	0.9700
C(12)-C(11)	1.358(6)
C(12)-C(13)	1.381(6)
C(12)-H(12)	0.9300
C(16)-C(17)	1.365(5)
C(16)-H(16)	0.9300
C(3)-C(2)	1.409(5)
C(10)-C(11)	1.377(5)
C(10)-H(10)	0.9300
C(17)-H(17)	0.9300
C(1)-C(2)	1.375(4)
C(1)-C(6)	1.390(4)
C(13)-C(14)	1.387(5)
C(13)-H(13)	0.9300
C(14)-H(14)	0.9300
C(5)-C(6)	1.371(5)
C(5)-H(5)	0.9300
C(2)-H(2)	0.9300
C(6)-H(6)	0.9300
C(11)-H(11)	0.9300
C(18)-H(18)	0.9300
O(4)-S(1)-O(2)	112.52(15)
O(4)-S(1)-O(1)	110.9(2)
O(2)-S(1)-O(1)	110.64(17)
O(4)-S(1)-O(3)	107.50(18)

o(2)-s(1)-o(3)	109.39(19)
o(1)-s(1)-o(3)	105.62(15)
o(23)-s(21)-o(22)	112.10(15)
o(23)-s(21)-o(21)	110.2(2)
o(22)-s(21)-o(21)	112.53(19)
o(23)-s(21)-o(24)	106.8(2)
o(22)-s(21)-o(24)	109.71(18)
o(21)-s(21)-o(24)	105.11(16)
s(1)-o(3)-H(3)	109.5
s(21)-o(24)-H(24)	109.5
c(30)-c(29)-c(34)	117.3(3)
c(30)-c(29)-c(27)	123.4(3)
c(34)-c(29)-c(27)	119.3(3)
c(21)-c(22)-c(23)	121.7(3)
c(21)-c(22)-H(22)	119.2
c(23)-c(22)-H(22)	119.2
c(37)-N(21)-c(38)	121.2(3)
c(37)-N(21)-H(21)	119.4
c(38)-N(21)-H(21)	119.4
c(37)-c(36)-c(35)	120.0(3)
c(37)-c(36)-H(36)	120.0
c(35)-c(36)-H(36)	120.0
c(31)-c(32)-c(33)	119.6(4)
c(31)-c(32)-H(32)	120.2
c(33)-c(32)-H(32)	120.2
c(24)-c(25)-c(26)	120.0(3)
c(24)-c(25)-H(25)	120.0
c(26)-c(25)-H(25)	120.0
c(38)-c(39)-c(35)	120.8(3)
c(38)-c(39)-H(39)	119.6
c(35)-c(39)-H(39)	119.6
c(29)-c(34)-c(33)	120.3(4)
c(29)-c(34)-H(34)	119.9
c(33)-c(34)-H(34)	119.9
c(32)-c(33)-c(34)	120.9(4)
c(32)-c(33)-H(33)	119.5
c(34)-c(33)-H(33)	119.5
c(39)-c(35)-c(36)	116.9(3)
c(39)-c(35)-c(28)	120.7(3)
c(36)-c(35)-c(28)	122.3(3)
N(21)-c(37)-c(36)	120.2(3)
N(21)-c(37)-H(37)	119.9
c(36)-c(37)-H(37)	119.9
c(29)-c(30)-c(31)	121.6(4)
c(29)-c(30)-H(30)	119.2
c(31)-c(30)-H(30)	119.2
c(22)-c(23)-c(24)	119.6(4)
c(22)-c(23)-c1(21)	119.9(3)
c(24)-c(23)-c1(21)	120.4(3)
c(22)-c(21)-c(26)	117.2(3)
c(22)-c(21)-c(27)	119.4(3)
c(26)-c(21)-c(27)	123.3(3)
N(21)-c(38)-c(39)	120.9(3)
N(21)-c(38)-H(38)	119.6
c(39)-c(38)-H(38)	119.6
c(35)-c(28)-c(27)	114.6(3)
c(35)-c(28)-H(28A)	108.6
c(27)-c(28)-H(28A)	108.6
c(35)-c(28)-H(28B)	108.6
c(27)-c(28)-H(28B)	108.6
H(28A)-c(28)-H(28B)	107.6
c(21)-c(27)-c(29)	113.6(2)
c(21)-c(27)-c(28)	113.8(3)
c(29)-c(27)-c(28)	109.5(3)
c(21)-c(27)-H(27)	106.4
c(29)-c(27)-H(27)	106.4
c(28)-c(27)-H(27)	106.4
c(25)-c(24)-c(23)	120.2(3)
c(25)-c(24)-c1(22)	118.6(3)
c(23)-c(24)-c1(22)	121.2(3)
c(32)-c(31)-c(30)	120.2(4)
c(32)-c(31)-H(31)	119.9

C(30)-C(31)-H(31)	119.9
C(21)-C(26)-C(25)	121.3(3)
C(21)-C(26)-H(26)	119.4
C(25)-C(26)-H(26)	119.4
C(16)-C(15)-C(19)	116.8(3)
C(16)-C(15)-C(8)	124.3(3)
C(19)-C(15)-C(8)	118.8(3)
C(1)-C(7)-C(9)	111.7(2)
C(1)-C(7)-C(8)	112.2(2)
C(9)-C(7)-C(8)	111.2(2)
C(1)-C(7)-H(7)	107.1
C(9)-C(7)-H(7)	107.1
C(8)-C(7)-H(7)	107.1
C(18)-N(1)-C(17)	121.1(3)
C(18)-N(1)-H(1)	119.4
C(17)-N(1)-H(1)	119.4
C(10)-C(9)-C(14)	117.4(3)
C(10)-C(9)-C(7)	123.6(3)
C(14)-C(9)-C(7)	119.0(3)
C(3)-C(4)-C(5)	119.1(3)
C(3)-C(4)-C1(2)	121.2(3)
C(5)-C(4)-C1(2)	119.7(3)
C(18)-C(19)-C(15)	121.4(3)
C(18)-C(19)-H(19)	119.3
C(15)-C(19)-H(19)	119.3
C(15)-C(8)-C(7)	116.2(2)
C(15)-C(8)-H(8A)	108.2
C(7)-C(8)-H(8A)	108.2
C(15)-C(8)-H(8B)	108.2
C(7)-C(8)-H(8B)	108.2
H(8A)-C(8)-H(8B)	107.4
C(11)-C(12)-C(13)	119.2(3)
C(11)-C(12)-H(12)	120.4
C(13)-C(12)-H(12)	120.4
C(17)-C(16)-C(15)	119.8(3)
C(17)-C(16)-H(16)	120.1
C(15)-C(16)-H(16)	120.1
C(4)-C(3)-C(2)	120.6(3)
C(4)-C(3)-C1(1)	121.1(3)
C(2)-C(3)-C1(1)	118.2(3)
C(9)-C(10)-C(11)	121.8(4)
C(9)-C(10)-H(10)	119.1
C(11)-C(10)-H(10)	119.1
N(1)-C(17)-C(16)	120.9(3)
N(1)-C(17)-H(17)	119.6
C(16)-C(17)-H(17)	119.6
C(2)-C(1)-C(6)	118.5(3)
C(2)-C(1)-C(7)	121.8(3)
C(6)-C(1)-C(7)	119.7(3)
C(12)-C(13)-C(14)	119.5(4)
C(12)-C(13)-H(13)	120.3
C(14)-C(13)-H(13)	120.3
C(9)-C(14)-C(13)	121.4(3)
C(9)-C(14)-H(14)	119.3
C(13)-C(14)-H(14)	119.3
C(6)-C(5)-C(4)	120.7(3)
C(6)-C(5)-H(5)	119.6
C(4)-C(5)-H(5)	119.6
C(1)-C(2)-C(3)	119.9(3)
C(1)-C(2)-H(2)	120.0
C(3)-C(2)-H(2)	120.0
C(5)-C(6)-C(1)	121.1(3)
C(5)-C(6)-H(6)	119.5
C(1)-C(6)-H(6)	119.5
C(12)-C(11)-C(10)	120.6(4)
C(12)-C(11)-H(11)	119.7
C(10)-C(11)-H(11)	119.7
N(1)-C(18)-C(19)	120.0(3)
N(1)-C(18)-H(18)	120.0
C(19)-C(18)-H(18)	120.0

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($\text{Å}^2 \times 10^3$) for Compound 10c.

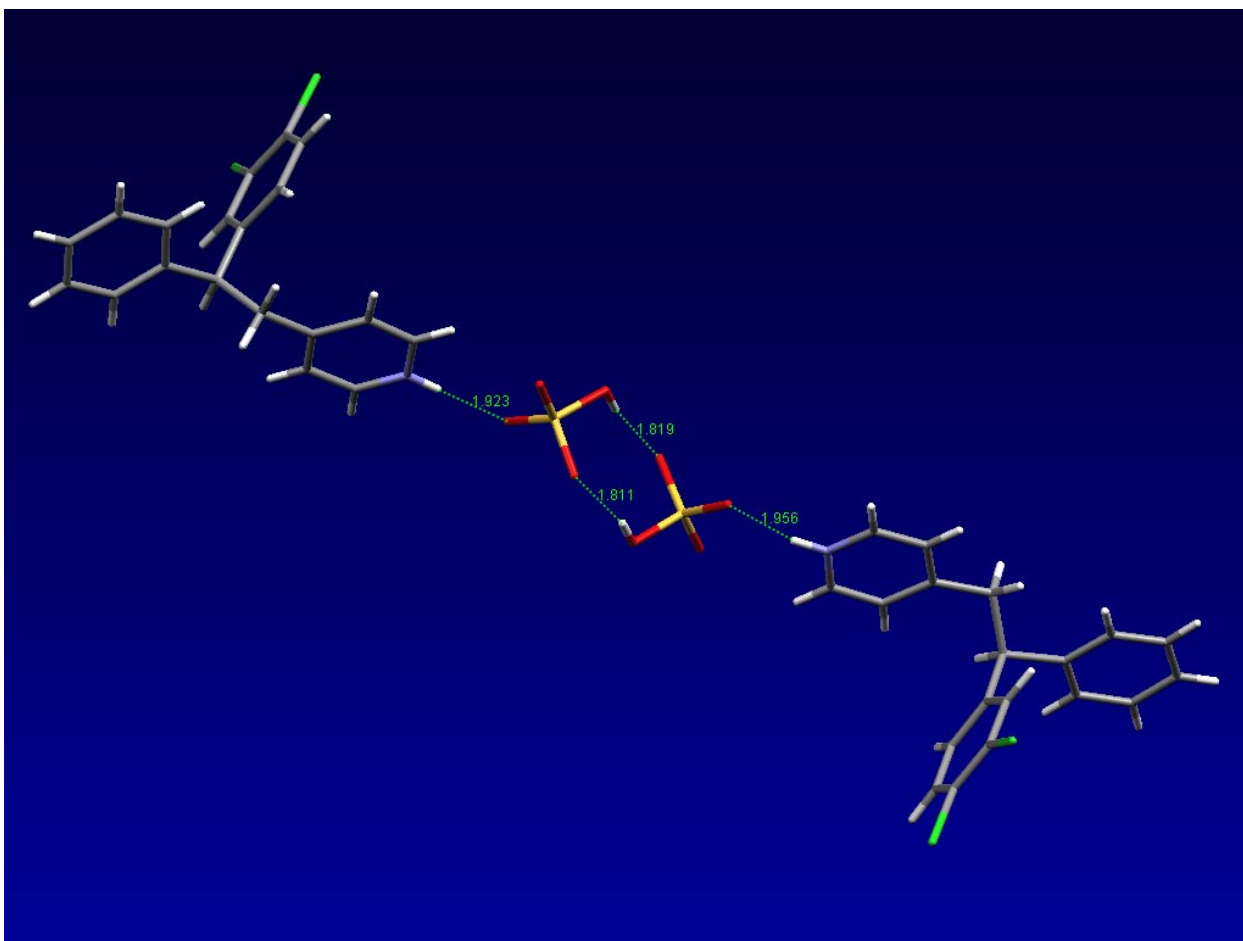
The anisotropic displacement factor exponent takes the form:
 $-2 \pi i^2 [h^2 a^*^2 U_{11} + \dots + 2 h k a^* b^* U_{12}]$

	U11	U22	U33	U23	U13	U12
S(1)	49(1)	66(1)	74(1)	20(1)	28(1)	16(1)
S(21)	59(1)	63(1)	70(1)	20(1)	31(1)	16(1)
O(1)	74(2)	82(2)	87(2)	-22(2)	19(1)	-3(2)
O(21)	80(2)	103(2)	95(2)	-41(2)	31(2)	-23(2)
O(22)	65(1)	67(2)	92(2)	28(1)	33(1)	17(1)
O(2)	61(1)	64(2)	90(2)	18(2)	34(1)	10(1)
O(23)	96(2)	89(2)	158(3)	62(2)	72(2)	39(2)
O(3)	65(2)	132(3)	63(2)	3(2)	12(1)	2(2)
O(24)	61(2)	121(3)	64(1)	1(2)	9(1)	14(2)
O(4)	83(2)	92(2)	132(2)	63(2)	56(2)	32(2)
C1(22)	112(1)	114(1)	74(1)	20(1)	-16(1)	28(1)
C1(21)	181(2)	60(1)	144(1)	30(1)	-39(1)	-17(1)
C(29)	45(2)	55(2)	56(2)	8(2)	12(1)	7(2)
C(22)	51(2)	54(2)	57(2)	-4(2)	10(2)	-8(2)
N(21)	56(2)	67(2)	57(2)	0(2)	23(1)	-2(2)
C(36)	47(2)	60(2)	59(2)	5(2)	7(1)	-6(2)
C(32)	60(2)	85(3)	100(3)	36(3)	37(2)	25(2)
C(25)	69(2)	68(3)	64(2)	-18(2)	-7(2)	1(2)
C(39)	62(2)	59(2)	58(2)	-9(2)	16(2)	-9(2)
C(34)	48(2)	54(2)	90(2)	-2(2)	4(2)	-3(2)
C(33)	43(2)	68(3)	130(4)	13(3)	5(2)	2(2)
C(35)	51(2)	52(2)	45(2)	15(2)	10(1)	1(2)
C(37)	69(2)	62(2)	48(2)	-6(2)	13(2)	1(2)
C(30)	58(2)	84(3)	68(2)	-9(2)	9(2)	7(2)
C(23)	71(2)	56(2)	61(2)	10(2)	12(2)	-3(2)
C(21)	36(1)	50(2)	50(2)	-1(2)	13(1)	5(1)
C(38)	56(2)	74(3)	69(2)	-7(2)	23(2)	-18(2)
C(28)	54(2)	54(2)	62(2)	11(2)	15(2)	7(2)
C(27)	49(2)	50(2)	52(2)	-4(1)	12(1)	0(1)
C(24)	60(2)	75(3)	51(2)	8(2)	6(2)	12(2)
C(31)	79(3)	98(3)	76(3)	-3(2)	27(2)	27(3)
C(26)	67(2)	43(2)	76(2)	-1(2)	0(2)	3(2)
C1(1)	121(1)	47(1)	111(1)	-13(1)	19(1)	9(1)
C(15)	42(1)	43(2)	41(1)	-10(1)	11(1)	-1(1)
C(7)	39(1)	36(1)	48(2)	2(1)	9(1)	3(1)
C1(2)	116(1)	97(1)	80(1)	-27(1)	-4(1)	-31(1)
N(1)	46(1)	71(2)	63(2)	-3(2)	24(1)	-1(1)
C(9)	41(2)	43(2)	50(2)	-3(1)	12(1)	-5(1)
C(4)	55(2)	63(2)	47(2)	-2(2)	11(2)	-13(2)
C(19)	51(2)	54(2)	55(2)	5(2)	13(1)	7(2)
C(8)	42(1)	56(2)	46(2)	-1(2)	11(1)	6(2)
C(12)	49(2)	73(3)	91(3)	-19(2)	29(2)	-23(2)
C(16)	50(2)	58(2)	53(2)	6(2)	18(1)	8(2)
C(3)	59(2)	48(2)	65(2)	-2(2)	28(2)	3(2)
C(10)	44(2)	58(2)	80(2)	12(2)	15(2)	-1(2)
C(17)	45(2)	68(2)	65(2)	3(2)	11(2)	16(2)
C(1)	38(1)	43(2)	45(2)	4(1)	16(1)	-1(1)
C(13)	82(3)	59(2)	90(3)	-2(2)	49(2)	-21(2)
C(14)	59(2)	52(2)	71(2)	7(2)	23(2)	2(2)
C(5)	68(2)	66(2)	56(2)	12(2)	-7(2)	-6(2)
C(2)	45(2)	51(2)	52(2)	4(2)	13(1)	2(1)
C(6)	62(2)	47(2)	59(2)	12(2)	0(2)	3(2)
C(11)	42(2)	74(2)	94(3)	2(2)	17(2)	-1(2)
C(18)	62(2)	58(2)	65(2)	8(2)	26(2)	-2(2)

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

	x	y	z	U(eq)
H(3)	-207	3823	1996	131
H(24)	10130	4975	2914	126
H(22)	5213	7374	6032	66
H(21)	2446	4865	7036	70
H(36)	4794	5543	7064	68
H(32)	8217	2029	6013	95
H(25)	3365	4422	4897	87
H(39)	3428	2034	6292	71
H(34)	7039	4913	6705	80
H(33)	8266	3848	6594	101
H(37)	3614	6294	7289	72
H(30)	5716	2143	5672	86
H(38)	2302	2834	6537	78
H(28A)	4839	2190	6297	68
H(28B)	5424	2673	6790	68
H(27)	5615	5219	6515	61
H(31)	6944	1162	5556	99
H(26)	4264	3150	5532	79
H(7)	4968	6682	1101	50
H(1)	2317	4240	1961	70
H(19)	4639	2971	2159	64
H(8A)	5497	4093	1734	58
H(8B)	5466	5896	1879	58
H(12)	8351	6332	1068	83
H(16)	3670	6583	1290	63
H(10)	6676	4050	1585	73
H(17)	2404	6143	1452	72
H(13)	7285	8000	642	86
H(14)	5922	7678	700	72
H(5)	3330	4609	-228	82
H(2)	5237	2404	1063	59
H(6)	4083	6243	360	71
H(11)	8035	4391	1543	85
H(18)	3363	2599	2298	72

Hydrogen bonding network of Compound 10c.



Two molecules of 10c are connected via hydrogen bonding to a pair of hydrogen bonded sulfate molecules. View generated using Mercury (Mercury 1.1.2; Copyright CDCC 2001-2002).